FLUPYRADIFURONE (285)

First draft prepared by Dr Paul Humphrey, Australian Pesticides and Veterinary Medicines Authority, Canberra, Australia

EXPLANATION

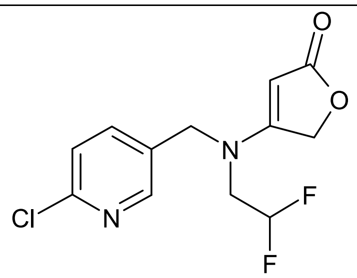
Flupyradifurone (fpd) is an insecticide belonging to the chemical class of butenolides. It acts as an agonist of the nicotinic acetylcholine receptor. It was scheduled for evaluation as a new compound by the 2015 JMPR at the 46th Session of the CCPR (2014). It was evaluated for toxicology in 2015. An ADI of 0–0.08 mg/kg bw and an ARfD of 0.2 mg/kg bw were established.

The manufacturer supplied information on identity, metabolism and environmental fate, methods of residue analysis, freezer storage stability, registered use patterns, supervised residue trials, fate of residues in processing and farm animal feeding studies.

SPECIFICATIONS

Specifications for flupyradifurone have not been developed by FAO.

IDENTITY

ISO common name:	Flupyradifurone
IUPAC name:	4-[(6-chloro-3-pyridylmethyl)(2,2-difluoroethyl)amino]furan-2(5H)-one
Chemical Abstract name:	2(5H)-furanone, 4-[[6-chloro-3-pyridinyl)methyl](2,2-difluoroethyl)amino]-
CAS No.:	951659-40-8
CIPAC No.:	Not allocated
Manufacturer's experimental name:	BYI 02960
Molecular Formula:	$C_{12}H_{11}ClF_2N_2O_2$
Structural Formula:	
Molecular mass:	288.68 g/mol

PHYSICAL AND CHEMICAL PROPERTIES

Pure Active Ingredient (except where noted as technical grade)

Property	Results	Test material purity and specification	Reference
Flupyradifurone			
Melting point	69.0 °C.	NLL 7780-47-4, purity 99.4% w/w	Smeykal 2010, 20090051.01
	67.1 °C.	PFV107N004, purity 97.6% w/w (technical grade)	Smeykal 2011a, 20110197.01

Property	Results	Test material purity and specification	Reference
Flupyradifurone			
Boiling point	Flupyradifurone has no boiling point at atmospheric pressure. Flupyradifurone decomposed at a temperature of approximately 270 °C.	NLL 7780-47-4, purity 99.4% w/w	Smeykal 2010, 20090051.01
	Flupyradifurone has no boiling point at atmospheric pressure. Flupyradifurone decomposed at a temperature of approximately 245 °C.	PFV107N004, purity 97.6% w/w (technical grade)	Smeykal 2011a, 20110197.01
Relative Density	Active substance, pure: 1.43 g/mL at 20 °C	NLL 7780-47-4, purity 99.4% w/w	Bogdoll and Strunk 2011a, PA09/006
	Active substance, technical grade: 1.52 g/mL at 20 °C	PFV107N004, purity 97.6% w/w	Eyrich and Bogdoll 2011b, PA11/063
Vapour pressure	Extrapolated: 9.1×10^{-7} Pa at 20 °C 1.7×10^{-6} Pa at 25 °C 2.6×10^{-5} Pa at 50 °C	NLL 7780-21-1, purity 99.9% w/w	Smeykal 2008, 20080615.01
Henry's law constant	Henry's law constants at 20 °C in distilled water (pH 7.0): 8.2×10^{-8} Pa × m^3 × mol^{-1}; at pH 4 8.2×10^{-8} Pa × m^3 × mol^{-1}; at pH 9 8.8×10^{-8} Pa × m^3 × mol^{-1}. A vapour pressure of 9.1×10^{-7} Pa and a water solubility of 3.2 g/L (pH 4 and 7) or 3.0 g/L (pH 9) were used to calculate the Henry’s law constant.		Bogdoll and Eyrich 2011, AF09/042
Description of the physical state and colour, purity of the ai. and of technical grade	Active substance, pure (at 21 °C): white powder	NLL 7780-47-4, purity 99.4% w/w	Bogdoll and Strunk 2011b, PA09/008
	Active substance technical grade (at 23°C): beige powder	PFV107N004, purity 97.6% w/w	Eyrich and Bogdoll 2011a, PA11/062
Solubility of purified active substance in water	pH 4 3.2 g/L at 20 °C pH 7 3.2 g/L at 20 °C pH 9 3.0 g/L at 20 °C	NLL 7780-47-4, purity 99.4% w/w (technical grade)	Wiche and Bogdoll 2011a, PA09/003
Solubility in organic solvents	[g/L at 20 °C] acetone > 250 dichloromethane > 250 dimethyl sulphoxide > 250 ethyl acetate > 250 methanol > 250 n-heptane 0.0005 toluene 3.7	NLL 7780-47-4, purity 99.4% w/w	Eyrich and Bogdoll 2011c, PA09/005
n-Octanol/ water partition coefficient	log P_{ow} at 25 °C pH 4 log P_{ow} 1.2 pH 7 log P_{ow} 1.2 pH 9 log P_{ow} 1.2	NLL 7780-47-4, purity 99.4% w/w	Bogdoll and Strunk 2011c, PA09/004
Hydrolysis rate at pH 4, 7 and 9 under sterile and dark conditions	Flupyradifurone is hydrolytically stable in aqueous solution at pH 4 to pH 9 (50 °C) for 5 days. No estimation of half-lives was done since flupyradifurone is stable in aqueous solution.	[Furanone-4-^{14}C] BYI 02960, vial no. C-1116 radiochemical purity 99%	Mislankar and Woodard 2011, MERVP019
Direct photo transformation in sterile water using artificial light	The first order half-life for photolytic degradation of flupyradifurone in sterile phosphate buffer (pH 7) was 13.8 experimental hours.	[Furanone-4-^{14}C] BYI 02960, vial no. C-1116A radiochemical purity 99.3%	Hall 2011a, MERVP042-1

Property	Results	Test material purity and specification	Reference
Flupyradifurone			
Quantum yield of direct transformation	A mean quantum yield of Φ = 0.000138 was calculated based on UV absorption data and the degradation kinetics determined from 2 degradation experiments.	NLL 7780-47-4, purity 99.4% w/w	Heinemann 2011a, MEF-11/554
Dissociation in water of purified active substance	No dissociation occurs in aqueous solutions in the pH-range 1 < pH < 12.	NLL 7780-47-4, purity 99.4% w/w	Wiche and Bogdoll 2011b, PA10/048
pH	pH 6.6 (1% suspension in distilled water at 24 °C)	NLL 7780-47-4, purity 99.4% w/w	Eyrich and Bogdoll 2011e, PA09/007
	pH 6.2 (1% suspension in distilled water at 22 °C)	PFV107N004, purity 97.6% w/w	Eyrich and Bogdoll 2011f, PA11/064

Formulations

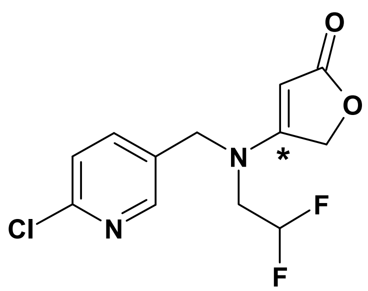
The formulations below are available for foliar and soil application.

Formulation type	Active substance and content
SL (Sivanto)—soluble concentrate	Flupyradifurone 200 g/L
FS—flowable concentrate	Flupyradifurone 480 g/L

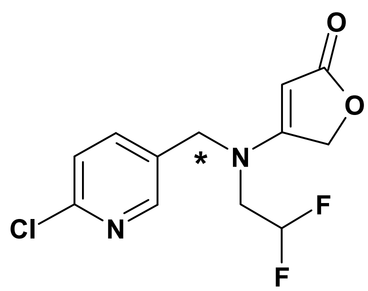
METABOLISM AND ENVIRONMENTAL FATE

General

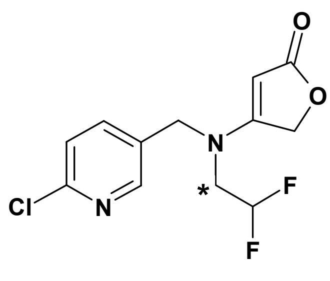
The studies for plant metabolism, animal metabolism and confined rotational crops were conducted with the test materials shown below, with the label position indicated in the following structural formulae:



[furanone-4-^{14}C]BYI 02960



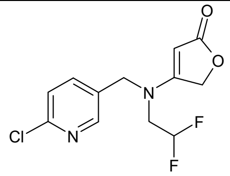
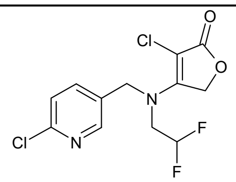
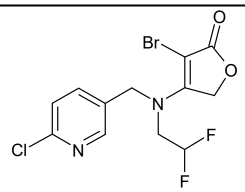
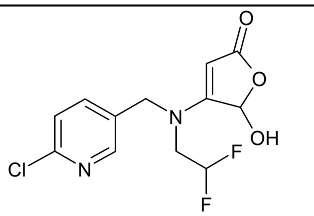
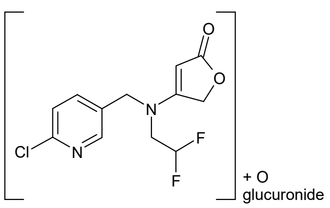
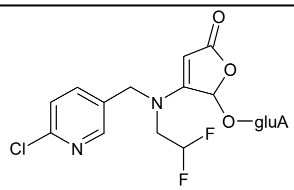
[pyridinylmethyl-^{14}C]BYI 02960

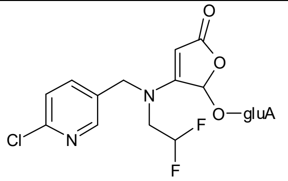
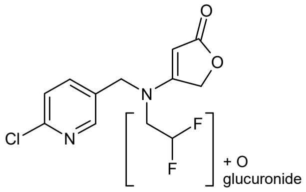
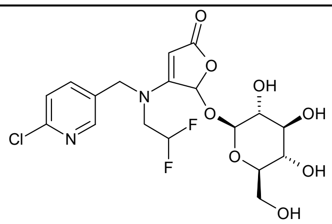
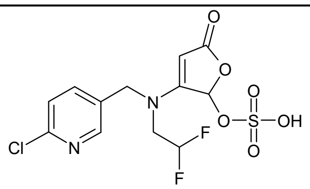
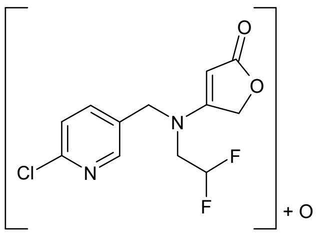
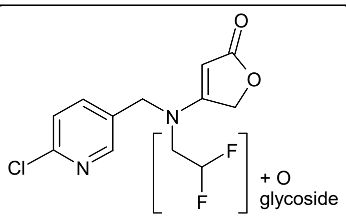
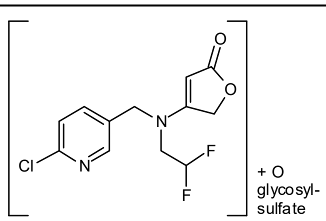
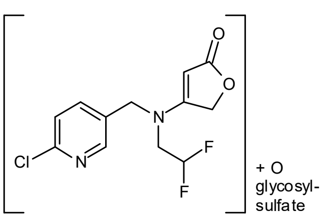


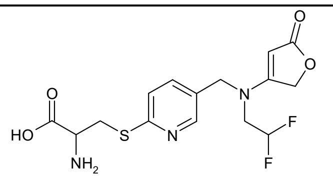
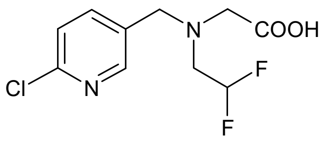
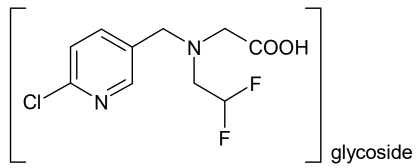
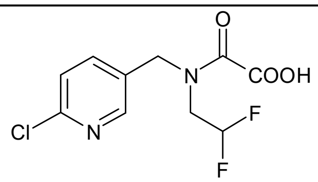
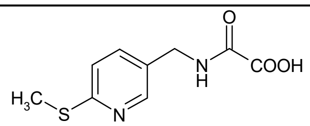
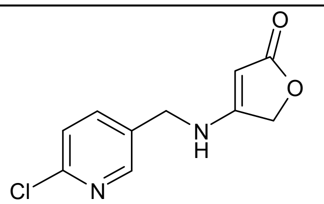
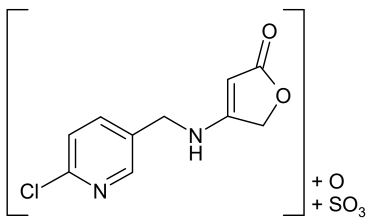
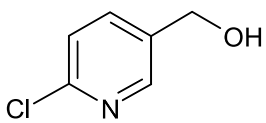
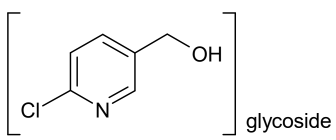
[ethyl-1-^{14}C]BYI 02960

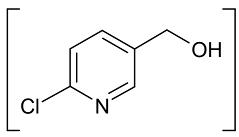
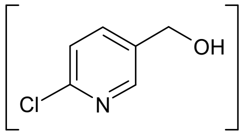
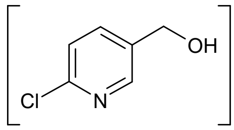
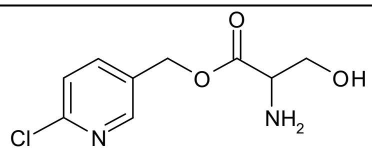
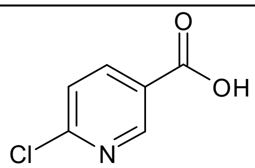
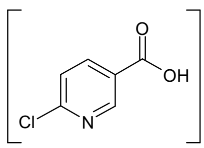
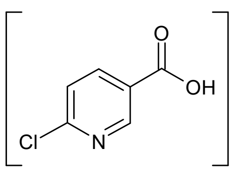
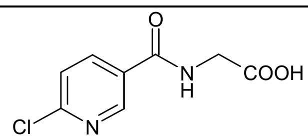
The table below summarizes the names, codes, and structures of the parent and principal metabolites found in plant, livestock, rat, rotational crop and soil studies.

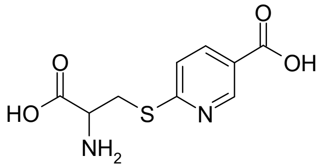
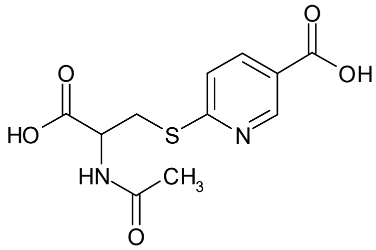
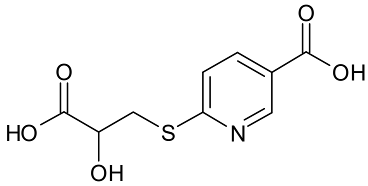
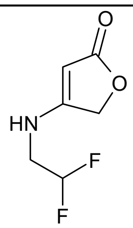
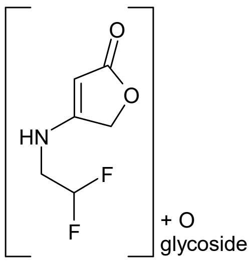
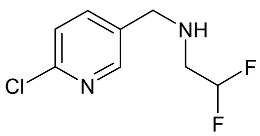
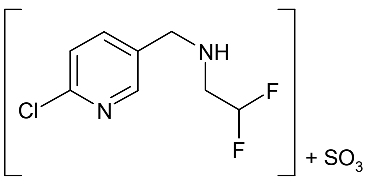
Flupyradifurone and metabolites/degradates found in metabolism and environmental fate studies:

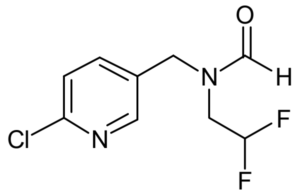
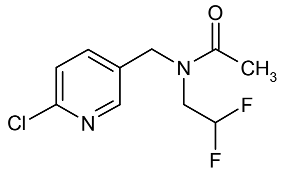
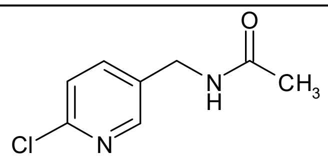
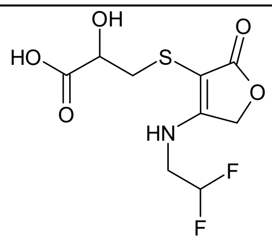
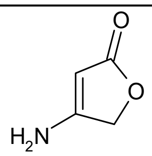
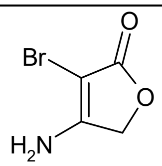
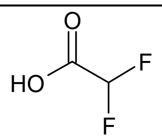
Abbreviation or Code	Chemical Structure	IUPAC Name	Found in
Active substance: Flupyradifurone fpd (BYI 02960)		4-[[(6-chloropyridin-3-yl)methyl](2,2-difluoroethyl)amino]furan-2(5H)-one	All matrices
BYI 02960-chloro		3-chloro-4-{[(6-chloropyridin-3-yl)methyl](2,2-difluoroethyl)amino}furan-2(5H)-one	Plant: rice cotton Swiss chard (CRC) [a] turnips (CRC) wheat (CRC) (co-elution with BYI 02960-bromo) Environment: aerobic soil (minor)
BYI 02960-bromo		3-bromo-4-{[(6-chloropyridin-3-yl)methyl](2,2-difluoroethyl)amino}furan-2(5H)-one	Plant: rice cotton Swiss chard (CRC) turnips (CRC) wheat (CRC) (co-elution with BYI 02960-chloro)
BYI 02960-OH		4-{[(6-chloropyridin-3-yl)methyl](2,2-difluoroethyl)amino}-5-hydroxyfuran-2(5H)-one	Animal: rat lactating goat laying hen Plant: tomato potato apple cotton rice Swiss chard (CRC) turnips (CRC) wheat (CRC)
BYI 02960-OH-gluA (isomer 1)			Animal: rat lactating goat
BYI 02960-OH-gluA (isomer 2 and 3, diastereomers)		3-{[(6-chloropyridin-3-yl) methyl](2,2-difluoroethyl) amino}-5-oxo-2,5-dihydrofuran-2-yl beta-D-glucopyranosiduronic acid	Animal: lactating goat

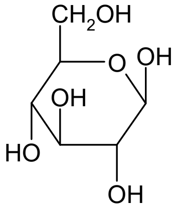
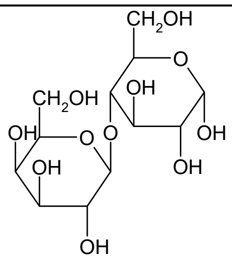
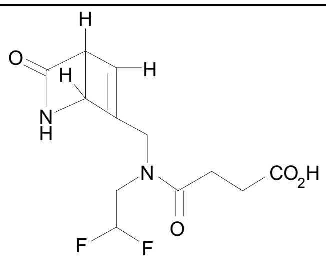
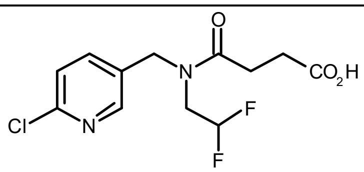
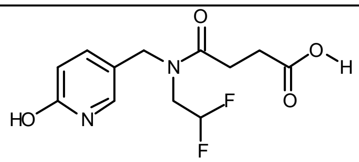
Abbreviation or Code	Chemical Structure	IUPAC Name	Found in
BYI 02960-OH-gluA (isomer 2 and 3, diastereomers)		3-{[(6-chloropyridin-3-yl) methyl](2,2-difluoroethyl) amino}-5-oxo-2,5-dihydrofuran-2-yl beta-D-glucopyranosiduronic acid	Animal: rat lactating goat
BYI 02960-OH-gluA (isomer 4)			Animal: lactating goat
BYI 02960-OH-glyc		3-{[(6-chloropyridin-3-yl)methyl](2,2-difluoroethyl)amino}-5-oxo-2,5-dihydrofuran-2-yl beta-D-glucopyranoside	Plant: tomato potato apple cotton (co-elution with BYI 02960-acetic acid) Swiss chard (CRC) turnips (CRC) wheat (CRC)
BYI 02960-OH-SA		3-{[(6-chloropyridin-3-yl)methyl](2,2-difluoroethyl)amino}-5-oxo-2,5-dihydrofuran-2-yl hydrogen sulfate	Animal: rat laying hen
BYI 02960-iso-OH)			Animal: rat lactating goat
BYI 02960-difluoroethyl-OH-glyc			Plant: apple
BYI 02960-OH-glyc-SA (isomer 1)			Plant: Swiss chard (CRC) turnips (CRC)
BYI 02960-OH-glyc-SA (isomer 2)			Plant: Swiss chard (CRC) turnips (CRC) wheat (CRC)

Abbreviation or Code	Chemical Structure	IUPAC Name	Found in
BYI 02960-cysteine		S-(5-{[(2,2-difluoroethyl)(5-oxo-2,5-dihydrofuran-3-yl)amino]methyl}pyridin-2-yl)cysteine	Animal: lactating goat
BYI 02960-acetic acid		N-[(6-chloropyridin-3-yl)methyl]-N-(2,2-difluoroethyl)glycine	Plant: apple rice cotton (co-elution with BYI 02960-OH-glyc) Swiss chard (CRC) turnips (CRC) wheat (CRC) Animal: laying hen
BYI 02960-acetic acid-glyc			Plant: apple rice cotton Swiss chard (CRC) turnips (CRC) wheat (CRC)
BYI 02960-glyoxylic acid		N-(6-chloropyridin-3-ylmethyl)-N-(2,2-difluoroethyl)oxamic acid	Plant: rice cotton Swiss chard (CRC) turnips (CRC) wheat (CRC)
BYI 02960-methylthio-glyoxylic acid		({[6-(methylsulfanyl)pyridin-3-yl]methyl}amino)(oxo)acetic acid	Animal: lactating goat
BYI 02960-des-difluoroethyl		4-[(6-chloropyridin-3-ylmethyl)amino]furan-2(5H)-one	Animal: rat lactating goat laying hen Environment: Aerobic soil (minor)
BYI 02960-difluoroethyl-OH-SA			Animal: laying hen
BYI 02960-CHMP		6-chloropyridin-3-ylmethanol	Plant: tomato potato apple turnips (CRC) wheat (CRC)
BYI 02960-CHMP-glyc			Plant: tomato potato apple

Abbreviation or Code	Chemical Structure	IUPAC Name	Found in
BYI 02960-CHMP-di-glyc			Plant: tomato potato apple Swiss chard (CRC) turnips (CRC) wheat (CRC)
BYI 02960-CHMP-glyc-di-SA			Plant: Swiss chard (CRC)
BYI 02960-CHMP-glyc-tri-SA			Plant: Swiss chard (CRC)
BYI 02960-CHMP-serinate			Animal: laying hen
CNA (6-CNA) (BYI 02960-CNA)		6-chloronicotinic acid	Animal: rat laying hen Plant: tomato potato apple cotton rice Swiss chard (CRC) turnips (CRC) wheat (CRC) Environment Aerobic soil (major)
BYI 02960-6-CNA-glycerol-gluA (isomer 1)			Plant: potato (isomer was not assigned) wheat (CRC)
BYI 02960-6-CNA-glycerol-gluA (isomers 2 and 3)			Plant: wheat (CRC)
BYI 02960-hippuric acid		N-[(6-chloropyridin-3-yl)carbonyl]glycine	Animal: rat lactating goat

Abbreviation or Code	Chemical Structure	IUPAC Name	Found in
BYI 02960-cysteinyl-nicotinic acid		6-[(2-amino-2-carboxyethyl)sulfanyl]nicotinic acid	Animal: lactating goat
BYI 02960-acetyl-cysteinyl-nicotinic acid			Animal: laying hen
BYI 02960-lactato-mercaptyl-nicotinic acid			Animal: laying hen
BYI 02960-difluoroethyl-amino-furanone (DFEAF)		4-[(2,2-difluoroethyl)amino]furan-2(5H)-one	Plant: tomato potato apple Swiss chard (CRC) turnips (CRC) wheat (CRC) Animal: rat
BYI 02960-difluoroethyl-amino-furanone-OH-glyc			Plant: Swiss chard (CRC) wheat (CRC)
BYI 02960-AMCP-difluoro-ethanamine		N-[(6-chloropyridin-3-yl)methyl]-2,2-difluoroethanamine	Plant: apple Animal: lactating goat
BYI 02960-AMCP-difluoro-ethanamine-SA			Animal: laying hen

Abbreviation or Code	Chemical Structure	IUPAC Name	Found in
BYI 02960-N-formyl-AMCP-difluoroethanamine		N-[(6-chloropyridin-3-yl)methyl]-N-(2,2-difluoroethyl)formamide	Plant: Swiss chard (CRC) turnips (CRC)
BYI 02960-N-acetyl-AMCP-difluoroethanamine		N-[(6-chloropyridin-3-yl)methyl]-N-(2,2-difluoroethyl)acetamide	Plant: Swiss chard (CRC) turnips (CRC)
BYI 02960-acetyl-AMCP			Animal: laying hen
BYI 02960-mercapto-lactic acid		3-({4-[(2,2-difluoroethyl)amino]-2-oxo-2,5-dihydrofuran-3-yl}sulfanyl)-2-hydroxypropanoic acid	Plant: wheat (CRC)
BYI 02960-amino-furanone		4-aminofuran-2(5H)-one	Plant: Swiss chard (CRC) turnips (CRC) wheat (CRC)
BYI 02960-bromo-amino-furanone		4-amino-3-bromofuran-2(5H)-one	Plant: wheat (CRC)
DFA (BYI 02960-DFA)		difluoroacetic acid	Animal: rat lactating goat laying hen Plant: tomato potato apple cotton rice Swiss chard (CRC) turnips (CRC) wheat (CRC) Environment aerobic soil (major) aerobic water/sediment (major)

Abbreviation or Code	Chemical Structure	IUPAC Name	Found in
Glucose (hexose)	 assumed structure		Plant: tomato apple rice turnips (CRC) wheat (CRC)
Lactose			Animal: lactating goat
BYI 02960-azabicyclosuccinamide		4-{(2,2-difluoroethyl)[(3-oxo-2-azabicyclo[2.2.0]hex-5-en-6-yl)methyl]amino}-4-oxobutanoic acid	Environment: water—aquatic photolysis (major)
BYI 02960-succinamide		4-{[(6-chloropyridin-3-yl)methyl](2,2-difluoroethyl)amino}-4-oxobutanoic acid	Environment: water—aquatic photolysis (major)
BYI 02960-deschlorohydroxysuccinamide		4-{(2,2-difluoroethyl)[(6-hydroxypyridin-3-yl)methyl]amino}-4-oxobutanoic acid	Environment: water—aquatic photolysis (minor)

[a] CRC = Confined rotational crop study

Plant metabolism

The metabolism of flupyradifurone (BYI 02960) has been investigated in tomato, potato, apple, cotton, and rice, using different application techniques (foliar application, soil application, seed piece treatment and granular application). A confined rotational crop study has also been conducted on wheat, Swiss chard and turnips grown after three plant back intervals.

Tomatoes

Four tomato plants (variety: *Philona*) were treated by soil drench application with [furanone-4-^{14}C]-BYI 02960 (Justus 2011a, MEF-11/016) or [pyridinylmethyl-^{14}C]-BYI 02960 (Justus 2011b, MEF-11/182) formulated as an SL 200. The first application was performed at BBCH 15 (5th leaf on main shoot unfolded) and a second application was made at BBCH 51 (1st inflorescence visible and first bud erect). The single soil drench application corresponded to 300 g ai/ha with a total seasonal application rate of 600 g ai/ha.

When the plants reached growth stage BBCH 61 (first flower of the first inflorescence open), the open flowers were sampled from one of the four tomato plants. Sampling of newly opened flowers was continued 2–3 times a week over a total period of approximately four (furanone label) or five (pyridinylmethyl label) weeks until end of flowering (at BBCH 69: nine or more inflorescences with open flowers). The sampling interval after the second application ranged from 3 to 36 days for the pyridinylmethyl label and 6 to 36 days for the furanone label.

When the three remaining tomato plants reached growth stage BBCH 81 (10% of fruits showed typical fully ripe colour), the ripe tomato fruits were harvested. Harvesting of newly ripe fruits was continued 2–3 times a week over a total period of approximately three weeks until the plants produced no new fruits (at BBCH 89: fully ripe). The PHI ranged from 69 to 92 days for the furanone label and 73 to 92 days for the pyridinylmethyl label.

The homogenised tomato flowers were extracted three times with a mixture of acetonitrile/water (8:2; v/v) and once with a mixture of acetonitrile/water (1:1; v/v). A portion of the homogenised tomato fruits was extracted four times with a mixture of acetonitrile/water (8:2; v/v). After each extraction step, extracts and solids were separated by centrifugation. The radioactivity in the extracts was determined by LSC and in the solids by combustion followed by LSC.

All tomato fruit extracts were combined and the first two tomato flower extracts were combined. The combined extracts were subjected to a clean-up step using a pre-conditioned SPE RP $_{18}$ cartridge (Phenomenex, Strata C_{18}-E). The flow-through fraction (percolate) was collected and the cartridge was rinsed with acetonitrile/water (8:2; v/v) and methanol/dichloromethane (1:1; v/v). The percolate and the acetonitrile/water fraction were combined and concentrated by rotary evaporation in vacuo.

The total radioactive residues (TRR) in fruits and flowers were 0.096 mg/kg equiv. and 0.721 mg/kg equiv. respectively for the furanone label and 0.130 mg/kg equiv. and 1.254 mg/kg equiv. respectively for the pyridinylmethyl label. The major portion of radioactivity was extracted conventionally by acetonitrile/water mixtures (84.8% to 93.6% of the TRR for furanone label and 96.5% to 98.5% of the TRR for the pyridinylmethyl label). Parent compound and metabolites in the concentrated extracts of fruits and flowers were analysed by HPLC. Identification was performed by HPLC and/or TLC co-chromatography with reference compounds, by comparison of HPLC profiles or by LC-MS.

Table 1 TRR values and distribution of parent and metabolites in tomatoes (fruit and flowers) after drench application with either [furanone-4-^{14}C]-BYI 02960 or [pyridinylmethyl-^{14}C]-BYI 02960

	[furanone-4-^{14}C]-flupyradifurone				[pyridinylmethyl-^{14}C]-flupyradifurone			
Matrix	Tomato fruits		Tomato flowers		Tomato fruits		Tomato flowers	
TRR [mg/kg]	0.096		0.721		0.130		1.254	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction								
BYI 02960 (parent)	35.9	0.034	77.9	0.561	24.2	0.031	66.2	0.829
glucose/carbohydrates	27.5	0.026	–	–				
difluoroethyl–amino-furanone	10.3	0.01	9.2	0.066				
OH-glyc	5.5	0.005	6.6	0.048	3.4	0.004	5.9	0.073
6-CNA					13.2	0.017	7.0	0.087
CHMP-di-glyc					37.1	0.048	8.0	0.100
CHMP-glyc					5.1	0.007	9.5	0.119
CHMP					3.3	0.004	–	–
Total identified	79.2	0.075	93.6	0.675	86.3	0.012	96.5	1.209
unknown 1	4.3	0.004	–	–	5.5	0.007	–	–
unknown 2					3.8	0.005	–	–
unknown 3					3.0	0.004	–	–
Total characterised	4.3	0.004	< 0.1	< 0.001	12.2	0.016	< 0.1	< 0.001
Analysed extract(s)	83.5	0.080	93.6	0.675	98.5	0.128	96.5	1.209
Extracts not analysed	1.3	0.001	–	–	–	–	–	–
Total extracted	84.8	0.081	93.6	0.675	98.5	0.128	96.5	1.209
Post-extraction solids (PES)	15.2	0.015	6.4	0.046	1.5	0.002	3.5	0.044
Accountability	100	0.096	100	0.721	100	0.130	100	1.254

Parent was the main residue in both extracts (35.9% of the TRR in fruits and 77.9% of the TRR in flowers for the furanone label and 24.2% of the TRR in tomato fruits and 66.2% of the TRR in tomato flowers for the pyridinylmethyl label).

Two additional major metabolites (> 10% of the TRR and ≥ 0.01 mg/kg equiv.) were detected in tomato fruits with the furanone label; a polar fraction which was assigned to glucose (or isomeric carbohydrates) (27.5% TRR, 0.026 mg/kg equiv.) and BYI 02960-difluoroethyl-amino-furanone (10.3% TRR, 0.01 mg/kg equiv.). The configuration of the hexose in BYI 02960-OH-glyc, found at 5.5% of the TRR, could be identified as D-glucose in the corresponding apple metabolism study due to the specific enzymatic treatment with ß-glucosidase.

In tomato fruits using the pyridinylmethyl label, the label-specific metabolite BYI 02960-CHMP-di-glyc was the main constituent (37.1% of the TRR, 0.048 mg/kg equiv.), followed by parent and the label-specific metabolite 6-CNA (13.2% of the TRR, 0.017 mg/kg equiv.). Additionally, three minor metabolites BYI 02960-CHMP-glyc (5.1% of the TRR), BYI 02960-OH-glyc (3.4% of the TRR) and BYI 02960-CHMP (3.3% of the TRR) were identified. In tomato flowers with this label, parent was the predominant component of the radioactivity besides four minor metabolites. The minor metabolites were identical to the metabolites identified in the tomato fruits, except for BYI 02960-CHMP, which was not present in flowers.

In a similar study three tomato plants (variety: *Philona*) were treated by soil drench application with [ethyl-1-^{14}C]-BYI 02960 formulated as an SL 200 (Unold and Justus 2011, MEF-11/498). Applications were made at similar timings and rates to those in the previous studies.

When the plants reached growth stage BBCH 61 (first flower of the first inflorescence open), the open flowers were sampled from one of the three tomato plants. Sampling of newly opened flowers was continued 2–3 times a week over a total period of approximately four weeks until end of flowering (at BBCH 69: nine or more inflorescences with open flowers).

When the two remaining tomato plants reached growth stage BBCH 81 (10% of fruits showed typical fully ripe colour), the ripe tomato fruits were harvested. Harvesting of newly ripe fruits was continued 2–3 times a week over a total period of approximately four weeks until the plants produced no new fruits (at BBCH 89: fully ripe). The PHI ranged from 56 to 86 days.

The TRR accounted for 0.201 mg/kg equiv. (in fruits) and 2.230 mg/kg equiv. (in flowers). Most the radioactivity was extracted conventionally by acetonitrile/ water mixtures (98.3% to 99.4% of the TRR). Parent compound and metabolites in the extracts of fruits and flowers were analysed by HPLC. Identification was performed by HPLC and/or TLC co-chromatography with reference compounds as well as by comparison of HPLC profiles.

Table 2 TRR values and distribution of parent and metabolites in tomatoes (fruit and flowers) after drench application of [ethyl-1-^{14}C]-BYI 02960

Matrix	Tomato fruits		Tomato flowers	
TRR [mg/kg]	0.201		2.230	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction				
BYI 02960 (parent)	10.0	0.020	33.0	0.736
difluoroacetic acid	86.6	0.174	59.8	1.334
difluoroethyl-amino-furanone	2.2	0.004	3.1	0.068
OH-glyc	0.6	0.001	2.4	0.054
Total identified	99.5	0.200	98.3	2.192
Total characterized	–	–	–	–
Analysed extract(s)	99.5	0.200	98.3	2.192
Extracts not analysed	–	–	–	–
Total extracted	99.5	0.200	98.3	2.192
Post-extraction solids (PES)	0.5	0.001	1.7	0.037
Accountability	100	0.201	100	2.230

The label-specific metabolite difluoroacetic acid (DFA) was the main residue in extracts of both matrices (86.6% TRR and 0.174 mg/kg equiv. in fruit and 59.8% TRR and 1.334 mg/kg equiv. in flowers), followed by parent (10.0% TRR and 0.020 mg/kg equiv. in fruit and 33.0% TRR and 0.736 mg/kg equiv. in flowers). Two additional minor metabolites (< 5% of the TRR), BYI 02960-difluoroethyl-amino-furanone and BYI 02960-OH-glyc, were identified in tomato fruits and flowers.

A metabolic pathway for flupyradifurone in tomatoes was proposed. The pathway involved:

- complete degradation of the furanone moiety and incorporation of carbon atoms into the natural compound pool, e.g. into glucose/carbohydrates
- cleavage of the pyridinylmethylamine bond and formation of BYI 02960-difluoroethylamino-furanone
- hydroxylation of the methylene group of the furanone moiety followed by conjugation with glucose
- cleavage of the pyridinylmethylamine bond followed by conjugation with carbohydrates or by oxidation of the methylene group to a carboxylic group, which were the major metabolic routes
- oxidative cleavage of the difluoroethylamine bond and formation of difluoroacetic acid.

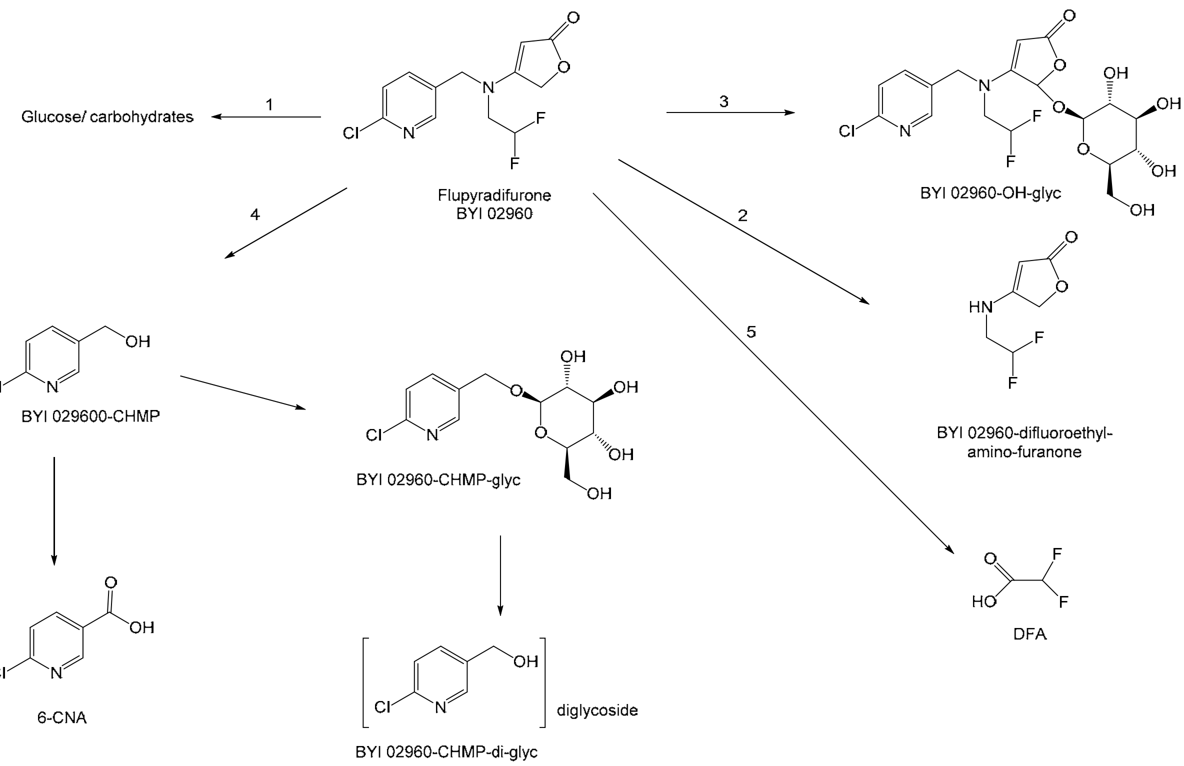


Figure 1 Proposed metabolic pathway for flupyradifurone in tomatoes

Potatoes

Two different methods of application to potatoes (variety: *Cilena*) were described. In one type of experiment, potato seed pieces were treated with either [furanone-4-[14]C]-BYI 02960 (Justus 2011c, MEF-10/769) or [pyridinylmethyl-[14]C]-BYI 02960 (Justus 2011d, MEF-10/710) formulated as an FS 480 at an application rate of 10.0 g ai/dt (254 g ai/ha, seed density 25 dt/ha for the furanone label and

270 g ai/ha, seed density 27 dt/ha for the pyridinylmethyl label). In the other type of experiment (same studies), either [furanone-4-^{14}C]-BYI 02960 or [pyridinylmethyl-^{14}C]-BYI 02960, formulated as an SL 200, was sprayed in-furrow onto the soil at an application rate of 626 g ai/ha (seed density 22 dt/ha or 28 dt/ha for the furanone and pyridinylmethyl labels respectively) prior to planting of the potato seed pieces. The potato tubers were harvested at maturity (BBCH 97) in both types of experiments. The leaves and roots, as well as the remainders of the potato seed pieces, were sampled at the same time.

The cut potato tubers of the seed piece treatment and the in-furrow treatment experiment were extracted three times with a mixture of acetonitrile/water (8:2; v/v) and once with a mixture of acetonitrile/water (1:1; v/v). The radioactivity in the extracts was determined by LSC and in the solids by combustion followed by LSC. The first two extracts were combined and subjected to a clean-up step using an SPE cartridge, which was conditioned with acetonitrile beforehand. The remaining solids (PES) for the furanone label, after conventional extraction of the potato tubers in the in-furrow treatment experiment, were subjected two times to exhaustive extraction with acetonitrile/water (1:1; v/v) using a microwave-assisted extraction system (120 °C for 20 min.).

At harvest (PHI = 97 days), the TRR in potato tubers were low after seed piece treatment and in-furrow application and accounted for 0.078 mg/kg equiv. and 0.171 mg/kg equiv., respectively for the furanone label and 0.076 mg/kg equiv. and 0.115 mg/kg equiv., respectively for the pyridinylmethyl label.). The TRR values in the remaining plant parts (leaves, roots and remainders of the potato seed pieces) were considerably higher. The TRR in potato leaves and roots was 6.97 mg/kg equiv. for treated seed pieces (tubers) and 7.01 mg/kg equiv. for in-furrow application for the furanone label, and 8.40 mg/kg equiv. from seed piece treatment and was 12.44 mg/kg equiv. from in furrow application for the pyridinylmethyl label.

Table 3 TRRs in potato matrices treated with either [furanone-4-^{14}C]-BYI 02960 or [pyridinylmethyl-^{14}C]-BYI 02960

			[furanone-4-^{14}C]-flupyradifurone		[pyridinylmethyl-^{14}C]-flupyradifurone	
Matrix	Application Type	Harvest growth stage	PHI (days)	TRR (mg ai. equiv./kg)	PHI (days)	TRR (mg ai. equiv./kg)
Potato tubers	Potato seed piece treatment at planting (10 g ai/dt)	BBCH 97	97	0.078	97	0.076
Potato leaves and roots			97	6.97	97	8.40
Remainders of the seed potato			97	36.21	97	33.33
Potato tubers	In-furrow treatment at planting (626 g ai/ha)	BBCH 97	97	0.171	97	0.115
Potato leaves and roots			97	7.01	97	12.44
Remainders of the seed potato			97	3.43	97	6.91

Parent compound and metabolites in the extracts of potato tubers were analysed by HPLC. Identification was performed by HPLC and/or TLC co-chromatography with reference compounds as well as by comparison of HPLC profiles. The distribution of parent and metabolites in tubers are shown below in Tables 4 and 5.

Table 4 Distribution of parent and metabolites in potato tubers after seed piece treatment or in-furrow application of [furanone-4-^{14}C]-BYI 02960

Application	Potato seed piece treatment		In-furrow application	
Matrix	Potato tubers		Potato tubers	
TRR [mg/kg]	0.078		0.171	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction				
BYI 02960 (parent)	40.0	0.031	50.6	0.086
difluoroethyl-amino-furanone	4.2	0.003	2.9	0.005
OH-glyc	6.6	0.005	4.4	0.007

Application	Potato seed piece treatment		In-furrow application	
Matrix	Potato tubers		Potato tubers	
TRR [mg/kg]	0.078		0.171	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg
Subtotal identified	50.8	0.039	57.9	0.099
unknown 1	7.2	0.006	5.5	0.009
unknown 2	1.7	0.001	–	–
unknown 3	2.4	0.002	1.9	0.003
unknown 4	1.1	0.001	–	–
Subtotal characterised	12.4	0.01	7.4	0.013
Conventional extracts losses	3.9	0.003	3.8	0.006
Total conventional extraction	67.0	0.052	69.0	0.118
Microwave extraction				
BYI 02960 (parent)	–	–	6.2	0.011
Subtotal identified	–	–	6.2	0.011
Microwave extracts losses	–	–	< 0.1	< 0.001
Total microwave extraction	–	–	6.3	0.011
Total identified	50.8	0.039	64.2	0.110
Total characterised	12.4	0.01	7.4	0.013
Analysed extract(s)	63.1	0.049	71.5	0.122
Extracts not analysed	3.9	0.003	3.8	0.006
Total extracted	67.0	0.052	75.3	0.129
Post–extraction solids (PES)	33.0	0.026	24.7	0.042
Accountability	100	0.078	100	0.171

Table 5 Distribution of parent and metabolites in potato tubers after seed piece treatment or in-furrow application of [pyridinylmethyl-^{14}C]-BYI 02960

Application	Potato seed piece treatment		In-furrow application	
Matrix	Potato tubers		Potato tubers	
TRR [mg/kg]	0.076		0.115	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction				
BYI 02960 (parent)	40.2	0.031	44.1	0.051
6-CNA	21.5	0.016	18.4	0.021
6-CNA-glycerol-gluA	–	–	2.3	0.003
CHMP-di-glyc	4.4	0.003	5.3	0.006
CHMP-glyc	3.7	0.003	2.4	0.003
CHMP	3.9	0.003	3.9	0.004
OH-glyc	6.7	0.005	4.7	0.005
Total identified	80.5	0.061	80.9	0.093
unknown 1	1.9	0.001	–	–
unknown 2	2.3	0.002	2.8	0.003
unknown 3	5.1	0.004	3.0	0.003
Total characterised	9.3	0.007	5.8	0.007
Analysed extract(s)	89.8	0.068	86.7	0.100
Extracts not analysed	3.7	0.003	3.6	0.004
Total extracted	93.4	0.071	90.4	0.104
Post-extraction solids (PES)	6.6	0.005	9.6	0.011
Accountability	100	0.076	100	0.115

A major portion of radioactivity in the potato tubers was extracted conventionally by acetonitrile/water mixtures (67.0% to 69.0% of the TRR for the furanone label and 90.4% to 93.4% of the TRR for the pyridinylmethyl label). Additionally, 6.3% of the TRR for the furanone label was released after exhaustive extraction of the remaining solids with a microwave assisted system as shown for the in-furrow experiment.

In total, 50.8% and 64.2% of the TRR were identified in potato tubers following seed piece treatment and in-furrow application, respectively for the furanone label while for the pyridinylmethyl label 80.5% and 80.9% of the TRR were identified in potato tubers following seed piece treatment and in-furrow application, respectively.

Parent was the main residue in the extracts of both seed piece and in-furrow treatments for both labels (0.031–0.086 mg/kg equiv., 40.0–50.6% TRR for the furanone label and 0.031–0.051 mg/kg equiv., 40.2–44.1% TRR for the pyridinylmethyl label). For the furanone label two additional minor metabolites, BYI 02960-difluoroethyl-amino-furanone and BYI 02960-OH-glyc, both < 10% of the TRR and < 0.01 mg/kg equiv. were also detected. For the pyridinylmethyl label several metabolites were identified, the major being 6-CNA at approximately 20% of the TRR (0.02 mg/kg equiv.). Other metabolites, like those observed in the pyridinylmethyl tomato study, were present at < 10% TRR and < 0.01 mg/kg equiv.

A metabolic pathway for flupyradifurone in potatoes was proposed. The pathway involved:

- hydroxylation of the methylene group of the furanone moiety followed by conjugation with glucose
- cleavage of the pyridinylmethylamine bond and either formation of BYI 02960-difluoroethylamino-furanone or conjugation with carbohydrates
- orby oxidation of the methylene group to a carboxylic group and conjugation with carbohydrates or derivatives.

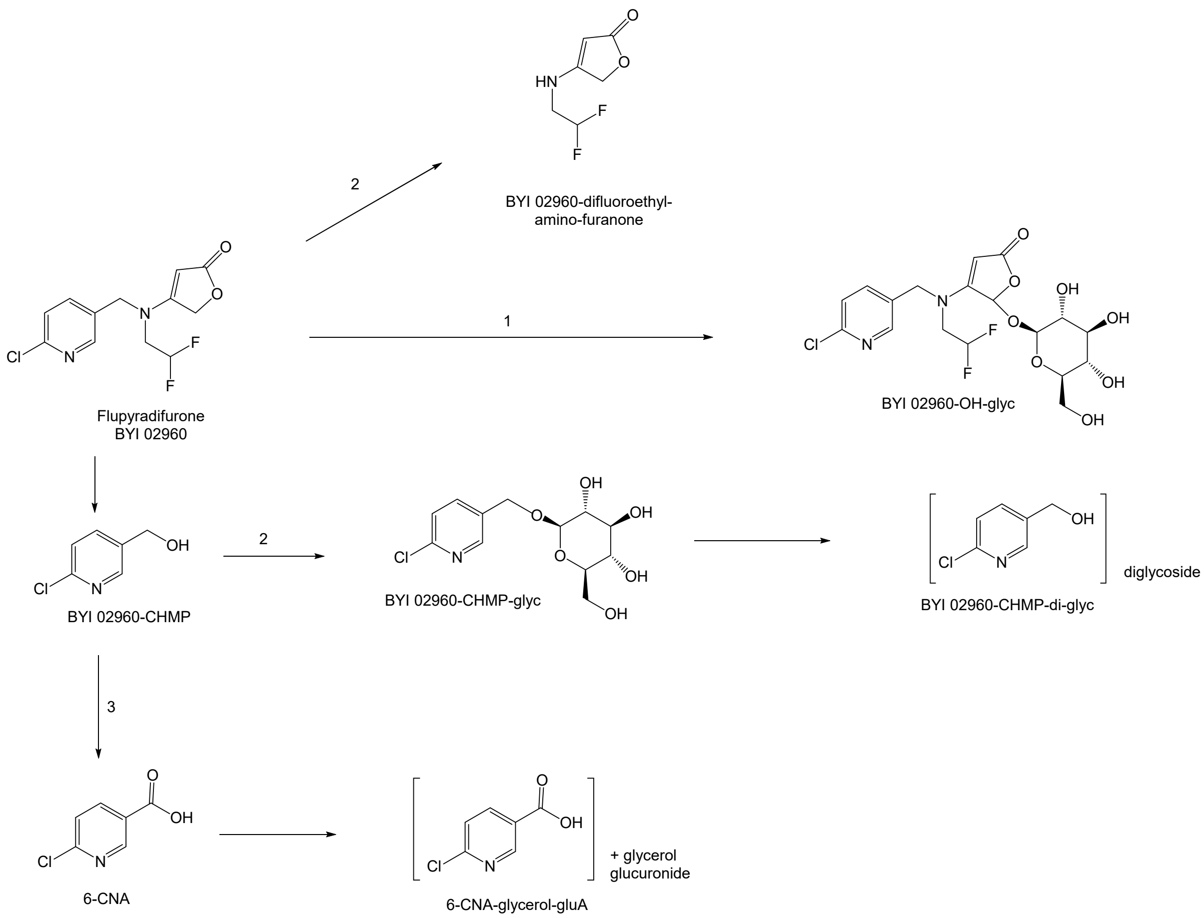


Figure 2 Metabolic pathway for flupyradifurone in potatoes

Apples

Two different methods of application to apples (variety: *James Grieve*) were described. In one type of experiment, (single application experiment), two apple trees were treated with either [furanone-4-^{14}C]-BYI 02960 (Justus 2011e, MEF-11/499) or [pyridinylmethyl-^{14}C]-BYI 02960 (Justus 2011f, MEF-11/198) as an SL formulation at an actual application rate of 86 g ai/ha/per meter canopy height (86 g ai/(ha × m CH)) for the furanone label and 87 g ai/(ha × m CH) for the pyridinylmethyl label, both at the end of flowering (BBCH 69). In another type of experiment (same studies), one apple tree was treated twice with 86 g ai/(ha × m CH) for the furanone label, and one time at 87 g ai/(ha × m CH) and a second time at 85 g ai/(ha × m CH) for the pyridinylmethyl label. For both labels, applications were made at the end of flowering (BBCH 69) and at 14 days before harvest.

Apple fruits from both experiments were sampled at maturity (BBCH 87–89). The PHI was 98 days for the single application and 14 days for the double application. On the same day, leaves were sampled.

From the single application experiment, the apple pieces were extracted three times with a mixture of acetonitrile/water (8:2; v/v) and once with a mixture of acetonitrile/water (1:1; v/v). Extracts and solids were separated. All extracts were combined and subjected to a clean-up step using an SPE cartridge, pre-conditioned with acetonitrile. The flow through fraction (percolate) and the acetonitrile/water rinse fraction were combined and concentrated by rotary evaporation in vacuo. The pH value of the concentrate was adjusted to pH 7 with ammonium carbonate before chromatographic analysis by HPLC. For the furanone label the solids remaining after the conventional extraction steps were twice extracted by a microwave-assisted step with acetonitrile/water (1:1; v/v). After each extraction step, extracts and solids were separated by centrifugation. Extracts were combined and concentrated by rotary evaporation. Prior to HPLC analysis an aliquot of the extract was treated with cellulose to reduce viscosity.

From the double application experiment, a sub-sample of the whole apple fruits was subjected to a surface wash with dichloromethane. The dichloromethane was removed by a nitrogen stream and the remaining concentrate was analysed by HPLC. The washed apples were cut into pieces. Extraction, purification and concentration of the extract were performed exactly as described for the single application experiment. Additionally, three whole apples from the double application experiment were extracted without prior surface wash. The resulting sample was adjusted to pH 7 with ammonium carbonate for HPLC analysis.

Aliquots of the apple leaves of the single and double application experiments were conventionally extracted as described for the fruits from the single application experiment.

The radioactivity in the extracts was determined by LSC and in the PES by combustion followed by LSC. The TRR levels in the apple fruits were 0.280 mg/kg equiv. from the single application furanone label and 0.079 mg/kg equiv. following single application of the pyridinylmethyl label. After double application of the furanone label, the apple fruits had a TRR of 1.133 mg/kg equiv. (extraction including surface wash) and 1.286 mg/kg equiv. (extraction without surface wash) while for the pyridinylmethyl label TRRs of 1.868 mg/kg equiv. (extraction including surface wash) and 0.545 mg/kg equiv. (extraction without surface wash) were observed. The TRR values of the apple leaves were high; 38.957 mg/kg equiv. in the leaves of the single application experiment and 102.92 mg/kg equiv. in the leaves of the double application experiment for the furanone label and 56.72 mg/kg equiv. and 134.84 mg/kg equiv. respectively for the pyridinylmethyl label experiments..

Table 6 TRRs in apple matrices after treatment with [furanone-4-^{14}C]-BYI 02960 or [pyridinylmethyl-^{14}C]-BYI 02960

			[furanone-4-^{14}C]-flupyradifurone		[pyridinylmethyl-^{14}C]-flupyradifurone	
Matrix	Application Type	Harvest growth stage	PHI (days)	TRR (mg/kg)	PHI (days)	TRR (mg/kg)
Apple fruits	1 foliar spray applications at BBCH 69, 86 g ai/ha/ mCH	BBCH 87–89	98	0.280	98	0.079
Apple leaves			98	38.96	98	56.72

			[furanone-4-^{14}C]-flupyradifurone		[pyridinylmethyl-^{14}C]-flupyradifurone	
Matrix	Application Type	Harvest growth stage	PHI (days)	TRR (mg/kg)	PHI (days)	TRR (mg/kg)
	(furanone label) or 87 g ai/ha/ mCH (pyridinylmethyl label)					
Apple fruits with surface wash	2 foliar spray applications at BBCH 69 and 14 day PHI, 2 × 86 g ai/ha/ mCH (furanone label) or 87 and 85 g ai/ha/ mCH (pyridinylmethyl label)	BBCH 87–89	14	1.133	14	1.868
Apple fruits without surface wash			14	1.286	14	0.545
Apple leaves			14	102.92	14	134.84

Parent compound and metabolites in the extracts of apple fruit and leaves were analysed by HPLC. Identification was performed by HPLC and/or TLC co-chromatography with reference compounds, by comparison of HPLC profiles or by LC-MS/MS spectrometry. The distribution of parent and metabolites in apples is shown below.

Table 7 Distribution of parent and metabolites in apple fruits and leaves after a single foliar treatment of [furanone-4-^{14}C]-BYI 02960

Application	Single application			
Matrix	Apple fruits		Apple leaves	
TRR [mg/kg]	0.280		38.96	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction				
BYI 02960 (parent)	7.4	0.021	26.0	10.14
glucose/carbohydrates	50.3	0.141	2.5	0.991
acetic acid-glyc	0.3	0.001	6.4	2.486
OH-glyc	0.4	0.001	36.1	14.06
acetic acid	0.2	0.001	2.5	0.956
difluoroethyl-OH-glyc	–	–	5.8	2.264
OH	–	–	0.6	0.244
Subtotal identified	58.7	0.164	79.9	31.14
unknown 1	–	–	0.3	0.120
unknown 3	–	–	1.7	0.646
unknown 4	–	–	–	–
unknown 5	–	–	2.6	1.002
unknown 6	–	–	1.6	0.609
unknown 7	–	–	1.5	0.578
unknown 8	–	–	4.9	1.895
unknown 9			1.6	0.612
Subtotal characterised	–	–	14.0	5.463
Conventional extracts not analysed	1.1	0.003	0.3	0.121
Total conventional extraction	59.8	0.168		
Microwave extraction				
glucose/carbohydrates	6.4	0.018		
difluoroethyl-amino-furanone	1.0	0.003		
Subtotal identified	7.5	0.021		
unknown 2	2.0	0.005		
Subtotal characterised	2.0	0.005		
Microwave extracts not analysed	< 0.1	< 0.001		
Total microwave extraction	9.5	0.027	–	–
Cellulase digestion				
glucose/carbohydrates	15.0	0.042		
difluoroethyl-amino-furanone	2.2	0.006		
Subtotal identified	17.2	0.048		
Total cellulase extraction	17.2	0.048	–	–
Total identified	83.4	0.234	79.9	31.14
Total characterised	2.0	0.005	14.0	5.463
Analysed extract(s)	88.5	0.239	94.0	36.60

Application	Single application			
Matrix	Apple fruits		Apple leaves	
TRR [mg/kg]	0.280		38.96	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg
Extracts not analysed	1.2	0.003	0.3	0.121
Total extracted	86.5	0.242	94.3	36.73
Post-extraction solids (PES)	13.5	0.038	5.7	2.232
Accountability	100	0.280	100	38.96

Table 8 Distribution of parent and metabolites in apple fruits and leaves after two foliar treatments of [furanone-4-^{14}C]-BYI 02960

Application	Double application experiment					
Matrix	Apple fruits with surface wash		Apple fruits without surface wash		Apple leaves	
TRR [mg/kg]	1.133		1.286		102.92	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Surface wash						
BYI 02960 (parent)	6.7	0.076	–	–	–	–
glucose/carbohydrates	0.2	0.002	–	–	–	–
OH-glyc	0.2	0.002	–	–	–	–
acetic acid	0.1	0.001	–	–	–	–
OH	0.1	0.001	–	–	–	–
Subtotal identified	7.2	0.082	–	–	–	–
unknown 8	< 0.1	< 0.001	–	–	–	–
unknown 9	0.3	0.003	–	–	–	–
Subtotal characterised	0.3	0.003	–	–	–	–
Total surface wash with CH_2Cl_2	7.5	0.085	–	–	–	–
Conventional extraction						
Parent	64.7	0.733	73.6	0.946	57.9	59.547
glucose/carbohydrates	13.6	0.154	14.2	0.182	3.6	3.687
difluoroethyl-amino-furanone			0.2	0.003	0.7	0.736
acetic acid-glyc	0.8	0.009	0.5	0.007	4.2	4.274
OH-glyc	1.0	0.012	1.1	0.014	17.3	17.86
acetic acid	0.6	0.007	0.7	0.009	1.2	1.214
difluoroethyl-OH-glyc	–	–	–	–	2.1	2.118
OH	0.9	0.01	0.8	0.01	0.6	0.630
Subtotal identified	81.6	0.925	91.1	1.171	87.5	90.06
unknown 1	–	–	–	–	0.6	0.646
unknown 3	–	–	–	–	0.5	0.474
unknown 4	–	–	–	–	0.5	0.530
unknown 5	–	–	–	–	1.9	1.960
unknown 6	–	–	–	–	0.8	0.851
unknown 7	–	–	–	–	0.5	0.501
unknown 8	–	–	–	–	2.5	2.609
unknown 9	–	–	0.1	0.002	1.2	1.210
Subtotal characterised	–	–	0.1	0.002	8.5	8.781
Conventional extracts not analysed	0.3	0.003	0.7	0.009	0.4	0.436
Total conventional extraction	81.9	0.928	91.9	1.182	96.5	99.28
Cellulase digestion						
glucose/carbohydrates	3.3	0.037				
difluoroethyl-amino-furanone	0.6	0.007				
Subtotal identified	3.9	0.044				
Total cellulase extraction	3.9	0.044	–	–	–	–
Total identified	92.7	1.051	91.1	1.171	87.5	90.06
Total characterised	0.3	0.003	0.1	0.002	8.5	8.781
Analysed extract(s)	93.0	1.054	91.2	1.173	96.0	98.84
Total Losses	0.3	0.003	0.7	0.009	0.4	0.436
Total extracted	93.3	1.057	91.9	1.182	96.5	99.28
Post-extraction solids (PES)	6.7	0.076	8.1	0.104	3.5	3.639
Accountability	100	1.133	100	1.286	100	102.92

Table 9 Distribution of parent and metabolites in apple fruits and leaves after a single foliar treatment with [pyridinylmethyl-^{14}C]-BYI 02960

Application	Single application

Matrix	Apple fruits		Apple leaves	
TRR [mg/kg]	0.079		56.72	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction				
BYI 02960 (parent)	43.1	0.034	24.5	13.88
6-CNA	5.0	0.004	–	–
CHMP-di-glyc	–	–	0.6	0.342
CHMP-glyc	4.7	0.004	14.4	8.141
CHMP	4.0	0.003	1.3	0.727
acetic acid-glyc	3.5	0.003	5.1	2.891
OH-glyc	4.9	0.004	19.9	11.278
acetic acid	3	0.002	1.4	0.767
difluoroethyl-OH-glyc	1.4	0.001	6.4	3.631
AMCP-difluoroethanamine	8.4	0.007	0.4	0.255
OH	0.8	0.001	0.9	0.484
Total identified	78.9	0.062	74.8	42.40
unknown 1	2.7	0.002	–	–
unknown 2	6.6	0.005	–	–
unknown 3	3.5	0.003	1.8	1
unknown 4	2.0	0.002	3.6	2.052
unknown 6	–	–	1.1	0.634
unknown 7	–	–	0.6	0.322
unknown 9	–	–	2.6	1.477
unknown 10	–	–	1.3	0.762
unknown 11	–	–	0.5	0.295
unknown 14	–	–	0.6	0.353
unknown 15	–	–	0.6	0.315
unknown 16	–	–	1.2	0.682
unknown 17	–	–	0.7	0.403
unknown 18	–	–	2.4	1.386
unknown 19	–	–	1.9	1.088
unknown 20	0.6	< 0.001	2.5	1.445
unknown 21	–	–	0.2	0.114
Total characterised	15.3	0.012	21.7	12.328
Analysed extract(s)	94.2	0.074	96.5	54.727
Losses	–	–	0.2	0.132
Total extracted	94.2	0.074	96.7	54.86
Post–extraction solids (PES)	5.8	0.005	3.3	1.855
Accountability	100	0.079	100	56.72

Table 10 Distribution of parent and metabolites in apple fruits and leaves after two foliar treatments with [pyridinylmethyl-^{14}C]-BYI 02960

Application	Double application					
Matrix	Apple fruits with surface wash		Apple fruits without surface wash		Apple leaves	
TRR [mg/kg] =	1.868		0.545		134.84	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Surface wash						
BYI 02960 (parent)	10.3	0.193				
OH-glyc	0.3	0.006				
acetic acid	0.2	0.003				
OH	< 0.1	0.001				
Subtotal identified	10.9	0.203				
unknown 20	0.1	0.002				
Subtotal characterised	0.1	0.002				
Total surface wash with CH_2Cl_2	11.0	0.205				
Conventional extraction						
BYI 02960 (parent)	78.1	1.459	85.6	0.467	48.2	64.98
6-CNA	0.5	0.009	1.5	0.008	0.3	0.436
CHMP-di-glyc	–	–	–	–	0.2	0.327
CHMP-glyc	0.5	0.01	0.9	0.005	7.3	9.842

Application	Double application					
Matrix	Apple fruits with surface wash		Apple fruits without surface wash		Apple leaves	
TRR [mg/kg] =	1.868		0.545		134.84	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
CHMP	0.7	0.013	0.8	0.004	0.6	0.777
acetic acid-glyc	0.6	0.012	0.8	0.004	5.0	6.799
OH-glyc	1.0	0.019	1.7	0.009	15.4	20.73
acetic acid	0.6	0.012	1.1	0.006	1.0	1.410
difluoroethyl-OH-glyc	–	–	–	–	4.9	6.674
AMCP-difluoroethanamine	4.5	0.085	4.1	0.023	0.8	1.015
OH	1.0	0.019	1.0	0.005	0.7	0.944
Subtotal identified	87.6	1.636				
unknown 3	0.2	0.004	–	–	0.8	1.070
unknown 4	–	–	–	–	2.0	2.652
unknown 5	–	–	–	–	0.2	0.222
unknown 6	–	–	–	–	0.3	0.406
unknown 7	–	–	–	–	0.4	0.601
unknown 8	–	–	–	–	0.4	0.504
unknown 9	–	–	–	–	1.6	2.150
unknown 10	–	–	–	–	0.5	0.721
unknown 11	–	–	–	–	0.5	0.654
unknown 12	–	–	–	–	0.2	0.323
unknown 13	0.3	0.005	–	–	0.4	0.518
unknown 16	–	–	–	–	1.0	1.380
unknown 17	–	–	–	–	0.5	0.669
unknown 18	–	–	–	–	1.7	2.291
unknown 19	–	–	–	–	0.8	1.091
unknown 20			0.5	0.002	1.7	2.248
unknown 21	–	–	–	–	0.3	0.413
Subtotal characterised	0.5	0.009				
Conventional extracts not analysed	0.2	0.003				
Total conventional extraction	88.2	1.648				
Total identified	98.5	1.839	97.6	0.532	84.5	113.93
Total characterised	0.6	0.011	0.5	0.002	13.3	17.91
Analysed extract(s)	99.0	1.850	98.0	0.534	97.8	131.85
Extracts not analysed	0.2	0.003	0.6	0.004	0.6	0.788
Total extracted	99.2	1.853	98.7	0.538	98.4	132.64
Post–extraction solids (PES)	0.8	0.015	1.3	0.007	1.6	2.206
Accountability	100	1.868	99.8	0.545	100	134.84

The radioactive residues were efficiently extracted with acetonitrile/water mixtures, including both conventional and exhaustive extraction procedures for the furanone label (86.5% to 96.5% of the TRR) and conventional procedures for the pyridinylmethyl label (94.2% to 99.2% of the TRR). For the apple fruits of the furanone label double application experiment, a portion of 7.5% of the TRR was removed by surface wash and another portion of 81.9% was extracted conventionally indicating a good uptake of the compound and its systemic behaviour. For the apple fruits from the pyridinylmethyl label double application experiment, a portion of 11.0% of the TRR was removed by surface wash and another portion of 88.2% was extracted conventionally.

The main compound in apple fruits of the single application furanone label experiment was the natural compound glucose (or a corresponding isomeric carbohydrate) (50.3% TRR, 0.141 mg/kg equiv.). It was also a major component in the apple fruits of the double application experiment (14% TRR, 0.154–0.182 mg/kg equiv.). Parent was the main component in the extracts of apple fruits (71.4–73.6% TRR, 0.733–0.946 mg/kg equiv.) and leaves (57.9% TRR, 59.5 mg/kg equiv.) from the double application furanone label experiment. In the surface wash, parent was the main component (6.7% TRR, 0.076 mg/kg equiv.). The high amount of parent may reflect the short PHI of 14 days. All other metabolites detected in the raw agricultural commodity fruit were minor metabolites (each < 10% of the TRR).

The main compound in apple fruits and leaves of the single (43.1% TRR, 0.034 mg/kg equiv. and 24.5% TRR, 13.88 mg/kg equiv. respectively) and the double application (78.1–88.4% TRR, 0.467–1.46 mg/kg equiv. and 48.2% TRR, 65.0 mg/kg equiv. respectively) experiments using the pyridinylmethyl label was parent. Parent was also the main component of the surface wash (10.3% TRR, 0.193 mg/kg equiv.). All other metabolites detected in the raw agricultural commodity fruit were minor metabolites which not exceed 10% of the TRR, in either the single or the double application experiment.

BYI 02960-OH-glyc was observed in apple leaves at 17–31% of the TRRs using both labels and in both the single and double application experiments.

A metabolic pathway for flupyradifurone in apples was proposed. The pathway involved:

- hydroxylation of the furanone or the difluoroethyl moiety followed by conjugation with carbohydrates
- complete degradation of the furanone moiety and incorporation of carbon atoms into the natural compound pool, *i.e.* into glucose/carbohydrates
- cleavage of the pyridinylmethylamine bond and either formation of BYI 02960-difluoroethyl-amino-furanone or conjugation with carbohydrates or oxidation of the methylene group to a carboxylic group
- oxidative degradation of the furanone moiety to an acetic acid group, followed either by conjugation with a carbohydrate or by further degradation of the moiety.

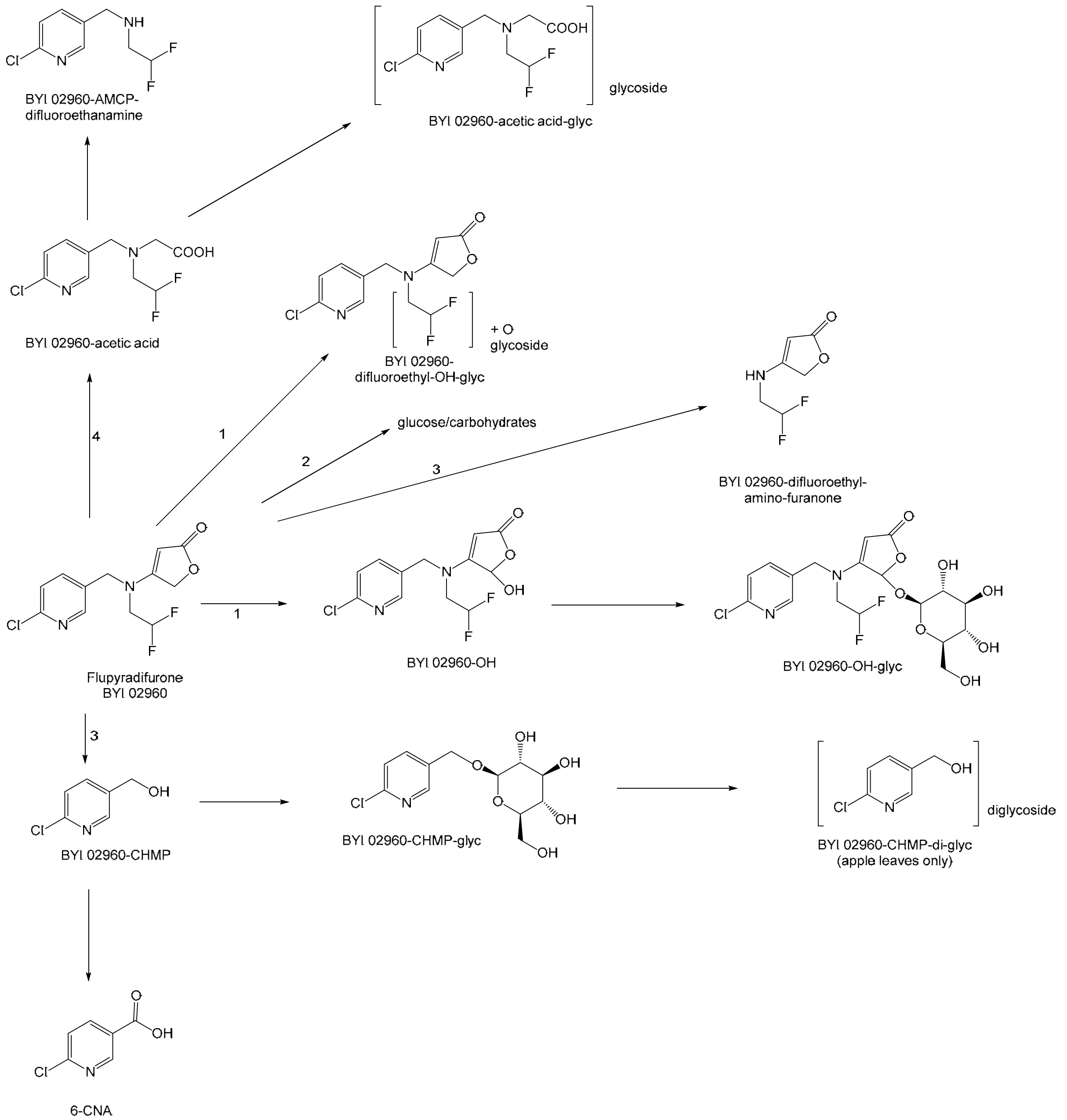


Figure 3 Metabolic pathway for flupyradifurone in apples

Cotton

Two different methods of application to cotton (variety: *Carmen*) were described. In one type of experiment, (single application experiment), five cotton plants were treated with [furanone-4-^{14}C]-BYI 02960 (Schmeling and Weber 2011a, MEF-11/392) or [pyridinylmethyl-^{14}C]-BYI 02960 (Schmeling and Weber 2011b, MEF-11/393) as an SL formulation at an actual application rate of 209 g ai/ha (furanone label) or 206 g ai/ha (pyridinylmethyl label), when the sixth true leaf was unfolded (BBCH 16).

In another type of experiment, one cotton plant was treated two times, once at an application rate of 209 g ai/ha at BBCH 15 for the furanone label and at 206 g ai/ha at BBCH 16 for the pyridinylmethyl label and a second time at an application rate of 176 g ai/ha at 14 days before harvest of the cotton bolls (furanone label) or 177 g ai/ha at 15 days before harvest of the cotton bolls (pyridinylmethyl label), when more than 90% of the bolls were open and approximately 50% to 70% of the leaves were discoloured or fallen, BBCH 95–97.

In both experiments undelinted cotton seeds, lint, and gin trash were harvested at maturity of the seeds (BBCH 99). The PHIs were 169 days for the single application (intermediate growth stage plant samples were also taken at 28 days) and 14 days for the double application.

Aliquots of all sample matrices were extracted three times with a mixture of acetonitrile/water (8:2; v/v) using a high speed blender. The cotton seed samples were additionally extracted with heptane prior to the first acetonitrile/water extraction step. The radioactivity in the extracts was determined by LSC and in the solids by combustion followed by LSC. For the furanone experiments the intermediate and the gin trash extracts were subjected to an SPE clean-up step, whereas the seed and the lint extracts were directly analysed by HPLC analysis after concentration. The intermediate, gin trash and seed extracts were subjected to a SPE clean-up step in the pyridinylmethyl experiments. The solids remaining after the conventional extraction steps of the gin trash samples were additionally submitted two times to microwave-assisted extraction with acetonitrile/water mixtures at increased temperatures (120 °C).

Residues in gin trash and lint from the double application were considerably higher compared to the single application experiment. Residues in seeds were not affected by the late second application. The TRR levels in cotton seeds in both the single and in the double application experiment were comparable: only 0.013 mg/kg equiv. and 0.016 mg/kg equiv. were observed, respectively, for the furanone label and 0.045 mg/kg equiv. and 0.068 mg/kg equiv. for the pyridinylmethyl label. Higher residues were detected in gin trash (2.767 and 2.344 mg/kg equiv.) and lint (4.993 and 8.846 mg/kg equiv.) in the double application experiment for the furanone and pyridinylmethyl labels respectively, where the last treatment was performed 14 or 15 days before harvest when more than 90% of the cotton bolls were open. In the single application experiment (169-day PHI), the TRR in gin trash accounted for 0.191 mg/kg equiv. and 0.310 mg/kg equiv. for the furanone and pyridinylmethyl labels respectively and the TRR in lint was < 0.01 mg/kg equiv. for both labelled compounds.

Table 11 TRRs in cotton matrices after treatment with [furanone-4-^{14}C]-BYI 02960 or [pyridinylmethyl-^{14}C]-BYI 02960

			[furanone-4-^{14}C]-flupyradifurone		[pyridinylmethyl-^{14}C]-flupyradifurone	
Matrix	Application Type	Growth stage	PHI (days)	TRR (mg/kg)	PHI (days)	TRR (mg/kg)
Intermediate	1 spray application, 209 g ai/ha (furanone label) or 206 g ai/ha (pyridinylmethyl label)	BBCH 22–24	28	12.39	28	14.15
Gin trash		BBCH 99	169	0.191	169	0.310
Lint			169	0.009	169	0.007
Seeds			169	0.013	169	0.045
Gin trash	2 spray applications, 209 g ai/ha at BBCH 15 and 176 g ai/ha at BBCH 95–97 (furanone label), 206 g ai/ha at BBCH 16 and 177 g ai/ha at BBCH 95–97 (pyridinylmethyl label)	BBCH 99	14	2.767	15	2.344
Lint			14	4.993	15	8.846
Seeds			14	0.016	15	0.068

Extraction efficiencies ranged from 23.4% and 28.3% of the TRR for seeds (single application experiment) to 96.6% and 99.2% for lint (double application experiment) for the furanone and pyridinylmethyl labels respectively. Due to the low radioactivity in seeds in the furanone label experiments, no further attempts were performed to analyse the extracts or to release additional radioactivity.

Parent compound and metabolites were analysed by HPLC. Identification was performed by HPLC and/or TLC co-chromatography with reference compounds, by comparison of HPLC profiles or by LC-MS/MS spectrometry. The distribution of parent and metabolites in cotton matrices is shown below in Tables 12 and 13.

Table 12 Distribution of parent and metabolites in cotton matrices after one or two foliar treatments with [furanone-4-^{14}C]-BYI 02960

Application	Single application				Double application			
Matrix	Intermediate plant		Gin trash		Gin trash		Lint	
TRR [mg/kg]	12.39		0.191		2.767		4.993	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction								
BYI 02960 (parent)	42.3	5.237	37.5	0.072	53.3	1.476	70.3	3.512
glyoxylic acid	–	–	0.9	0.002	1.6	0.044	0.2	0.009
acetic acid-glyc	8.6	1.068	–	–	2.2	0.062	–	–
OH-glyc / acetic acid	24.9	3.082	12.8	0.024	20.0	0.553	13.9	0.694
OH	–	–	12.0	0.023	0.6	0.016	–	–
bromo/chloro	0.7	0.089	0.6	0.001	2.3	0.063	1.6	0.078
Subtotal identified	76.5	9.476	63.7	0.122	80.0	2.214	86.0	4.292
unknown 1	2.2	0.268	2.6	0.005	4.9	0.135	3.8	0.190
unknown 2	–	–	–	–	0.5	0.014	0.4	0.018
unknown 3	1.0	0.122	–	–	0.4	0.012	0.9	0.043
unknown 4	–	–	0.6	0.001	2.0	0.056	–	–
unknown 5	6.6	0.823	–	–	0.8	0.023	0.3	0.014
unknown 6	–	–	–	–	0.5	0.013	–	–
unknown 7	2.5	0.310	–	–	0.2	0.005	0.6	0.028
unknown 8	–	–	–	–	–	–	0.5	0.024
unknown 9	–	–	–	–	–	–	2.7	0.135
unknown 10	–	–	–	–	0.3	0.009	1.2	0.062
unknown 11	1.1	0.141	–	–	–	–	–	–
unknown 12	–	–	–	–	–	–	0.3	0.016
Subtotal characterised	13.4	1.665	3.2	0.006	9.7	0.268	10.6	0.530
Total conventional extraction	89.9	11.14	66.9	0.128	89.7	2.482	96.6	4.822
Microwave extraction								
BYI 02960 (parent)	–	–	2.5	0.005	1.1	0.029	–	–
acetic acid-glyc	–	–	–	–	< 0.1	0.001	–	–
OH-glyc / acetic acid	–	–	2.9	0.006	0.8	0.023	–	–
OH	–	–	1.2	0.002	–	–	–	–
Subtotal identified	–	–	6.6	0.013	1.9	0.053	–	–
unknown 1	–	–	4.2	0.008	1.7	0.046	–	–
unknown 3	–	–	–	–	0.1	0.001	–	–
unknown 5	–	–	–	–	0.1	0.002	–	–
unknown 7	–	–	–	–	0.1	0.002	–	–
Subtotal characterised	–	–	4.2	0.008	1.8	0.051		
Total microwave extraction	–	–	10.9	0.021	3.8	0.104		
Total identified	76.5	9.476	70.3	0.134	81.9	2.268	86.0	4.292
Total characterised	13.4	1.665	7.4	0.014	11.5	0.319	10.6	0.530
Analysed extracts	89.9	11.141	77.7	0.148	93.4	2.586	96.6	4.822
Not analysed/ losses	0.4	0.053	2.6	0.005	2.3	0.065	< 0.1	< 0.001
Total extracted	90.3	11.19	80.3	0.153	95.8	2.651	96.6	4.822
Post-extraction solids (PES)	9.7	1.197	19.7	0.038	4.2	0.116	3.4	0.170
Accountability	100	12.39	100	0.191	100	2.767	100	4.993

Seeds (one application): TRR = 0.013 mg/kg, 23.4% conventional extraction, 76.6% PES

Seeds (two applications): TRR = 0.016 mg/kg, 57.8% conventional extraction, 42.2% PES

Table 13 Distribution of parent and metabolites in cotton matrices after one or two foliar treatments with [pyridinylmethyl-^{14}C]-BYI 02960

	Single Application						Double Application					
Matrix	Intermediate		gin trash		Seeds		gin trash		lint		Seeds	
TRR [mg/kg]	14.15		0.310		0.045		2.344		8.846		0.068	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction												
BYI 02960 (parent)	36.9	5.221	24.7	0.077	–	–	51.3	1.204	73.0	6.455	23.4	0.016
6-CNA	2.1	0.298	18.5	0.057	16.2	0.007	2.0	0.047	0.4	0.031	5.0	0.003
glyoxylic acid	1.5	0.209	2.1	0.007	–	–	1.5	0.035	0.2	0.015	–	–
acetic acid-glyc	6.4	0.899	–	–	–	–	3.6	0.085	–	–	–	–
OH-glyc / acetic acid	25.1	3.559	12.0	0.037	–	–	20.6	0.484	14.6	1.295	4.9	0.003
OH	1.2	0.168	13.4	0.042	–	–	1.3	0.030	–	–	–	–
bromo/chloro	0.5	0.064	–	–	–	–	2.1	0.049	1.6	0.140	–	–
Subtotal identified	73.6	10.42	70.7	0.219	16.2	0.007	82.6	1.935	89.7	7.936	33.3	0.023
unknown 1	–	–	2.5	0.008	5.7	0.003	0.4	0.01	–	–	–	–
unknown 2	–	–	0.6	0.002	–	–	0.2	0.005	–	–	–	–
unknown 3	1.4	0.196	–	–	–	–	0.1	0.004	–	–	–	–
unknown 4	2.8	0.403	3.4	0.01	–	–	2.2	0.052	–	–	–	–
unknown 5	1.1	0.159	1.0	0.003	–	–	1.3	0.031	0.3	0.027	–	–
unknown 6	6.6	0.935	0.6	0.002	–	–	–	–	0.6	0.051	–	–
unknown 7	3.9	0.551	–	–	–	–	–	–	0.7	0.064	–	–
unknown 8	–	–	–	–	–	–	–	–	4.9	0.432	–	–
unknown 9	–	–	–	–	–	–	–	–	1.5	0.129	–	–
unknown 10	1.9	0.270	–	–	–	–	0.4	0.009	0.3	0.023	–	–
unknown 11	1.9	0.273	1.2	0.004	–	–	1.4	0.033	0.8	0.070	5.2	0.003
unknown 12	0.9	0.130	0.6	0.002	–	–	–	–	0.5	0.040	–	–
Subtotal characterised	20.6	2.917	9.9	0.031	5.7	0.003	6.1	0.144	9.5	0.837	5.2	0.003
Total conventional extraction	94.2	13.34	80.6	0.250	21.9	0.01	88.7	2.079	99.2	8.774	38.5	0.026
Microwave extraction												
BYI 02960 (parent)	–	–	1.6	0.005	–	–	1.9	0.044	–	–	–	–
6-CNA	–	–	1.7	0.005	–	–	0.2	0.005	–	–	–	–
acetic acid-glyc	–	–	–	–	–	–	0.1	0.002	–	–	–	–
OH-glyc / acetic acid	–	–	1.7	0.005	–	–	1.8	0.042	–	–	–	–
OH	–	–	1.1	0.003	–	–			–	–	–	–
Subtotal identified	–	–	6.1	0.019	–	–	3.9	0.092	–	–	–	–
unknown 1	–	–	3.8	0.012	–	–	0.1	0.004	–	–	–	–
unknown 2	–	–	–	–	–	–	0.1	0.002	–	–	–	–
unknown 5	–	–	–	–	–	–	0.1	0.003	–	–	–	–
unknown 6	–	–	–	–	–	–	0.2	0.004	–	–	–	–
unknown 11	–	–	0.5	0.002	–	–	0.2	0.006	–	–	–	–
Subtotal characterised	–	–	4.3	0.013	–	–	0.8	0.018	–	–	–	–
Total microwave extraction	–	–	10.4	0.032	–	–	4.7	0.110	–	–	–	–
Total identified	73.6	10.42	76.8	0.238	16.2	0.007	86.5	2.028	89.7	7.936	33.3	0.023
Total characterised	20.6	2.917	14.2	0.044	5.7	0.003	6.9	0.162	9.5	0.837	5.2	0.003
Analysed extracts	94.2	13.336	91.0	0.282	21.9	0.01	93.4	2.190	99.2	8.774	38.5	0.026
Not analysed/losses	0.7	0.105	0.9	0.003	6.5	0.003	3.8	0.089	< 0.1	< 0.001	27.6	0.019
Total extracted	95.0	13.44	92.0	0.285	28.3	0.013	97.2	2.279	99.2	8.774	66.1	0.045
Post-extraction solids (PES)	5.0	0.713	8.0	0.025	71.7	0.032	2.8	0.065	0.8	0.073	33.9	0.023
Accountability	100	14.15	100	0.310	100	0.045	100	2.344	100	8.846	100.0	0.068

The main component of the extracted radioactivity in all cotton matrices from experiments with both labels, except for seeds of the single application experiment, was parent (23–73% TRRs), even after only one treatment with a long PHI (169 days). The fraction comprising the metabolites BYI 02960-OH-glyc and BYI 02960-acetic acid represented a major portion of the TRR in all sample matrices except for seeds (12–25% TRRs), while another major compound in gin trash of the first application experiment was identified as BYI 02960-OH (13.4% TRR). In seeds of the single application pyridinylmethyl label experiment, 6-CNA was the only component identified (16.2% TRR, 0.007 mg/kg equiv.). It was also observed in gin trash of the single application pyridinylmethyl label experiment (18.5% TRR, 0.057 mg/kg equiv.).

All other metabolites detected in gin trash, lint, seeds and in the intermediate plant material were minor metabolites, which did not exceed 10% of the TRR in either the single or the double application experiment.

A metabolic pathway for flupyradifurone in cotton was proposed. The pathway involved:

- hydroxylation of the methylene group of the furanone moiety followed by conjugation (*i.e.* glycosylation)
- oxidative degradation of the furanone moiety to an acetic acid group followed either by conjugation with a carbohydrate or by further oxidation
- halogenation (bromination/chlorination) of the furanone moiety
- cleavage of the pyridinylmethylamine bond followed by oxidation of the methylene group to 6-CNA.

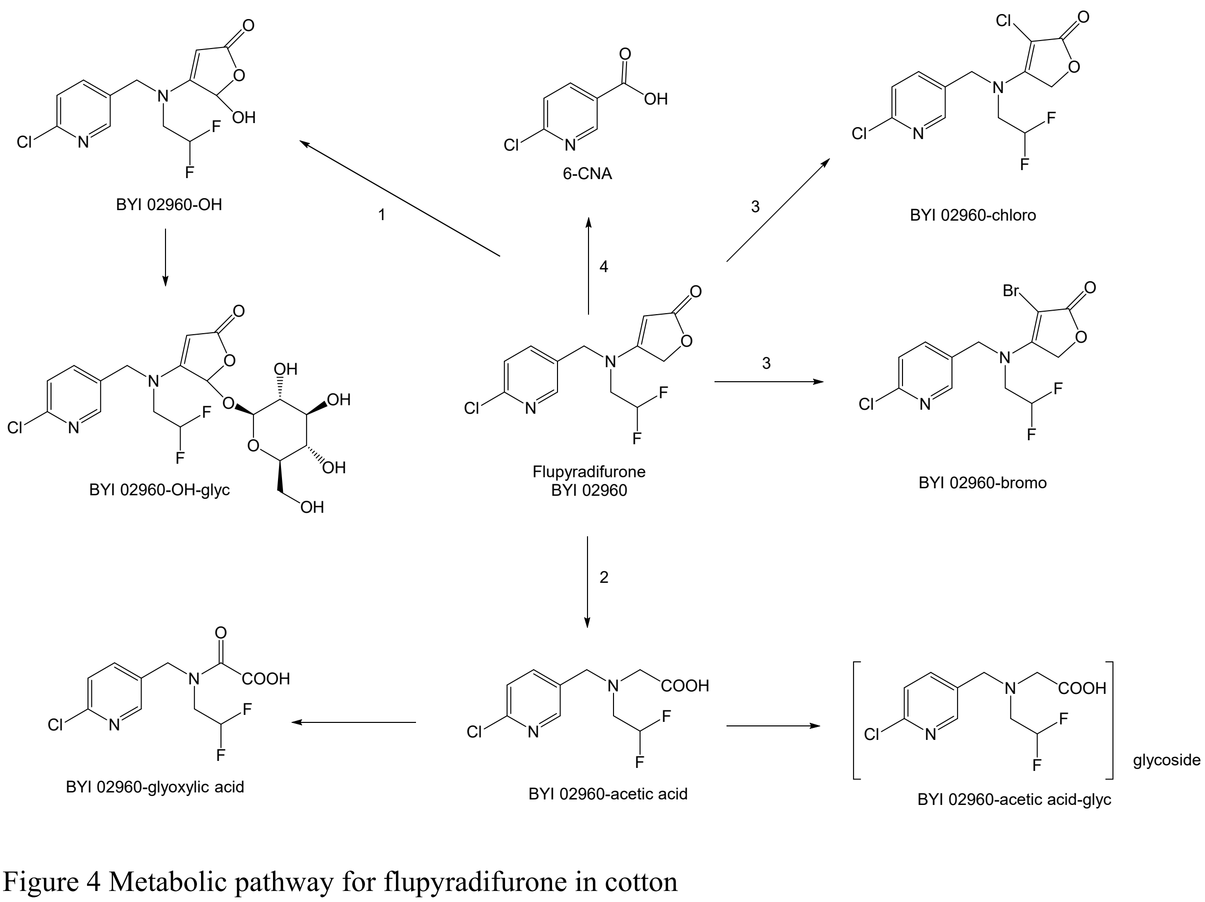


Figure 4 Metabolic pathway for flupyradifurone in cotton

Paddy rice

Two different methods of application to paddy rice (variety: *Nihonbare*) were described. One experiment consisted of a granular treatment (GR) with either [furanone-4-^{14}C]-BYI 02960 (Schmeling and Weber 2011c, MEF-11/058) or [pyridinylmethyl-^{14}C]-BYI 02960 (Schmeling and Weber 2011d, MEF-11/059) applied during the transplanting of the rice seedlings at an actual application rate of 409 g ai/ha (furanone label) or 434 g ai/ha (pyridinylmethyl label). The granules were equally distributed among the planting holes directly before the seedlings were placed and then the planting container was flooded with water. In the second experiment, [furanone-4-^{14}C]-BYI 02960 or [pyridinylmethyl-^{14}C]-BYI 02960 was formulated as SL 200 and applied twice as a foliar treatment (SP) onto plants as well as the water surface. The first spray application took place directly after transplanting of the rice seedlings at a rate of 175 g ai/ha (furanone label) or 178 g ai/ha (pyridinylmethyl label) and the second approximately one month before harvest at a rate of 240 g ai/ha (furanone label) or 236 g ai/ha (pyridinylmethyl label). In both experiments, the rice (kernels, husks and straw) was harvested at maturity (BBCH 89–92). The PHI for the granular application was 127 days while the PHI for the foliar spray application was 29 days.

Prior to extraction, the homogenised sample materials were soaked in acetonitrile/water (8:2; v/v) for one night. Three extraction steps were conducted with the same solvent system using a high-speed blender. The radioactivity in the extracts was determined by LSC and in the solids by combustion followed by LSC. The extracts of the husks and straw were subjected to a clean-up step using an SPE cartridge prior to HPLC analysis. Extracts of kernels were concentrated and analysed by HPLC without any additional purification step. The post-extraction solids (PES) of kernels and straw of both experiments were subjected to an exhaustive extraction procedure. The solids were extracted in a first step with acetonitrile/water (8:2; v/v) and in a second step with acetonitrile/water (1:1; v/v) using a microwave-assisted extraction (120 °C for 15 min.). Solids of rice kernels were subjected to an additional third microwave extraction step with sodium chloride /water (1/99; w/v). The microwave NaCl extract of kernels of the spray application experiment was treated with the enzyme diastase, to further characterise the radioactivity incorporated into starch components.

The TRR levels in the samples from the single granular treatment were lower compared to those of the double foliar treatment. The TRR in RAC rice kernels accounted for 0.140 mg/kg equiv. (furanone label) and 0.05 mg/kg equiv. (pyridinylmethyl label) after granular application and 0.659 mg/kg equiv. (furanone label) and 0.620 mg/kg equiv. (pyridinylmethyl label) after foliar application. Straw showed a TRR of 2.879 mg/kg equiv. (furanone label) and 3.280 mg/kg equiv. (pyridinylmethyl label) after granular application, whereas the TRRs were 19.89 mg/kg equiv. (furanone label) and 24.73 mg/kg equiv. (pyridinylmethyl label) in straw from the foliar experiment.

Table 14 TRRs in rice matrices after treatment with [furanone-4-^{14}C]-BYI 02960 or [pyridinylmethyl-^{14}C]-BYI 02960

			[furanone-4-^{14}C]-flupyradifurone		[pyridinylmethyl-^{14}C]-flupyradifurone	
Matrix	Application Type	Harvest growth stage	PHI (days)	TRR (mg ai. equiv./kg)	PHI (days)	TRR (mg ai. equiv./kg)
Rice kernels	1 granular application at transplant, 409 g ai/ha (furanone label) or 434 g ai/ha (pyridinylmethyl label)	BBCH 89–92	127	0.140	127	0.05
Rice husks			127	1.404	127	1.602
Rice straw			127	2.879	127	3.280
Rice kernels	2 foliar applications, 175 g ai/ha at BBCH 13–15 and 240 g ai/ha at BBCH 87–89 (furanone label) or 178 g ai/ha/ at BBCH 13–15 and 236 g ai/ha at BBCH 87–89 (pyridinylmethyl label)	BBCH 89–92	29	0.659	29	0.620
Rice husks			29	24.10	29	23.96
Rice straw			29	19.89	29	24.73

Parent compound and metabolites were analysed by HPLC. Identification was performed by co-chromatography with reference compounds, by comparison of HPLC profiles. The distribution of parent and metabolites in rice matrices is shown below.

Table 15 Distribution of parent and metabolites in rice matrices after a single granular application or two foliar applications of [furanone-4-^{14}C]-BYI 02960

Application	Granular application						Foliar application					
Matrix	Kernels		Husks		Straw		Kernels		Husks		Straw	
TRR [mg/kg]	0.140		1.404		2.879		0.659		24.10		19.89	
(BYI 02960-) Equivalents	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Conventional extraction												
BYI 02960 (parent)	17.5	0.024	72.3	1.016	57.6	1.658	53.3	0.351	74.6	17.97	52.9	10.53
glucose/carbohydrates	1.7	0.002	1.0	0.014	3.1	0.088	1.9	0.012	2.1	0.497	2.0	0.401
glyoxylic acid	–	–	–	–	1.7	0.05	–	–	–	–	2.0	0.393
acetic acid-glyc	–	–	–	–	–	–	0.4	0.003	0.2	0.054	1.7	0.329
acetic acid	–	–	0.4	0.006	1.8	0.051	5.9	0.039	7.0	1.693	7.2	1.432
OH	–	–	–	–	0.8	0.024	–	–	–	–	0.4	0.081
bromo/chloro	–	–	0.6	0.008	10.6	0.306	1.7	0.011	1.6	0.389	10.1	2.016
Subtotal identified	19.2	0.027	74.3	1.043	75.6	2.176	63.1	0.416	85.5	20.61	76.3	15.18
unknown 1	–	–	–	–	0.8	0.023	–	–	0.4	0.092	0.6	0.126
unknown 2	–	–	–	–	–	–	–	–	–	–	0.3	0.053
unknown 3	–	–	1.3	0.019	1.1	0.031	0.9	0.006	0.6	0.138	0.6	0.123
unknown 4	–	–	–	–	–	–	–	–	0.1	0.033	0.1	0.025
unknown 5	–	–	–	–	1.1	0.033	–	–	–	–	0.9	0.173
unknown 6	–	–	–	–	–	–	0.9	0.006	0.4	0.103	1.2	0.229
unknown 7	–	–	–	–	0.4	0.012	0.8	0.005	2.5	0.608	0.2	0.044
unknown 9	–	–	–	–	–	–	–	–	0.9	0.211	3.1	0.609
Subtotal characterised	–	–	1.3	0.019	3.4	0.099	2.6	0.017	4.9	1.185	7.0	1.382
Total conventional extraction	19.2	0.027	75.6	1.062	79.0	2.275	65.8	0.433	90.4	21.79	83.3	16.56
Microwave extraction												
BYI 02960 (parent)	5.6	0.008			6.4	0.183	3.3	0.021			3.6	0.720
glucose/carbohydrates	–	–			2.3	0.066	1.7	0.011			1.4	0.287
glyoxylic acid	–	–			0.2	0.007	–	–			0.1	0.023
acetic acid-glyc	–	–			–	–	–	–			0.7	0.132
acetic acid	-	-			0.2	0.007	0.3	0.002			0.8	0.149
OH	-	-			0.2	0.005	–	–			0.1	0.025
bromo/chloro	–	–			0.8	0.022	–	–			0.6	0.116
Subtotal identified	5.6	0.008			10.1	0.290	5.2	0.034			7.3	1.451
unknown 1	–	–					–	–			0.3	0.062
unknown 3	–	–			0.4	0.011	–	–			0.1	0.028
unknown 4	–	–			0.3	0.007					–	–
unknown 5	–	–			0.2	0.005	–	–			0.2	0.033
unknown 8	–	–			–	–	–	–			0.2	0.031
unknown 9	–	–			–	–	–	–			1.8	0.359
Subtotal characterized	–	–			0.8	0.023	–	–			2.6	0.513
Total microwave extr.	5.6	0.008	–	–	10.9	0.313	5.2	0.034	–	–	9.9	1.964
Exhaustive NaCl extraction [a]												
organic phase	0.8	0.001					0.9	0.006				
aqueous phase	28.6	0.040					13.7	0.090				
glucose/carbohydrates	25.2	0.035										
Subtotal identified	25.2	0.035					–	–				
unknown 2	3.4	0.005										
Subtotal	4.2	0.006					14.6	0.096				

Application	Granular application						Foliar application					
Matrix	Kernels		Husks		Straw		Kernels		Husks		Straw	
TRR [mg/kg]	0.140		1.404		2.879		0.659		24.10		19.89	
(BYI 02960-) Equivalents	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
characterized												
Total exh. NaCl extraction	29.4	0.041	–	–	–	–	14.6	0.096	–	–	–	–
Total identified	50.1	0.070	74.3	1.043	85.7	2.466	68.4	0.450	85.5	20.61	83.6	16.63
Total characterised	4.2	0.006	1.3	0.019	4.2	0.122	17.2	0.113	4.9	1.185	9.5	1.895
Analysed extract(s)	54.3	0.076	75.6	1.062	89.9	2.588	85.6	0.564	90.4	21.79	93.1	18.526
Not analysed/ losses	14.4	0.020	n.q.	n.q.	1.3	0.037	1.6	0.01	0.5	0.127	0.5	0.094
Total extracted	68.7	0.096	75.6	1.062	91.2	2.625	87.2	0.574	90.9	21.92	93.6	18.62
Post-extraction solids (PES)	31.3	0.044	24.4	0.342	8.8	0.254	12.8	0.085	9.1	2.182	6.4	1.271
Accountability	100	0.140	100	1.404	100	2.879	100	0.659	100	24.10	100	19.89

[a] Kernels of granular application extracted with NaCl only, kernels of spray application with NaCl + diastase

Table 16 Distribution of parent and metabolites in rice matrices after a single granular application or two foliar applications of [pyridinylmethyl-^{14}C]-BYI 02960

Application	Granular application						Foliar application					
Matrix	Kernels		Husks		Straw		Kernels		Husks		Straw	
TRR [mg/kg]	0.05		1.602		3.280		0.620		23.96		24.73	
(BYI 02960-) equivalents	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Conventional extraction												
BYI 02960 (parent)	62.8	0.032	77.7	1.244	56.2	1.842	72.0	0.447	77.3	18.53	56.9	14.07
6-CNA	–	–	0.5	0.009	2.9	0.093	2.1	0.013	0.4	0.107	0.9	0.227
glyoxylic acid	–	–	–	–	0.2	0.007	0.4	0.003	–	–	2.0	0.505
acetic acid-glyc	–	–	–	–	–	–	0.6	0.003	0.2	0.048	1.2	0.308
acetic acid	–	–	–	–	1.8	0.058	7.8	0.048	6.5	1.548	6.6	1.631
OH	–	–	–	–	1.1	0.037	0.4	0.002	–	–	0.6	0.146
bromo/chloro	–	–	0.6	0.01	11.7	0.385	1.5	0.009	1.2	0.295	7.9	1.955
Subtotal identified	62.8	0.032	78.8	1.263	73.9	2.423	84.8	0.526	85.7	20.52	76.2	18.84
unknown 1	–	–	–	–	–	–	–	–	–	–	0.1	0.033
unknown 2	–	–	–	–	–	–	–	–	–	–	0.1	0.031
unknown 3	–	–	–	–	0.7	0.023	–	–	–	–	0.3	0.079
unknown 4	–	–	0.8	0.012	0.7	0.022	–	–	0.4	0.085	0.3	0.068
unknown 5	–	–	–	–	–	–	–	–	–	–	0.2	0.047
unknown 6	–	–	–	–	0.5	0.016	–	–	0.2	0.044	0.2	0.049
unknown 7	–	–	–	–	1.4	0.047	–	–	–	–	0.1	0.022
unknown 8	–	–	–	–	–	–	–	–	–	–	0.2	0.042
unknown 9	–	–	–	–	0.3	0.011	0.5	0.003	0.4	0.090	0.8	0.188
unknown 10	–	–	–	–	–	–	1.2	0.008	1.7	0.401	0.3	0.068
unknown 11	–	–	–	–	0.4	0.015	–	–	–	–	0.3	0.070
unknown 12	–	–	–	–	0.8	0.027	–	–	0.2	0.044	0.7	0.171
unknown 13	–	–	–	–	0.9	0.031	1.1	0.007	0.4	0.106	1.1	0.279
unknown 14	–	–	–	–	1.0	0.033	0.8	0.005	0.9	0.221	2.9	0.718
Subtotal characterised	–	–	0.8	0.012	6.9	0.225	3.6	0.022	4.1	0.990	7.5	1.864
Total conventional extraction	62.8	0.032	79.6	1.275	80.7	2.648	88.4	0.548	89.8	21.51	83.7	20.701
Microwave extraction												
BYI 02960 (parent)	6.8	0.003	–	–	3.7	0.122	3.2	0.020	–	–	3.9	0.958
6-CNA	4.7	0.002	–	–	1.0	0.031	0.9	0.006	–	–	0.3	0.073

Application	Granular application						Foliar application					
Matrix	Kernels		Husks		Straw		Kernels		Husks		Straw	
TRR [mg/kg]	0.05		1.602		3.280		0.620		23.96		24.73	
(BYI 02960-) equivalents	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
glyoxylic acid	–	–	–	–	–	–	–	–	–	–	0.2	0.052
acetic acid-glyc	–	–	–	–	–	–	–	–	–	–	0.7	0.166
acetic acid	–	–	–	–	0.2	0.006	–	–	–	–	0.7	0.183
OH	–	–	–	–	0.1	0.004	–	–	–	–	0.2	0.043
bromo/chloro	–	–	–	–	0.5	0.018	–	–	–	–	0.6	0.139
Subtotal identified	11.5	0.005	–	–	5.5	0.181	4.1	0.026	–	–	6.5	1.615
unknown 1	–	–	–	–	–	–	–	–	–	–	< 0.1	0.011
unknown 2	–	–	–	–	0.2	0.006	–	–	–	–	0.1	0.036
unknown 3	–	–	–	–	0.1	0.003	–	–	–	–	–	–
unknown 5	–	–	–	–	0.2	0.005	–	–	–	–	–	–
unknown 6	–	–	–	–	0.1	0.003	–	–	–	–	–	–
unknown 12	–	–	–	–	0.1	0.005	–	–	–	–	–	–
unknown 13	–	–	–	–	0.8	0.025	0.8	0.005	–	–	1.1	0.276
unknown 14	–	–	–	–	0.3	0.01	–	–	–	–	1.8	0.435
Subtotal characterised	–	–	–	–	1.7	0.056	0.8	0.005	–	–	3.1	0.758
Total microwave extr.	11.5	0.006	–	–	7.2	0.237	4.9	0.031	–	–	9.6	2.374
Exhaustive NaCl extraction [a]												
Subtotal identified							–	–				
organic phase							1.2	0.007				
aqueous phase							1.8	0.011				
Subtotal characterised							3.1	0.021				
Total NaCl extr.							3.1	0.021	–	–	–	–
Total identified	74.3	0.037	78.8	1.263	79.4	2.603	88.9	0.552	85.7	20.52	82.7	20.46
Total characterised	–	–	0.8	0.012	8.6	0.281	7.4	0.045	4.1	0.990	10.6	2.622
Analysed extract(s)	74.3	0.037	79.6	1.275	87.9	2.884	96.4	0.597	89.8	21.51	93.3	23.08
Not analysed/ losses	n.q.	n.q.	n.q.	n.q.	1.4	0.046	0.9	0.007	0.5	0.111	0.3	0.075
Total extracted	74.3	0.037	79.6	1.275	89.3	2.930	97.3	0.604	90.3	21.63	93.6	23.16
Post-extraction solids	25.7	0.013	20.4	0.327	10.7	0.350	2.7	0.017	9.7	2.332	6.4	1.573
Accountability	100	0.05	100	1.602	100	3.280	100	0.620	100	23.96	100	24.73

[a] Kernels of granular application extracted with NaCl only, kernels of spray application with NaCl + diastase

The radioactive residues were efficiently extracted with acetonitrile/water mixtures. When necessary, additional exhaustive extraction steps were applied to the solids of the conventional extraction. Extraction efficiencies for the furanone label ranged from 68.7% of the TRR for kernels (GR) to 93.6% for straw (SP) after exhaustive extraction while for the pyridinylmethyl label they ranged from 74.3% of the TRR for kernels (GR) to 97.3% for kernels (SP) after exhaustive extraction.

The main component of the extracted radioactivity was parent in all rice matrices, except for rice kernels after granular application for the furanone label, where the main metabolite was the natural compound glucose/carbohydrates. In rice straw for both labels, BYI 02960-bromo was present at approximately 8–11% of the TRR. It co-eluted with BYI 02960-chloro, which accounted for significant lower concentrations. It was assumed that halogenation of the furanone moiety of the active substance occurred in the paddy soil and the metabolite was taken up by the rice plants. All other metabolites detected in the sample matrices of rice were minor metabolites which did not exceed 10% of the TRR in both application experiments.

A metabolic pathway for [furanone-4-^{14}C]-BYI 02960 in rice was proposed. The pathway involved:

- hydroxylation of the methylene group of the furanone moiety, complete degradation of the furanone moiety and incorporation of carbon atoms into the natural compound pool (*i.e.* glucose/ carbohydrates)
- oxidative degradation of the furanone moiety to an acetic acid group followed by conjugation with a carbohydrate or further oxidation
- halogenation (mostly bromination, to a minor extent chlorination) of the furanone moiety and cleavage of the pyridinylmethylamine bond followed by oxidation of the methylene group to 6-CNA. It was speculated that halogenation of the furanone moiety of the active substance occurred in the paddy soil/ sediment.

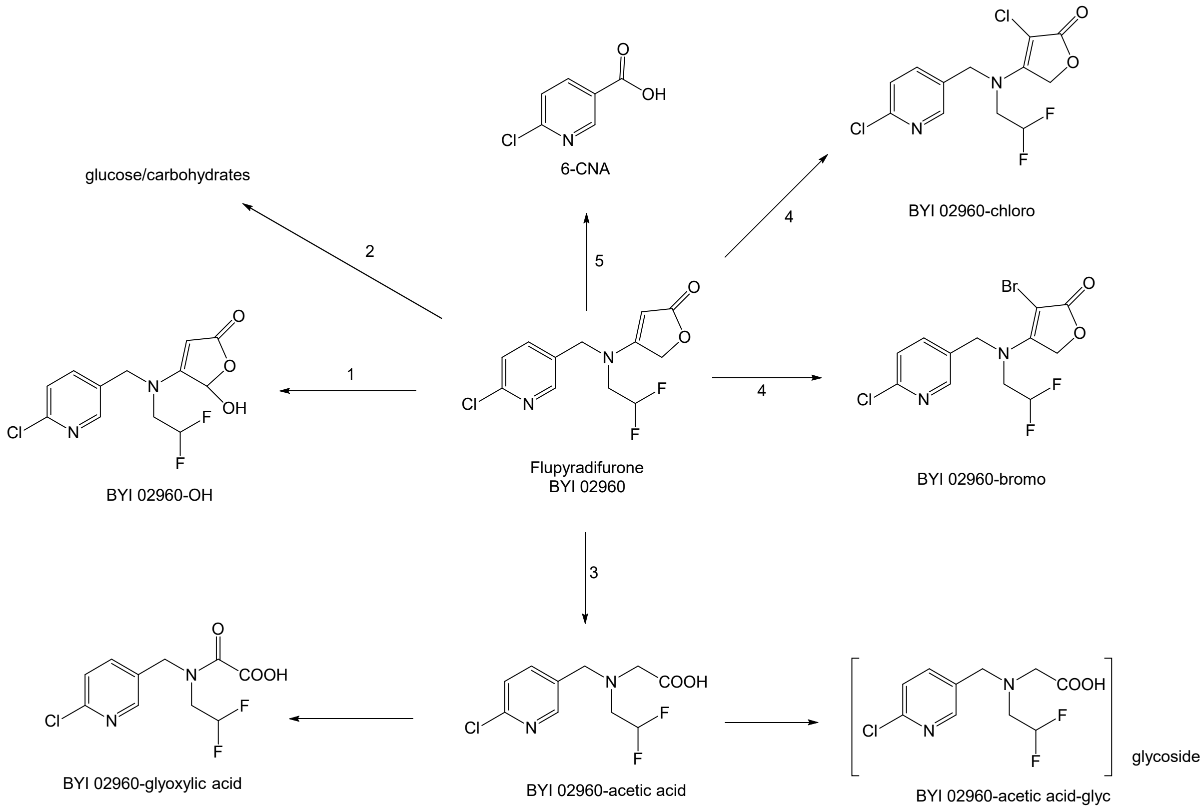


Figure 5 Metabolic pathway for flupyradifurone in rice

Analysis for difluoroacetic acid (DFA)

A study was conducted to determine the fate of the difluoroethane moiety of parent BYI 02960 by determination of the non-radiolabelled difluoroacetic acid content of extracts from the plant metabolism studies (apples, potatoes, cotton and rice) conducted with either [furanone-4-^{14}C]- or [pyridinylmethyl-^{14}C]-BYI 02960 (Schoening and Ruhl, 2012, MR-11/050). (Only one tomato metabolism study was performed with [ethyl-1-^{14}C]-BYI 02960, precursor to radiolabelled difluoroacetic acid). Samples were extracted in their respective metabolism studies. Results are summarized below in Table 17.

Table 17 Summary of difluoroacetic acid residues in crop matrices from the BYI 02960 plant metabolism studies

Metabolism study	Crop	Sample material	Use pattern	Difluoroacetic acid residues [mg/kg]	Reference
Metabolism of [furanone-4-^{14}C]-BYI 02960 in apples	Apple	fruits	one foliar spray application at BBCH 69, 86 g ai/(ha × m CH)	0.23	Justus 2011, MEF-11/499
		leaves		0.62	
		fruits	two foliar spray applications, at BBCH 69 and 14 days PHI, 2 x 86 g ai/(ha × m CH)	0.04	
		leaves		0.45	
Metabolism of [pyridinylmethyl-^{14}C]-BYI 02960 in potatoes	Potato	tuber	seed piece treatment at planting (BBCH 03), 10.0 g ai/dt	0.13	Justus 2011, MEF-10/710
			in-furrow spray application at planting (BBCH 03), 626 g ai/ha	0.18	
Metabolism of [pyridinylmethyl-^{14}C]-BYI 02960 in Cotton after Spray Application	Cotton	gin trash	one spray application, 206 g ai/ha (at BBCH 16)	0.04	(Schmelling and Weber 2011, MEF-11/393)
		seeds		0.03	
		gin trash	two spray applications, 206 g ai/ha (at BBCH 16), 177 g ai/ha (at BBCH 95–97)	0.02	
		seeds		0.02	
Metabolism of [pyridinylmethyl-^{14}C]-BYI 02960 in paddy rice	Rice	straw	two spray applications onto the plants at different growth stages, 178 g ai/ha (at BBCH 13–15) 236 g ai/ha (at BBCH 87–89)	0.39	(Schmelling and Weber 2011, MEF-11/059)
		husk		0.46	
		grains		0.08	
		straw	one granular application at the time of transplanting, 434 g ai/ha (at BBCH 13–15)	0.12	
		husk		0.20	
		grains		0.02	

Residue analysis was performed using HPLC coupled with electrospray and MS/MS detection per Method 01304. Significant levels of difluoroacetic acid (DFA) were detected. The application technique did not significantly influence the level, with residues of a similar order of magnitude after soil or foliar application. High difluoroacetic acid concentrations after foliar spray application indicate that this metabolite is also formed in plants and not only in soil.

Difluoroacetic acid (DFA) represents a significant proportion of the residue in all edible matrices of primary crops when considering the results of the studies conducted with [^{14}C]-BYI 02960. However, a direct comparison of the DFA concentrations, expressed as BYI 02960 equivalents, to the TRR is not appropriate as the DFA determination is for a non-radiolabelled degradation/metabolism molecule.

Summary of plant metabolism

Metabolism in primary crops was seen to be similar in all plant groups investigated. In the studies using [furanone-4-^{14}C]-BYI 02960 and [pyridinylmethyl-^{14}C]-BYI 02960, flupyradifurone was consistently observed to be the major component of the radioactive residues, accounting for approximately 23 to 88% TRR in all plant parts analysed, except for the single application to apple fruits using the furanone label.

As well as flupyradifurone, the following significant metabolites were identified in different plant matrices, the conjugate flupyradifurone-hydroxy-glycoside, up to 36% in apple leaves, the conjugate CHMP-diglycoside, up to 37% TRR (0.05 mg/kg equiv.) in tomato fruits and the metabolite 6-CNA in the range of 13–22% TRR in tomato fruit, potato tuber and cotton seed at approximately 0.02 mg/kg equiv., both resulting from the cleavage of the molecule at the ethylamine bond and containing the pyridinyl moiety. In contrast metabolites containing the furanone moiety were almost not detected and the radioactivity in the [^{14}C]furanone studies was mostly recovered as incorporated in natural glycoside and carbohydrate components, indicating an extensive degradation of the furanone counterpart.

The study conducted on tomato with the ^{14}C labelling on the difluoroethyl amino group showed that, following soil drench application, significant proportions (87% TRR) and levels (0.17 mg/kg equiv.) of difluoroacetic acid (DFA) are present in tomato fruits. Samples from the other radiolabelled studies were therefore re-analysed for non-radiolabelled DFA and residues, expressed as DFA equivalent, were measured in the range of 0.02–0.23 mg/kg in apple fruits, potato tuber, cotton seed and rice grain, irrespective of mode of application.

Confined rotational crop studies

Studies were conducted to investigate the metabolism of flupyradifurone in the representative rotational crops wheat, Swiss chard and turnips from three consecutive rotations using either [furanone-4-^{14}C]-BYI 02960 (Klempner 2011, MEF-11/365) or [pyridinylmethyl-^{14}C]-BYI 02960 (Breuer-Rehm 2011, MEF-10/892) formulated as an SL 300 and sprayed onto the soil of a planting container (approximately 1 m^2). The actual application rate corresponded to 436 g ai/ha for the furanone label and 433 g ai/ha for the pyridinylmethyl label. The crops were each sown at 29, 135 and 296 days after the soil application, representing the first, second and third rotation.

Raw agricultural commodities (RAC) sampled included the immature samples of forage and hay from wheat, and the immature samples from Swiss chard. All other samples (wheat straw and wheat grain, Swiss chard, turnip leaves and turnip roots) were harvested in each rotation at maturity.

An aliquot of each homogenised RAC was extracted three or four times with acetonitrile/water (8:2, v/v). The extracts were combined, purified using a pre-conditioned SPE RP $_{18}$ cartridge, concentrated, and analysed by HPLC. The radioactivity in the extracts was determined by LSC and in the solids by combustion followed by LSC.

If needed, solids were further extracted exhaustively using microwave conditions. The solids of the microwave extraction step from wheat straw were subjected to a treatment with sodium chloride solution (2 hours at 100 °C under microwave assistance), a diastase incubation (approx. 20 hours or 6 hours at 26 °C for the furanone label and pyridinylmethyl labels respectively), a treatment with EDTA solution (3 h or 1 hr at 100 °C under microwave assistance for the furanone and pyridinylmethyl labels respectively) and a cellulase treatment (approx. 20 hours or 12 hours at 40 °C for the furanone and pyridinylmethyl labels respectively) at adjusted temperatures to break down the plant cell walls and liberate residues bound to cell walls or in the cells.

For the furanone label the remaining solids of the sequential extraction were extracted finally in two steps with a 5 N HCl and a 5 N NaOH solution (each 2 hours at 100 °C). Similarly post extraction solids of wheat grain from the 1st and 2nd rotation were subjected to three subsequent exhaustive extraction steps: Microwave extraction with acetonitrile/water at 100 °C followed by a digestion step with diastase (20 h at 26 °C) and subsequent microwave extraction with a sodium chloride solution (2 hours at 100 °C).

For the pyridinylmethyl label the remaining solids of the sequential extraction were extracted finally in two steps with a 5 N HCl and a 0.1 N NaOH solution. Post-extraction solids of grains (1st rotation) were subjected to four subsequent exhaustive extraction steps under microwave conditions at increased temperature. Extraction was performed with acetonitrile/water mixtures, 0.1 N HCl and 0.1 N NaOH.

Parent compound and metabolites in the extracts were analysed by reversed phase HPLC coupled to a radioactivity detector with a glass scintillator cell. TRRs in the crops from the three different rotations are summarized below in Table 18. TRR values of all RACs declined significantly from the first to the third rotation. Highest residues were detected in the non-edible commodities wheat straw and wheat hay. The TRR exceeded 0.01 mg/kg in all commodities except turnip roots from the 3rd rotation.

Table 18 TRRs in the different RACs of the three rotations after soil application of [furanone-4-^{14}C] BYI 02960 or [pyridinylmethyl-^{14}C] BYI 02960

TRR [mg/kg]	Wheat				Swiss chard		Turnips	
[furanone-4-^{14}C] BYI 02960								
	forage	hay	straw	grain	imm.	mature	leaves	roots
1st rotation 29 day PBI	0.783	2.003	6.290	0.478	0.848	0.871	0.679	0.074
2nd rotation 135 day PBI	0.193	1.081	1.519	0.103	0.311	0.263	0.158	0.014
3rd rotation 296 day PBI	0.111	0.254	0.462	0.047	0.180	0.152	0.090	0.008
[pyridinylmethyl-^{14}C] BYI 02960								
	forage	hay	straw	grain	imm.	mature	leaves	roots
1st rotation 29 day PBI	1.407	2.409	9.015	0.177	1.358	1.483	0.815	0.072
2nd rotation 135 day PBI	0.308	1.009	2.148	0.057	0.332	0.438	0.230	0.022
3rd rotation 296 day PBI	0.117	0.321	0.491	0.017	0.135	0.130	0.083	0.008

Radioactive residues from the furanone label were efficiently extracted from all commodities of all rotations with acetonitrile/water mixtures, except for wheat grain and wheat straw (only 14.6% to 20.4% and 70.3% to 77.6% of the TRR were detected after conventional extraction, respectively). Exhaustive extraction steps released an additional portion of 68–73% of the TRR from the solids of grains and about 21–27% of the TRR from the solids of straw resulting in total extraction efficiencies ranging between 83% and 98% of the TRR.

The radioactive residues from the pyridinylmethyl label were efficiently extracted from all commodities of all rotations with acetonitrile/water mixtures, except for wheat grain where only 35.4% to 52.2% of the TRR was detected after conventional extraction. For wheat grain, exhaustive extraction released additional high amounts of radioactivity from the post-extraction solids. The extracted portions increased from 52.2% to 83.8% of the TRR (1st rotation) and from 35.4% to 89.4% of the TRR (2nd rotation). For the other wheat matrices, the additionally extracted portions ranged between 3.5% and 20.5% of the TRR depending on the from the furanone label exhaustive extraction steps applied.

Distribution of parent and metabolites in the RACs for each rotation from use of the furanone label are summarized below in Tables 19–24.

Table 19 Distribution of parent compound and metabolites in wheat matrices after 29 day plant back interval (1st rotation), [furanone-4-^{14}C]BYI 02960 label

1st Rotation	wheat forage		wheat hay		wheat straw		wheat grains	
TRR [mg/kg]	0.783		2.003		6.290		0.478	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction								
BYI 02960 (parent)	46.6	0.365	33.6	0.672	33.9	2.135	0.4	0.002
glucose/carbohydrates	–	–	–	–	–	–	–	–
amino-furanone	1.9	0.015	3.8	0.076	1.9	0.120	0.5	0.002
mercapto-lactic acid	1.6	0.013	0.4	0.008	1.1	0.068	–	–
bromo-amino-furanone	2.0	0.016	1.6	0.033	2.6	0.161	–	–
difluoroethyl-amino-furanone-OH-glyc	3.2	0.025	5.8	0.116	3.5	0.217	–	–
difluoroethyl-amino-furanone	9.9	0.077	10.2	0.205	5.3	0.333	–	–
glyoxylic acid	15.8	0.124	11.3	0.227	14.7	0.925	5.1	0.024
acetic acid-glyc	–	–	1.2	0.024	0.7	0.043	–	–
acetic acid	1.7	0.013	1.6	0.031	1.7	0.106	0.6	0.003
OH-glyc	3.6	0.028	3.4	0.067	3.6	0.228	1.4	0.007
OH	1.3	0.01	1.9	0.038	2.3	0.147	2.3	0.011
bromo / chloro	0.3	0.003	–	–	0.2	0.011	–	–

1st Rotation	wheat forage		wheat hay		wheat straw		wheat grains	
TRR [mg/kg]	0.783		2.003		6.290		0.478	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Subtotal identified	88.0	0.689	74.7	1.496	71.4	4.493	10.3	0.049
unknown 1a[a]	1.5	0.012	3.7	0.075	3.6	0.227	2.3	0.011
unknown 2	–	–	–	–	0.3	0.018	–	–
unknown 3	–	–	–	–	0.1	0.008	–	–
unknown 4	–	–	–	–	0.2	0.011	–	–
unknown 5	–	–	–	–	0.2	0.01	–	–
unknown 6	–	–	–	–	0.3	0.020	–	–
unknown 7	–	–	0.3	0.007	0.5	0.032	–	–
unknown 9	–	–	–	–	–	–	1.2	0.006
unknown 10	–	–	–	–	–	–	–	–
unknown 11	–	–	–	–	–	–	0.7	0.003
unknown 16	–	–	–	–	0.3	0.022	–	–
unknown 17	–	–	2.0	0.041	–	–	–	–
Subtotal characterised	1.5	0.012	6.1	0.122	5.5	0.347	4.2	0.020
Total conventional extr.	89.5	0.701	80.8	1.619	76.9	4.840	14.5	0.069
1st Rotation	wheat forage		wheat hay		wheat straw		wheat grains	
Microwave extraction I (ACN/water)								
BYI 02960 (parent)					1.6	0.102		
amino-furanone					0.2	0.013		
bromo-amino-furanone					0.2	0.011		
difluoroethyl-amino-furanone-OH-glyc					0.2	0.012		
difluoroethyl-amino-furanone					0.2	0.013		
glyoxylic acid					0.5	0.029		
acetic acid					0.1	0.003		
OH-glyc					0.1	0.007		
OH					0.1	0.007		
Subtotal identified					3.1	0.196		
unknown 1a[a]					1.4	0.085		
Subtotal characterised					1.4	0.085		
Total microwave extr. I					4.5	0.282		
Microwave extraction II (1% NaCl)								
BYI 02960 (parent comp.)					2.0	0.124		
amino-furanone					0.7	0.044		
difluoroethyl-amino-furanone					0.3	0.019		
glyoxylic acid					0.1	0.008		
acetic acid					< 0.1	0.003		
OH-glyc					0.1	0.006		
OH					0.1	0.006		
Subtotal identified					3.3	0.209		
unknown 1a[a]					1.4	0.086		
Subtotal characterised					1.4	0.086		
Total 1% NaCl extraction					4.7	0.295		
Diastase digestion								
BYI 02960 (parent comp.)					0.7	0.042	–	–
glucose/carbohydrates					–	–	70.5	0.338
amino–furanone					0.3	0.016	–	–
difluoroethyl-amino-furanone					0.1	0.009	–	–
glyoxylic acid					0.1	0.003	–	–
OH					< 0.1	0.001	–	–
Subtotal identified					1.1	0.072	70.5	0.338
unknown 1a[a]					0.8	0.05	–	–
Subtotal characterised					0.8	0.05	–	–
Total diastase digestion					1.9	0.122	70.5	0.338
EDTA + cellulase + 5 N HCl extraction								
BYI 02960 (parent comp.)					0.9	0.057		
amino-furanone					0.5	0.032		
Subtotal identified					1.4	0.089		
unknown 1a[a]					3.6	0.229		

1st Rotation	wheat forage		wheat hay		wheat straw		wheat grains	
TRR [mg/kg]	0.783		2.003		6.290		0.478	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Subtotal characterised					3.6	0.229		
Total EDTA + cellul. + 5 N HCl extraction					5.1	0.318		
5 N NaOH extraction					4.6	0.291		
Total identified	88.0	0.689	74.7	1.496	80.4	5.059	80.8	0.387
Total characterised	1.5	0.012	6.1	0.122	17.3	1.089	4.2	0.020
Not analysed/losses	0.3	0.002	0.7	0.015	0.7	0.042	3.0	0.014
Total extractable	89.7	0.703	81.6	1.634	98.4	6.190	88.0	0.421
Post-extraction solids	10.3	0.081	18.4	0.369	1.6	0.099	12.0	0.058
Overall total	100.0	0.783	100.0	2.003	100.0	6.290	100.0	0.478

[a] Polar unknown peak 1a in the conventional extract of wheat straw of the 1st rotation consisted of four different metabolites, all of them were minor per TLC sub-quantification

Table 20 Distribution of parent compound and metabolites in wheat matrices after 135 day plant back interval (2nd rotation), [furanone-4-^{14}C]BYI 02960 label

2nd Rotation	wheat forage		wheat hay		wheat straw		wheat grains	
TRR [mg/kg]	0.193		1.081		1.519		0.103	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction								
BYI 02960 (parent)	63.9	0.124	29.1	0.314	28.5	0.433	0.5	0.001
glucose/carbohydrates	–	–	–	–	–	–	–	–
amino-furanone	2.0	0.004	6.2	0.067	4.1	0.062	1.3	0.001
mercapto-lactic acid	1.6	0.003	3.5	0.037	4.4	0.067	–	–
bromo-amino-furanone	3.3	0.006	9.9	0.107	6.1	0.093	–	–
difluoroethyl-amino-furanone-OH-glyc	1.3	0.003	7.3	0.078	4.2	0.064	–	–
difluoroethyl-amino-furanone	8.2	0.016	6.9	0.075	5.3	0.081	–	–
glyoxylic acid	0.3	< 0.001	1.9	0.020	1.2	0.018	0.8	0.001
acetic acid-glyc	–	–	0.5	0.005	0.9	0.013	–	–
acetic acid	1.6	0.003	1.6	0.017	2.3	0.035	1.0	0.001
OH-glyc	3.4	0.007	3.4	0.037	5.0	0.076	2.1	0.002
OH	1.8	0.003	1.9	0.021	2.7	0.042	3.4	0.003
bromo / chloro	–	–	–	–	–	–	–	–
Subtotal identified	87.3	0.169	71.9	0.778	64.7	0.982	9.2	0.009
unknown 1a [a]	1.3	0.002	4.3	0.047	5.1	0.078	2.5	0.003
unknown 4	0.4	0.001	0.6	0.006	0.6	0.009	–	–
unknown 7	0.2	< 0.001	0.5	0.006	–	–	–	–
unknown 9	–	–	–	–	–	–	0.7	0.001
unknown 11	–	–	–	–	–	–	0.9	0.001
unknown 12	0.3	0.001	–	–	–	–	–	–
Subtotal characterised	2.2	0.004	5.4	0.059	5.8	0.087	4.2	0.004
Total conventional extr.	89.6	0.173	77.4	0.837	70.4	1.070	13.4	0.014
2nd Rotation	wheat forage		wheat hay		wheat straw		wheat grains	
Microwave extraction I (ACN/water)								
BYI 02960 (parent)					2.0	0.031	–	–
amino-furanone					0.7	0.011	–	–
difluoroethyl-amino-furanone-OH-glyc					0.1	0.002	–	–
difluoroethyl-amino-furanone					0.3	0.005	–	–
glyoxylic acid					0.1	0.001	–	–
acetic acid					0.1	0.001	–	–
OH-glyc					0.2	0.003	–	–
OH					0.2	0.004	–	–
Subtotal identified					3.8	0.057	–	–
unknown 1a [a]					1.0	0.015	–	–
Subtotal characterised					1.0	0.015	–	–
Total microwave extr. I	–	–	–	–	4.8	0.073	22.5	20.003

2nd Rotation	wheat forage		wheat hay		wheat straw		wheat grains	
TRR [mg/kg]	0.193		1.081		1.519		0.103	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Microwave extraction II (1% NaCl)								
BYI 02960 (parent)					2.3	0.035	–	–
amino-furanone					0.8	0.012	–	–
difluoroethyl-amino-furanone					0.3	0.004	–	–
acetic acid					< 0.1	0.001	–	–
OH-glyc					0.1	0.001	–	–
OH					0.1	0.002	–	–
Subtotal identified					3.6	0.055	–	–
unknown 1a [a]					1.5	0.023	–	–
Subtotal characterised					1.5	0.023	–	–
Total 1% NaCl extraction	–	–	–	–	5.2	0.078	241.7	20.043
Diastase digestion								
BYI 02960 (parent comp.)					1.0	0.015	–	–
amino-furanone					0.4	0.006	–	–
Subtotal identified					1.4	0.021	–	–
unknown 1a [a]					0.9	0.014	–	–
Subtotal characterised					0.9	0.014	–	–
Total diastase digestion	–	–	–	–	2.3	0.035	223.7	20.024
EDTA + cellulase + 5 N HCl extraction								
BYI 02960 (parent)					1.6	0.024		
amino-furanone					1.3	0.019		
Subtotal identified					2.9	0.043		
unknown 1a [a]					3.3	0.05		
Subtotal characterised					3.3	0.05		
Total EDTA + cellul. + 5 N HCl extraction	-	–	–	–	6.2	0.094		
5 N NaOH extraction								
Subtotal characterised					8.7	0.131		
Total identified	87.3	0.169	71.9	0.778	76.4	1.159	9.2	0.009
Total characterised	2.2	0.004	5.4	0.059	12.5	0.102	4.2	0.004
Total extractable	89.6	0.173	77.9	0.842	97.8	1.485	82.6	0.085
Not analysed/losses	–	–	0.5	0.006	0.3	0.005	69.2[b]	0.071[b]
Post-extraction solids	10.4	0.020	22.1	0.239	2.2	0.033	17.4	0.018
Overall total	100.0	0.193	100.0	1.081	100.0	1.431	100.0	0.103

[a] Polar unknown peak 1a in the conventional extract of wheat straw of the 1st rotation consisted of four different metabolites, all of them were minor per TLC subquantification

[b] No analysis performed, but presumably glucose/carbohydrates as identified in grains of 1st rotation

Table 21 Distribution of parent compound and metabolites in wheat matrices after 296 day plant back interval (3rd rotation), [furanone-4-^{14}C]BYI 02960 label

3rd Rotation	wheat forage		wheat hay		wheat straw		wheat grains	
TRR [mg/kg]	0.111		0.254		0.462		0.047	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction								
BYI 02960 (parent)	43.0	0.048	18.3	0.047	20.7	0.096	2.0	0.001
glucose/carbohydrates	–	–	–	–	–	–	–	–
amino-furanone	3.6	0.004	7.4	0.019	4.9	0.023	–	–
mercapto-lactic acid	2.6	0.003	5.1	0.013	1.5	0.007	–	–
bromo-amino-furanone	5.7	0.006	10.7	0.027	7.4	0.034	–	–
difluoroethyl-amino-furanone-OH-glyc	5.5	0.006	8.6	0.022	8.7	0.040	–	–
difluoroethyl-amino-furanone	12.1	0.013	8.3	0.021	5.2	0.024	–	–
glyoxylic acid	–	–	0.5	0.001	–	–	–	–
acetic acid	1.2	0.001	1.1	0.003	1.6	0.007	0.8	< 0.001
OH-glyc	2.5	0.003	2.3	0.006	3.4	0.016	1.7	0.001
OH	0.8	0.001	1.6	0.004	2.1	0.01	2.8	0.001
bromo / chloro	–	–	–	–	–	–	–	–

3rd Rotation	wheat forage		wheat hay		wheat straw		wheat grains	
TRR [mg/kg]	0.111		0.254		0.462		0.047	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Total identified	77.1	0.085	64.0	0.163	55.6	0.257	7.4	0.003
unknown 1a [a]	2.4	0.003	4.7	0.012	12.0	0.055	3.2	0.001
unknown 4	0.7	0.001	0.8	0.002	0.9	0.004	–	–
unknown 9	–	–	–	–	–	–	1.1	0.001
Total characterised	3.1	0.003	5.5	0.014	12.9	0.060	4.3	0.002
Total extractable	80.2	0.089	69.5	0.177	70.3	0.325	20.4	0.01
Not analysed/losses	–	–	–	–	1.8	0.008	8.8	0.004
Post–extraction solids	19.8	0.022	30.5	0.077	29.7	0.137	79.6	0.037
Overall total	100.0	0.111	100.0	0.254	100.0	0.462	100.0	0.047

[a] Polar unknown peak 1a in the conventional extract of wheat straw of the 1st rotation consisted of four different metabolites, all of them were minor per TLC subquantification.

Table 22 Distribution of parent compound and metabolites in Swiss chard and turnip matrices after a 29 day plant back interval (1st rotation), [furanone‾4-^{14}C]BYI 02960 label

1st Rotation	Swiss chard immature		Swiss chard mature		turnip leaves		turnip roots	
TRR [mg/kg]	0.848		0.871		0.679		0.074	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction								
BYI 02960 (parent)	54.3	0.460	42.6	0.371	64.3	0.437	55.9	0.041
glucose/carbohydrates	–	–	–	–	–	–	3.4	0.003
amino-furanone	1.8	0.015	3.1	0.027	0.9	0.006	4.1	0.003
difluoroethyl-amino-furanone-OH-glyc	–	–	1.2	0.01	–	–	–	–
difluoroethyl-amino-furanone	16.6	0.141	16.6	0.145	1.0	0.007	–	–
glyoxylic acid	3.7	0.031	4.7	0.041	6.6	0.045	12.2	0.009
OH-glyc-SA, isomer 1	1.2	0.01	2.5	0.022	–	–	–	–
acetic acid-glyc	0.5	0.004	1.6	0.014	3.5	0.024	–	–
OH-glyc-SA, isomer 2	3.3	0.028	3.2	0.028	–	–	–	–
acetic acid	0.4	0.003	0.6	0.005	1.2	0.008	0.2	< 0.001
OH-glyc	8.5	0.072	13.6	0.119	11.2	0.076	2.1	< 0.001
OH	2.0	0.017	1.9	0.017	1.7	0.012	0.4	< 0.001
bromo / chloro	0.6	0.005	0.4	0.003	1.1	0.007	1.3	0.001
Total identified	92.8	0.787	92.0	0.802	91.5	0.621	79.6	0.058
unknown 1a [a]	2.5	0.021	3.5	0.031	1.4	0.01	–	–
unknown 1b	–	–	–	–	–	–	6.0	0.004
unknown 6	–	–	–	–	0.8	0.006	–	–
unknown 7	–	–	–	–	0.8	0.005	–	–
unknown 8	–	–	–	–	0.5	0.003	–	–
unknown 10	–	–	–	–	0.5	0.003	–	–
unknown 11	–	–	–	–	0.2	0.002	–	–
unknown 14	–	–	0.3	0.002	0.8	0.006	–	–
unknown 17	–	–	–	–	–	–	2.4	0.002
Total characterised	2.5	0.021	13.8	0.033	5.1	0.035	8.4	0.006
Total extractable	95.7	0.812	96.1	0.838	96.6	0.656	88.1	0.065
Not analysed/losses	0.4	0.003	0.3	0.002	–	–	–	–
Post-extraction solids	4.3	0.037	3.9	0.034	3.4	0.023	11.9	0.009
Overall total	100.0	0.848	100.0	0.871	100.0	0.679	100.0	0.073

[a] Polar unknown peak 1a in Swiss chard consisted of eight different metabolites, all of them were minor per TLC subquantification

Table 23 Distribution of parent compound and metabolites in Swiss chard and turnip matrices after a 135 day plant back interval (2nd rotation), [furanone-4-^{14}C]BYI 02960 label

2nd Rotation	Swiss chard immature	Swiss chard mature	turnip leaves	turnip roots

TRR [mg/kg]	0.311		0.263		0.158		0.014	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction								
BYI 02960 (parent)	55.0	0.171	27.5	0.072	68.1	0.108	31.0	0.004
glucose/carbohydrates	–	–	–	–	–	–	16.0	0.002
amino-furanone	8.8	0.027	8.8	0.023	0.6	0.001	–	–
difluoroethyl-amino-furanone-OH-glyc	–	–	–	–	–	–	–	–
difluoroethyl-amino-furanone	10.3	0.032	17.4	0.046	1.0	0.002	–	–
glyoxylic acid	–	–	–	–	1.5	0.002	1.8	< 0.001
OH-glyc-SA, isomer 1	2.9	0.009	3.5	0.009	–	–	–	–
acetic acid-glyc	1.8	0.006	1.1	0.003	0.8	0.001	–	–
OH-glyc-SA, isomer 2	2.0	0.006	3.2	0.008	–	–	–	–
acetic acid	0.5	0.002	0.8	0.002	1.3	0.002	0.2	< 0.001
OH-glyc	11.7	0.036	18.0	0.047	12.7	0.020	2.3	< 0.001
OH	2.0	0.006	2.4	0.006	1.1	0.002	–	–
bromo / chloro	0.5	0.001	–	–	1.3	0.002	1.6	< 0.001
Total identified	95.5	0.297	82.7	0.218	88.5	0.140	53.0	0.007
unknown 1a [a]	0.9	0.003	9.3	0.025	1.4	0.002	–	–
unknown 1b	–	–	–	–	–	–	28.0	0.004
unknown 6	–	–	–	–	0.8	0.001	–	–
unknown 7	–	–	–	–	0.6	0.001	–	–
unknown 8	–	–	–	–	0.5	0.001	–	–
unknown 14	0.4	0.001	–	–	2.8	0.004	1.3	< 0.001
unknown 15	0.4	0.001	–	–	–	–	–	–
Total characterised	1.6	0.005	9.3	0.025	6.0	0.01	29.3	0.004
Total extractable	97.1	0.302	92.5	0.243	94.5	0.150	82.3	0.012
Not analysed/losses	–	–	0.5	0.001	–	–	–	–
Post-extraction solids	2.9	0.009	7.5	0.020	5.5	0.009	17.7	0.003
Accountability	100.0	0.311	100.0	0.263	100.0	0.158	100.0	0.014

[a] Polar unknown peak 1a in Swiss chard consisted of eight different metabolites, all of them were minor per TLC sub-quantification

Table 24 Distribution of parent compound and metabolites in Swiss chard and turnip matrices after a 296 day plant back interval (3rd rotation), [furanone-4-^{14}C]BYI 02960 label

3rd Rotation	Swiss chard immature		Swiss chard mature		turnip leaves		turnip roots	
TRR [mg/kg]	0.180		0.152		0.090		0.008	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction								
BYI 02960 (parent)	36.7	0.066	33.4	0.051	72.4	0.065	69.9	0.006
glucose/carbohydrates	–	–	–	–	–	–	5.3	< 0.001
amino-furanone	1.3	0.002	1.8	0.003	0.5	< 0.001	5.0	< 0.001
difluoroethyl-amino-furanone-OH-glyc	2.1	0.004	1.9	0.003	–	–	–	–
difluoroethyl-amino-furanone	13.7	0.025	15.6	0.024	1.6	0.001	–	–
glyoxylic acid	0.2	< 0.001	–	–	1.7	0.002	–	–
OH-glyc-SA, isomer 1	1.7	0.003	2.4	0.004	–	–	–	–
acetic acid-glyc	0.2	< 0.001	0.2	< 0.001	0.5	< 0.001	–	–
OH-glyc-SA, isomer 2	4.2	0.008	2.4	0.004	–	–	–	–
acetic acid	0.9	0.002	0.9	0.001	1.1	0.001	0.3	< 0.001
OH-glyc	22.2	0.040	21.9	0.033	10.2	0.009	3.1	< 0.001
OH	3.6	0.007	3.1	0.005	0.6	0.001	–	–
bromo / chloro	0.5	0.001	0.6	0.001	1.8	0.002	2.5	< 0.001
Total identified	87.3	0.157	84.2	0.128	90.2	0.081	86.2	0.007
unknown 1a [a]	3.4	0.006	1.8	0.003	1.1	0.001	–	–
unknown 1b	–	–	–	–	–	–	9.2	0.001
unknown 3	0.5	0.001	0.6	0.001	–	–	–	–
unknown 4	0.8	0.001	0.8	0.001	–	–	–	–
unknown 5	0.6	0.001	0.2	< 0.001	–	–	–	–

3rd Rotation	Swiss chard immature		Swiss chard mature		turnip leaves		turnip roots	
TRR [mg/kg]	0.180		0.152		0.090		0.008	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
unknown 6	–	–	–	–	1.1	0.001	–	–
unknown 11	0.3	< 0.001	0.2	< 0.001	–	–	–	–
unknown 13	–	–	0.6	0.001	–	–	–	–
unknown 14	0.2	< 0.001	–	–	–	–	–	–
unknown 15	0.8	0.001	0.8	0.001	–	–	–	–
Total characterised	6.5	0.012	4.9	0.008	2.3	0.002	9.2	0.001
Total extractable	93.8	0.169	89.1	0.135	92.5	0.083	95.5	0.008
Not analysed/losses	–	–	–	–	–	–	–	–
Post-extraction solids	6.2	0.011	10.9	0.017	7.5	0.007	4.5	< 0.001
Accountability	100.0	0.180	100.0	0.152	100.0	0.090	100.0	0.008

[a] Polar unknown peak 1a in Swiss chard of 1st rotation consisted of eight different metabolites, all of them were minor per TLC subquantification

Distribution of parent and metabolites in the RACs for each rotation from use of the pyridinylmethyl label are summarized below in Tables 25–30.

Table 25 Distribution of parent compound and metabolites in wheat matrices after 29 day plant back interval (1st rotation), [pyridinylmethyl-^{14}C] BYI 02960 label

1st Rotation	wheat forage		wheat hay		wheat straw		wheat grains	
TRR [mg/kg]	1.407		2.409		9.015		0.177	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction								
BYI 02960 (parent)	43.8	0.619	25.0	0.601	31.4	2.834	1.8	0.003
6-CNA	2.0	0.029	3.0	0.072	2.8	0.249	3.8	0.007
6-CNA-glycerol-gluA (1) [a]	3.2	0.045	3.4	0.083	2.1	0.193	2.3	0.004
6-CNA-glycerol-gluA (2 + 3) [b]	14.1	0.200	23.6	0.569	19.2	1.733	15.8	0.028
CHMP-glyc	1.7	0.024	5.0	0.120	2.4	0.213	–	–
CHMP	1.2	0.016	1.5	0.036	1.2	0.107	–	–
glyoxylic acid	12.3	0.173	7.3	0.176	5.6	0.508	6.0	0.011
acetic acid-glyc	0.6	0.009	–	–	0.8	0.072	–	–
OH-glyc	3.4	0.048	4.0	0.095	3.2	0.284	5.0	0.009
acetic acid	1.2	0.017	1.4	0.033	1.1	0.100	1.7	0.003
OH	1.4	0.020	2.2	0.052	2.4	0.212	6.8	0.012
bromo/chloro	–	–	0.9	0.021	0.4	0.036	–	–
Subtotal identified	84.9	1.200	77.2	1.858	72.6	6.542	43.3	0.077
unknown 1	0.8	0.011	–	–	–	–	–	–
unknown 2	–	–	–	–	0.3	0.024	–	–
unknown 3	–	–	0.7	0.017	–	–	–	–
unknown 5	–	–	0.5	0.013	0.2	0.020	–	–
unknown 6	–	–	1.1	0.026	–	–	–	–
unknown 7	–	–	–	–	0.2	0.022	–	–
unknown 9	–	–	–	–	0.6	0.056	–	–
unknown 10	–	–	–	–	0.6	0.05	2.2	0.004
unknown 14	0.7	0.01	–	–	0.5	0.042	–	–
unknown 16	–	–	0.9	0.023	0.5	0.044	–	–
unknown 17	–	–	–	–	0.3	0.028	–	–
unknown 18	–	–	–	–	0.5	0.042	–	–
unknown 21	–	–	0.9	0.022	–	–	4.1	0.007
unknown 24	–	–	–	–	0.4	0.034	–	–
unknown 25	0.7	0.01	0.8	0.018	0.3	0.023	2.6	0.005
unknown 26	–	–	–	–	0.6	0.052	–	–
unknown 27	–	–	1.0	0.023	0.7	0.060	–	–
unknown 28	1.8	0.025	0.6	0.014	0.9	0.077	–	–
unknown 29	1.0	0.014	0.8	0.019	0.6	0.055	–	–
unknown 33	–	–	–	–	1.4	0.123	–	–
Subtotal characterised	5.0	0.071	7.3	0.176	8.4	0.754	8.9	0.016

1st Rotation	wheat forage		wheat hay		wheat straw		wheat grains	
TRR [mg/kg]	1.407		2.409		9.015		0.177	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Total conventional extr.	89.9	1.271	84.5	2.034	81.0	7.296	52.2	0.093
Microwave extraction I (ACN/water)								
BYI 02960 (parent)	1.6	0.023	3.1	0.075	2.4	0.214	6.5	0.012
6-CNA	–	–	–	–	0.1	0.012	–	–
6-CNA-glycerol-gluA (2 + 3) [b]	–	–	–	–	1.4	0.123	4.4	0.008
glyoxylic acid	–	–	–	–	0.6	0.058	–	–
OH-glyc	–	–	1.6	0.040	0.1	0.012	–	–
acetic acid	–	–	0.6	0.014	0.1	0.004	–	–
OH	–	–	–	–	0.2	0.017	3.6	0.006
Subtotal identified	1.6	0.023	5.3	0.129	4.9	0.440	14.5	0.026
unknown 1	0.5	0.007	2.7	0.065	0.4	0.036	4.9	0.009
unknown 29	1.3	0.019	–	–	0.1	0.011	–	–
Subtotal characterised	1.8	0.026	2.7	0.065	0.5	0.047	4.9	0.009
Total microwave extr. I	3.5	0.049	8.0	0.193	5.4	0.487	19.4	0.034
Microwave extraction II (1% NaCl)								
BYI 02960 (parent)					1.0	0.087		
6-CNA					0.8	0.076		
6-CNA-glycerol-gluA (1) [a]					0.2	0.016		
6-CNA-glycerol-gluA (2 + 3) [b]					0.2	0.020		
OH					0.1	0.006		
Subtotal identified					2.3	0.205		
unknown 1					0.4	0.040		
unknown 29					0.2	0.015		
Subtotal characterised					0.6	0.055		
Total microwave extr. II	–	–	–	–	2.9	0.260	–	–
Diastase digestion								
BYI 02960 (parent comp.)					0.7	0.064		
6-CNA					0.6	0.055		
6-CNA-glycerol-gluA (2 + 3) [b]					0.1	0.005		
glyoxylic acid					0.2	0.020		
OH					< 0.1	0.004		
Subtotal identified					1.6	0.148		
unknown 1					0.3	0.024		
unknown 5					0.1	0.006		
unknown 9					0.1	0.008		
unknown 29					0.1	0.013		
Subtotal characterised					0.6	0.051		
Total diastase digestion	–	–	–	–	2.2	0.199	–	–
EDTA + cellulase + 5 N HCl extraction (wheat straw) or 0.1 N HCl (wheat grain)								
BYI 02960 (parent)					0.7	0.062		
6-CNA					0.7	0.066		
6-CNA-glycerol-gluA (2 + 3) [b]					0.2	0.019		
glyoxylic acid					0.3	0.028		
Subtotal identified					1.9	0.176		
unknown 1					0.8	0.071		
unknown 29					1.0	0.088		
Subtotal characterised					1.8	0.159		
Total exhaustive extraction	–	–	–	–	3.7	0.335	2.33	0.004
5 N NaOH extraction								
Total NaOH extraction							19.9	0.018
Total identified	86.5	1.220	82.5	1.987	83.4	7.511	57.8	0.103
Total characterised	6.9	0.097	10.0	0.240	11.8	1.066	13.8	0.025
Total extractable	93.5	1.316	92.5	2.227	95.2	8.578	83.8	0.149
Not analysed/losses	0.3	0.004	–	–	4.5	0.403	12.2	0.022
Post extraction solids	6.2	0.087	7.5	0.181	0.4	0.035	16.2	0.029
Overall total	100.0	1.407	100.0	2.409	100.0	9.015	100.0	0.177

[a] (1) isomer 1

[b] (2 + 3) isomer 2 and/or isomer 3

Table 26 Distribution of parent compound and metabolites in wheat matrices after 135 day plant back interval (2nd rotation), [pyridinylmethyl-^{14}C] BYI 02960 label

2nd Rotation	wheat forage		wheat hay		wheat straw		wheat grains	
TRR [mg/kg]	0.308		1.009		2.148		0.057	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction								
BYI 02960 (parent)	59.6	0.183	28.1	0.283	31.9	0.684	1.1	0.001
6-CNA	1.6	0.005	3.2	0.032	4.0	0.086	–	–
6-CNA-glycerol-gluA (1) [a]	4.4	0.014	2.4	0.024	2.4	0.051	2.5	0.001
6-CNA-glycerol-gluA (2 + 3) [b]	14.4	0.044	23.9	0.242	22.0	0.472	11.9	0.007
CHMP-glyc	0.7	0.002	0.8	0.008	2.2	0.048	–	–
CHMP	–	–	0.5	0.005	–	–	–	–
glyoxylic acid	–	–	1.0	0.01	1.7	0.036	0.9	0.001
acetic acid-glyc	0.7	0.002	–	–	–	–	–	–
OH-glyc	2.6	0.008	4.5	0.045	5.3	0.112	4.6	0.003
acetic acid	0.9	0.003	1.6	0.016	1.8	0.039	1.6	0.001
OH	1.7	0.005	1.9	0.019	3.4	0.072	6.4	0.004
Subtotal identified	86.5	0.266	67.9	0.685	74.6	1.600	29.0	0.017
unknown 1	0.3	0.001	0.4	0.004	1.3	0.028	–	–
unknown 2	1.2	0.004	–	–	–	–	–	–
unknown 3	–	–	0.5	0.005	–	–	–	–
unknown 5	–	–	0.4	0.004	–	–	–	–
unknown 7	–	–	0.6	0.006	–	–	–	–
unknown 10	–	–	0.5	0.005	–	–	1.1	0.001
unknown 14	–	–	2.3	0.023	1.1	0.024	–	–
unknown 17	–	–	1.2	0.012	–	–	–	–
unknown 18	–	–	0.6	0.006	–	–	–	–
unknown 20	–	–	0.9	0.009	–	–	–	–
unknown 21	–	–	–	–	–	–	2.1	0.001
unknown 24	0.5	0.001	0.8	0.008	–	–	–	–
unknown 25	–	–	–	–	1.8	0.039	2.3	0.001
unknown 27	0.4	0.001	0.8	0.008	–	–	–	–
unknown 28	0.3	0.001	0.2	0.002	–	–	–	–
unknown 29	0.6	0.002	0.5	0.005	–	–	–	–
Subtotal characterised	3.3	0.01	9.8	0.099	4.2	0.091	5.6	0.003
Total conventional extr.	89.9	0.276	77.6	0.784	78.8	1.692	34.6	0.020
Microwave extraction I (ACN/water)								
BYI 02960 (parent)					3.4	0.072		
6-CNA					1.5	0.032		
OH					0.3	0.007		
Subtotal identified					5.2	0.111		
unknown 1					1.6	0.034		
Subtotal characterised					1.6	0.034		
2nd Rotation	wheat forage		wheat hay		wheat straw		wheat grains	
Total microwave extr. I					6.7	0.145		
Microwave extraction II (1% NaCl)								
BYI 02960 (parent)					0.9	0.020		
6-CNA					0.9	0.019		
Subtotal identified					1.8	0.040		
unknown 1					0.3	0.006		
Subtotal characterised					0.3	0.006		
Total NaCl extraction					2.1	0.046		
Diastase digestion								
BYI 02960 (parent)					0.6	0.013		
6-CNA					0.5	0.011		
Subtotal identified					1.1	0.024		
unknown 1					0.3	0.006		
unknown 29					0.1	0.002		
Subtotal characterised					0.4	0.008		
Total diastase digestion					1.5	0.032		

2nd Rotation	wheat forage		wheat hay		wheat straw		wheat grains	
TRR [mg/kg]	0.308		1.009		2.148		0.057	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
EDTA + cellulase + 5 N HCl extraction								
BYI 02960 (parent)					0.7	0.013		
6-CNA					0.5	0.011		
Subtotal identified					1.1	0.024		
unknown 1					0.7	0.014		
unknown 29					0.8	0.017		
Subtotal characterised					1.5	0.032		
Total EDTA + cellul. + 5 N HCl extraction					2.6	0.056		
Total identified	86.5	0.266	67.9	0.685	83.8	1.799	29.0	0.017
Total characterised	3.3	0.01	9.8	0.099	8.0	0.171	5.6	0.003
Total extractable	89.9	0.276	77.6	0.784	91.8	1.970	34.6	0.020
Not analysed/losses	–	–	–	–	7.9	0.169	54.8	0.032
Post extraction solids	10.1	0.031	22.4	0.226	0.3	0.007	10.5	0.006
Overall total	100.0	0.308	100.0	1.009	100.0	2.148	100.0	0.057

[a] (1) isomer 1

[b] (2 + 3) isomer 2 and/or isomer 3

Table 27 Distribution of parent compound and metabolites in wheat matrices after 296 day plant back interval (3rd rotation), [pyridinylmethyl-^{14}C] BYI 02960 label

3rd Rotation	wheat forage		wheat hay		wheat straw		wheat grains	
TRR [mg/kg]	0.117		0.321		0.491		0.017	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction								
BYI 02960 (parent)	45.3	0.053	19.6	0.063	26.2	0.129	13.9	0.002
6-CNA	2.7	0.003	4.3	0.014	4.8	0.024	–	–
6-CNA-glycerol-gluA (1) [a]	3.1	0.004	3.6	0.011	4.5	0.022	–	–
6-CNA-glycerol-gluA (2 + 3) [b]	19.1	0.022	29.7	0.095	28.4	0.140	11.2	0.002
CHMP-glyc	3.1	0.004	4.9	0.016	2.9	0.014	–	–
CHMP	1.4	0.002	2.9	0.009	–	–	–	–
glyoxylic acid	0.8	0.001	–	–	2.6	0.013	–	–
OH-glyc	3.2	0.004	3.0	0.01	2.9	0.014	5.2	0.001
acetic acid	1.1	0.001	1.1	0.003	1.0	0.005	1.8	< 0.001
OH	1.0	0.001	1.7	0.005	2.1	0.01	5.5	0.001
Total identified	80.7	0.094	70.8	0.227	75.4	0.370	37.7	0.006
unknown 1	–	–	0.7	0.002	–	–	–	–
unknown 7	–	–	0.5	0.002	–	–	–	–
unknown 10	–	–	1.1	0.003	–	–	–	–
unknown 16	–	–	1.3	0.004	–	–	–	–
unknown 21	–	–	–	–	–	–	2.4	< 0.001
unknown 24	1.5	0.002	–	–	–	–	–	–
unknown 25	–	–	–	–	–	–	1.8	< 0.001
unknown 27	–	–	0.6	0.002	–	–	–	–
unknown 28	–	–	–	–	–	–	2.8	0.001
Total characterised	1.5	0.002	4.2	0.014	–	–	7.1	0.001
Total extractable	82.2	0.096	75.0	0.241	75.4	0.370	44.8	0.007
Not analysed/losses	–	–	–	–	1.2	0.006	–	–
Post-extraction solids	17.8	0.021	25.0	0.080	23.4	0.115	55.2	0.009
Overall total	100.0	0.117	100.0	0.321	100.0	0.491	100.0	0.017

[a] (1) isomer 1

[b] (2 + 3) isomer 2 and/or isomer 3

Table 28 Distribution of parent compound and metabolites in Swiss chard and turnip after 29 day plant back interval (1st rotation), [pyridinylmethyl-^{14}C] BYI 02960 label

1st Rotation	Swiss chard immature		Swiss chard mature		turnip leaves		turnip roots	
TRR [mg/kg]	1.358		1.483		0.815		0.072	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction								
BYI 02960 (parent)	57.4	0.779	46.3	0.687	62.4	0.508	57.8	0.042
CHMP-glyc-tri-SA	1.8	0.025	2.3	0.034	–	–	–	–
6-CNA	7.3	0.099	7.0	0.103	1.0	0.008	8.5	0.006
CHMP-glyc-di-SA	2.5	0.035	3.0	0.045	–	–	–	–
CHMP-glyc	5.5	0.075	5.4	0.080	4.5	0.037	–	–
CHMP	–	–	–	–	–	–	4.1	0.003
glyoxylic acid	1.5	0.021	2.6	0.039	2.6	0.021	8.6	0.006
acetic acid-glyc	1.6	0.022	–	–	0.4	0.003	–	–
acetic acid-glyc/ OH-glyc-SA (2)2	–	–	2.5	0.037	–	–	–	n
OH-glyc	7.4	0.101	10.9	0.162	9.4	0.076	2.5	0.002
OH-glyc-SA (1) [a]	1.1	0.015	1.7	0.025	0.6	0.005	–	–
OH-glyc-SA (2) [b]	0.5	0.006	–	–	1.7	0.014	–	–
N-formyl- /N-acetyl-AMCP-difluoroethanamine	4.0	0.055	2.9	0.043	3.6	0.030	3.5	0.003
OH	1.8	0.024	1.6	0.023	1.4	0.011	0.5	< 0.001
bromo/chloro	0.7	0.009	0.5	0.008	1.3	0.011	1.4	0.001
Total identified	93.2	1.266	86.6	1.285	88.9	0.724	86.8	0.063
unknown 1	–	–	0.3	0.005	0.4	0.004	2.2	0.002
unknown 2	–	–	0.5	0.007	–	–	–	–
unknown 6	–	–	–	–	1.3	0.011	–	–
unknown 7	–	–	–	–	–	–	1.1	0.001
unknown 11	0.7	0.01	1.1	0.016	–	–	–	–
unknown 12	–	–	0.4	0.006	0.6	0.005	–	–
unknown 13	3.0	0.041	5.0	0.075	–	–	–	–
unknown 14	–	–	0.4	0.006	–	–	–	–
unknown 15	0.8	0.011	0.3	0.004	–	–	–	–
unknown 16	–	–	1.2	0.018	2.0	0.016	–	–
unknown 18	–	–	–	–	0.9	0.007	–	–
unknown 25	–	–	–	–	0.5	0.004	–	–
unknown 26	–	–	–	–	1.1	0.009	–	–
unknown 27	–	–	–	–	< 0.1	< 0.001	–	–
unknown 28	–	–	–	–	0.4	0.004	–	–
unknown 30	–	–	0.3	0.004	1.6	0.013	–	–
unknown 31	–	–	0.2	0.003	–	–	–	–
unknown 32	–	–	0.3	0.004	–	–	–	–
Total characterised	4.5	0.062	9.9	0.146	8.8	0.071	3.2	0.002
Total extractable	97.7	1.327	96.5	1.431	97.7	0.796	90.0	0.065
Not analysed/losses	0.3	0.004	0.5	0.008	–	–	–	–
Post extraction solids	2.1	0.028	2.9	0.043	2.3	0.019	10.0	0.007
Overall total	100.0	1.358	100.0	1.483	100.0	0.815	100.0	0.072

[a] (1) isomer 1

[b] (2) isomer 2

Table 29 Distribution of parent compound and metabolites in Swiss chard and turnip after 135 day plant back interval (2nd rotation), [pyridinylmethyl-^{14}C] BYI 02960 label

2nd Rotation	Swiss chard immature		Swiss chard mature		turnip leaves		turnip roots	
TRR [mg/kg]	0.332		0.438		0.230		0.022	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction								
BYI 02960 (parent)	51.2	0.170	24.6	0.108	66.8	0.153	48.4	0.011

2nd Rotation	Swiss chard immature		Swiss chard mature		turnip leaves		turnip roots	
TRR [mg/kg]	0.332		0.438		0.230		0.022	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
CHMP-glyc-tri-SA	2.8	0.009	2.0	0.009	–	–	–	–
6-CNA	2.9	0.01	2.9	0.013	0.8	0.002	7.7	0.002
CHMP-glyc-di-SA	1.8	0.006	2.4	0.01	–	–	–	–
CHMP-glyc	5.1	0.017	1.3	0.006	4.9	0.011	2.0	< 0.001
CHMP	–	–	–	–	–	–	1.1	< 0.001
acetic acid-glyc	–	–	3.0	0.013	–	–	–	–
OH-glyc	17.4	0.058	25.3	0.111	11.1	0.025	2.4	0.001
OH-glyc-SA (1) [a]	1.6	0.005	2.9	0.013	–	–	–	–
OH-glyc-SA (2) [b]	2.5	0.008	6.3	0.028	–	–	–	–
N-formyl- /N-acetyl-AMCP-difluoroethanamine	1.6	0.005	–	–	1.2	0.003	–	–
OH	3.9	0.013	3.6	0.016	1.6	0.004	–	–
bromo/chloro	0.5	0.002	0.2	0.001	1.6	0.004	2.0	< 0.001
Total identified	91.4	0.303	74.6	0.327	88.0	0.202	63.6	0.013
unknown 1	1.4	0.005	6.8	0.030	–	–	5.6	0.001
unknown 5	–	–	–	–	–	–	1.1	< 0.001
unknown 11	–	–	1.5	0.006	–	–	–	–
unknown 13	3.6	0.012	4.3	0.019	–	–	0.6	< 0.001
unknown 14	–	–	1.2	0.005	0.7	0.002	–	–
unknown 15	0.9	0.003	0.7	0.003	–	–	–	–
unknown 16	–	–	1.1	0.005	–	–	–	–
unknown 18	–	–	–	–	1.7	0.004	–	–
unknown 19	–	–	4.8	0.021	–	–	–	–
unknown 25	–	–	–	–	1.9	0.004	–	–
unknown 30	0.8	0.003	0.6	0.003	5.7	0.013	6.5	0.001
unknown 31	–	–	0.6	0.002	–	–	–	–
Total characterised	6.7	0.022	14.7	0.064	10.0	0.023	13.9	0.003
Total extractable	98.1	0.325	96.1	0.421	98.1	0.225	77.5	0.015
Not analysed/losses	–	–	0.7	0.003	–	–	0.6	< 0.001
Post extraction solids	1.9	0.006	3.2	0.014	1.9	0.004	21.9	0.005
Overall total	100.0	0.332	100.0	0.438	100.0	0.230	100.0	0.022

[a] (1) isomer 1

[b] (2) isomer 2

Table 30 Distribution of parent compound and metabolites in Swiss chard and turnip after 296 day plant back interval (3rd rotation), [pyridinylmethyl-^{14}C] BYI 02960 label

3rd Rotation	Swiss chard immature		Swiss chard mature		turnip leaves		turnip roots	
TRR [mg/kg]	0.135		0.130		0.083		0.008	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction								
BYI 02960 (parent)	31.6	0.042	27.4	0.036	69.2	0.058	64.8	0.005
CHMP-glyc-tri-SA	2.5	0.003	0.4	0.001	–	–	–	–
6-CNA	5.3	0.007	5.1	0.007	0.8	0.001	4.7	< 0.001
CHMP-glyc-di-SA	2.6	0.003	2.8	0.004	–	–	–	–
CHMP-glyc	5.1	0.007	6.2	0.008	5.9	0.005	–	–
CHMP	–	–	–	–	–	–	1.4	< 0.001
glyoxylic acid	2.1	0.003	1.0	0.001	–	–	–	–
acetic acid-glyc/ OH-glyc-SA (2) [a]	–	–	2.8	0.004	0.9	0.001	–	–
OH-glyc	24.6	0.033	28.1	0.036	9.6	0.008	3.8	< 0.001
OH-glyc-SA (1) [b]	5.3	0.007	2.9	0.004	0.5	< 0.001	–	–
OH-glyc-SA (2) [a]	0.5	0.001	–	–	–	–	–	–
N-formyl- /N-acetyl-AMCP-difluoroethanamine	0.8	0.001	1.8	0.002	2.2	0.002	–	–
OH	3.8	0.005	3.4	0.004	–	–	–	–

3rd Rotation	Swiss chard immature		Swiss chard mature		turnip leaves		turnip roots	
TRR [mg/kg]	0.135		0.130		0.083		0.008	
Compound (BYI 02960-)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
bromo/chloro	0.7	0.001	0.4	0.001	1.6	0.001	2.5	< 0.001
Total identified	84.9	0.114	82.4	0.107	90.7	0.075	77.1	0.005
unknown 1	–	–	0.8	0.001	0.4	< 0.001	8.4	0.001
unknown 2	–	–	–	–	0.2	< 0.001	–	–
unknown 4	0.4	0.001	–	–	–	–	–	–
unknown 5	0.4	0.001	0.6	0.001	–	–	–	–
unknown 6	0.5	0.001	0.5	0.001	–	–	–	–
unknown 7	-	-	0.5	0.001	0.3	< 0.001	–	–
unknown 11	1.0	0.001	1.4	0.002	–	–	–	–
unknown 12	–	–	–	–	1.2	0.001	–	–
unknown 13	4.4	0.006	–	–	–	–	3.2	< 0.001
unknown 14	0.6	0.001	5.7	0.007	–	–	–	–
unknown 15	0.5	0.001	–	–	0.4	< 0.001	–	–
unknown 16	0.2	< 0.001	1.0	0.001	–	–	–	–
unknown 18	–	–	–	–	1.8	0.001	–	–
unknown 30	1.1	0.001	–	–	–	–	1.1	< 0.001
unknown 32	-	-	1.2	0.002	–	–	–	–
Total characterised	9.0	0.012	11.6	0.016	4.2	0.002	12.7	0.001
Total extractable	93.9	0.126	94.0	0.123	94.9	0.078	89.8	0.006
Not analysed/losses	–	–	–	–	–	–	3.3	< 0.001
Post extraction solids	6.1	0.008	6.0	0.008	5.1	0.004	6.9	0.001
Overall total	100.0	0.135	100.0	0.130	100.0	0.083	100.0	0.008

[a] (2) isomer 2

[b] (1) isomer 1

Parent compound BYI 02960 and about 30 (furanone label) or 50 (pyridinylmethyl label) metabolites were detected in the conventional and exhaustive extracts of the various samples of the three rotations.

Parent compound was the main component detected in all matrices of all rotations, except for wheat grain. Parent accounted for 34% to 64% (furanone label) or 28% to 62% (pyridinylmethyl label) of the TRR in the commodities of the 1st rotation, 28% to 68% (furanone label) or 25% to 67% (pyridinylmethyl label) TRR in the 2nd rotation, and covered 18% to 72% (furanone label) or 20% to 69% (pyridinylmethyl label) TRR in the 3rd rotation, not considering grains. In wheat grains, only trace amounts of parent compound were detected (< 1% and 2% of the TRR) in the furanone label experiments and 1–14% of the TRR in the pyridinylmethyl label experiments. The highest proportion of parent compound was always detected in turnip leaves.

Six of the identified metabolites were specific to the furanone radiolabel; BYI 02960-difluoroethyl-amino-furanone, its conjugate BYI 02960-mercapto-lactic acid, BYI 02960-difluoroethyl-amino-furanone-OH-glyc, BYI 02960-amino-furanone, BYI 02960-bromo-amino-furanone, and the natural compound glucose (or probably an isomeric carbohydrate). The natural compound was identified as the main component in wheat grain after subjecting the post extraction solids of the conventional extraction (1st rotation) to a diastase digestion step.

Eight of the identified metabolites were specific to the pyridinylmethyl radiolabel; BYI 02960-CHMP, three different conjugates of this metabolite, the corresponding oxidation product 6-CNA and its glycerol-glucuronic acid conjugates (3 isomers) and two oxidation products of the postulated cleavage product BYI 02960-AMCP-difluoroethanamine. The isomers of 6-CNA-glycerol-gluA were detected as major metabolites in all matrices of wheat. In wheat grains they represented the highest proportion of the TRR. It is probable that the weak acid 6-CNA with its pronounced phloem mobility was transported into the seeds as a phloem sink, with conjugation occurring after transport. The 6-CNA conjugates were not identified in the matrices of Swiss chard or turnips.

Identified metabolites common to both radiolabels were BYI 02960-OH and its conjugates, BYI 02960-acetic acid, its conjugates and its successor molecule BYI 02960-glyoxylic acid and the chlorinated/brominated parent compound. Halogenation of the furanone moiety of the active substance probably occurred in the soil which is supported by the fact that small amounts of halogenated parent compound were identified in the aerobic soil degradation studies.

For some RACs (e.g. wheat straw and wheat grain) from the furanone label, high identification rates were only shown for the 1st and 2nd rotation (or in the case of grains for the 1st rotation only) since exhaustive extraction and following analysis of the exhaustive extracts was only conducted for these samples. In the case of turnip roots of the 2nd rotation, the identification rate was also quite low (53%) since one unknown compound represented a high percentage of the TRR, but at a very low residue level (0.004 mg/kg).

Overall, identification rates using the pyridinylmethyl label were high and ranged from 68–87% of the TRR in all RACs apart from wheat grains. In grains the identification rate was lower (29–58% of the TRR), but at least additional significant portions of the TRR were characterized by the extraction behaviour of the residues.

Four general metabolic transformation reactions were observed (cleavage, hydroxylation, conjugation and halogenation). These reactions are consistent with the reactions observed in primary crop metabolism studies. The following metabolic pathway was proposed:

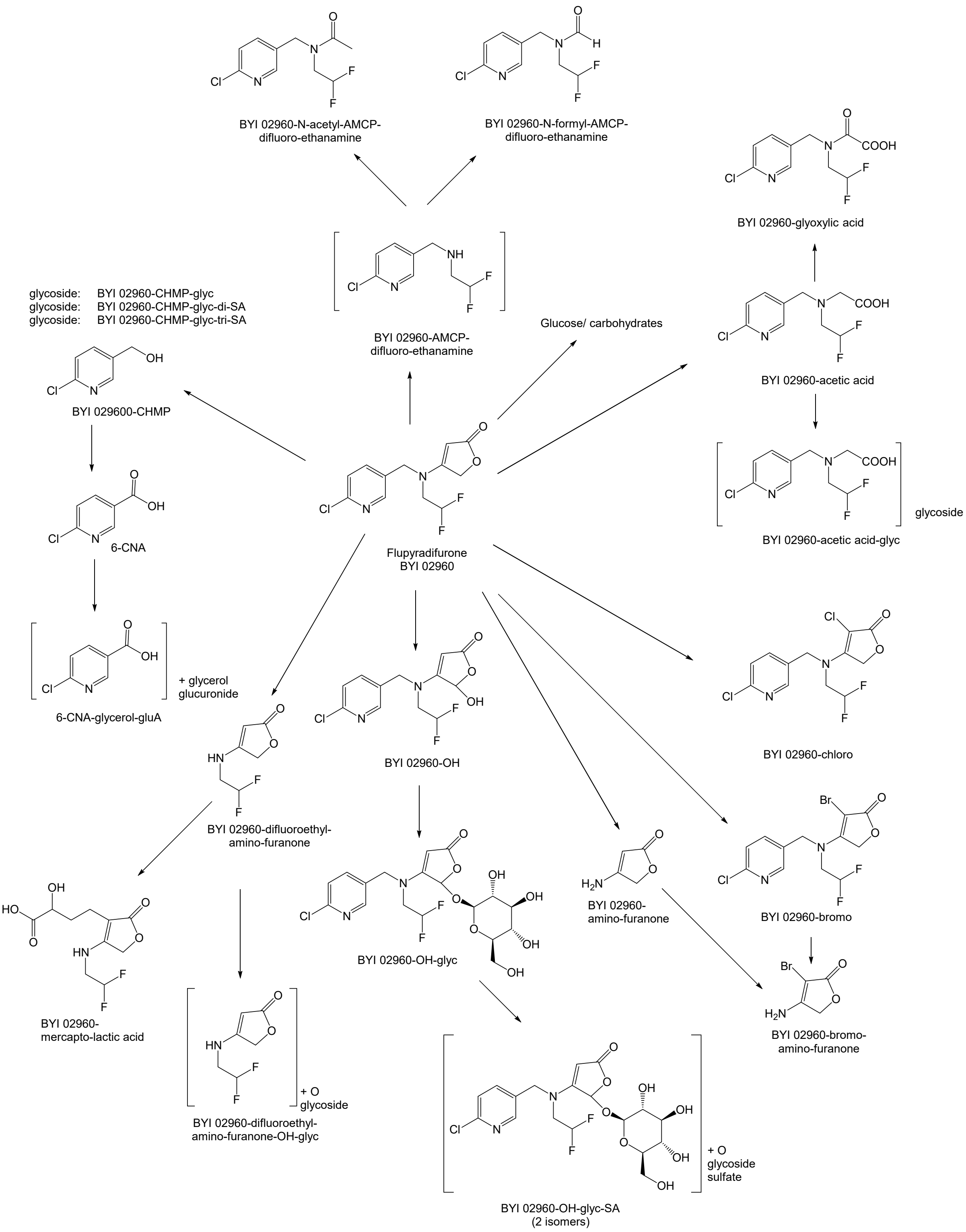


Figure 6 Proposed metabolic pathway of flupyradifurone in confined rotational crops

Analysis for difluoroacetic acid (DFA)

Samples from the confined rotational crop studies were also analysed for non-radiolabelled difluoroacetic acid (DFA) (Schoening and Ruhl 2012, MR-11/050). Results are summarized below in Table 31.

Table 31 Summary of difluoroacetic acid residues in rotational crops after soil application of BYI 02960

Metabolism study	Rotation	Crop	Sample material	Difluoroacetic acid residues [mg/kg] as DFA
Metabolism of [furanone-4-^{14}C]BYI 02960 in Confined Rotational Crops	1st rotation	wheat	forage	0.09
			hay	0.32
			straw	0.20
			grain	1.15
		Swiss chard	intermediate	0.08
			mature	0.16
		turnip	leaves	0.08
			roots	0.02
	2nd rotation	wheat	forage	0.02
			hay	0.14
			straw	0.06
			grain	0.26
		Swiss chard	intermediate	0.04
			mature	0.05
		turnip	leaves	0.03
			roots	< 0.01
	3rd rotation	wheat	forage	< 0.01
			hay	0.01
			straw	0.02
			grain	0.05
		Swiss chard	intermediate	< 0.01
			mature	0.01
		turnip	leaves	< 0.01
			roots	< 0.01

Significant levels of difluoroacetic acid were detected in all plant matrices of the first and second rotation except turnip roots of the second rotation. DFA represented a major proportion of the residues in the edible crops wheat grain, Swiss chard and turnip roots and as well as in the feed item wheat hay. In wheat forage and straw and turnip leaves, DFA was less prominent than parent, but it was still a major compound.

Animal metabolism

Metabolism in the rat

Evaluation of the metabolism studies in rodents was carried out by the WHO Core Assessment Group.

In studies conducted in rats using [^{14}C]flupyradifurone, maximum plasma concentrations of radioactivity (Cmax) were reached at 1 hour after a single dose of 2 mg/kg bw and 2–4 hours after a single dose of 200 mg/kg bw. Based on the mass balance of radioactivity in urine and tissues following oral dosing, estimates of gastrointestinal absorption ranged from 79% in males to 91% in females. Based on a comparison of the dose-normalised area-under-the-plasma-time concentration curve (AUC) following equivalent oral and intravenous doses in males, gastrointestinal absorption was estimated to be 93%. The plasma elimination half-life ($t_{1/2}$) ranged from 3–8 hours. The majority (up to 90%) of radioactivity was excreted in urine within 24 hours. There was no evidence of tissue accumulation. Whilst parent was the main compound detected in excreta (up to 50% of radioactivity in males and 70% in females), flupyradifurone was metabolized to eight identified metabolites and up to 19 unidentified metabolites involving hydroxylation, conjugation and cleavage reactions. The extent of metabolism was greater in male than female rats.

Metabolism in lactating goats

Two studies on the metabolism of flupyradifurone in lactating goats were conducted with the test compound labelled either in the [furanone-4-^{14}C] or the [pyridinylmethyl-^{14}C]-position. A lactating goat (Weiße deutsche Edelziege, 24 months, 48 kg mean body weight through the study for the furanone label and 36 months, 62 kg mean body weight through the study for the pyridinylmethyl label) was orally dosed once daily for five consecutive days in the morning after milking with 1.0 mg of the active substance per kg body weight which corresponded to 28.8 mg ai/kg dry feed/day for the furanone label (Bongartz and Koester 2011b, MEF-11/268) and 24.4 mg ai/kg dry feed/day for the pyridinylmethyl label (Bongartz and Koester 2011a, MEF-11/269). The goats were milked in the morning immediately prior to each administration, about 8 hours later in the afternoon and directly before the scheduled termination. Urine and faeces were collected in intervals of 24 hours after administrations 1–4 and 6 hours after the fifth administration. Each animal was sacrificed approximately 6 hours after the last administration. Total radioactive residues (TRR) were determined in milk and excreta at various sampling intervals, and in muscle, fat, kidney and liver at sacrifice. Milk, edible organs and tissues and urine were analysed for the unchanged parent and metabolites.

The radioactivity levels and concentrations measured in the milk ranged from 0.755 mg/kg equiv. at 24 hours to 1.213 mg/kg equiv. at 56 hours after the first administration for the furanone label. At sacrifice, the residue concentration was 1.165 mg/kg equiv. Residues increased significantly during the eight-hour period after the second up to the fifth administration followed by a decrease measured prior to the delivery of the next dose. A plateau level of about 1.1 mg/kg equiv. was reached approximately 50 hours after the first administration. The highest TRR values in tissues were found in liver (1.746 mg/kg equiv.) and kidney (1.472 mg/kg equiv.), corresponding to 0.65 and 0.09% of the total dose respectively. The overall recovery of radioactivity accounted for approximately 79% of the total administered dose.

For the pyridinylmethyl label the concentrations of radioactivity in milk ranged from 0.053 mg/kg equiv. at 96 hours to 1.345 mg/kg equiv. at sacrifice, 102 hours after the first administration. Residues increased significantly during the eight-hour period after each administration followed by a decrease to a very low level of about 0.05 mg/kg equiv. measured prior to the administration of the next dose. A plateau level of about 0.3 mg/kg equiv. was reached at 8 hours after the first administration. The highest TRR values in tissues were found in kidney (1.869 mg/kg equiv.) and liver (1.215 mg/kg equiv.), corresponding to 0.10 and 0.50% of the total dose respectively. The overall recovery of the administered dose was approximately 89%).

For both labels it was speculated that much of the remaining radioactivity was expected to still be present in the gastro-intestinal tract at sacrifice due to the short period between the last administration and sacrifice.

Table 32 Distribution of residues in tissues and milk from goat after dosing with [pyridinylmethyl-^{14}C]-flupyradifurone or [furanone-4-^{14}C]-flupyradifurone

	[furanone-4-^{14}C]-flupyradifurone		[pyridinylmethyl-^{14}C]-flupyradifurone	
Matrices	Percent of total dose administered (%)	Concentration of total radioactivity (mg/kg equiv.)	Percent of total dose administered (%)	Concentration of total radioactivity (mg/kg equiv.)
Liver	0.65	1.746	0.50	1.215
Kidney	0.09	1.472	0.10	1.869
Muscle, total	2.91	0.539	2.10	0.356
Fat, total	0.57	0.265	0.25	0.106
Total of organs/tissues	4.22	–	2.94	–
Milk [0–102 h]	2.58	0.961	0.78	0.186
Urine [0–102 h]	69.15	–	71.74	–
Faeces [0–102 h]	3.00	–	13.28	–
Total Excreted	72.14	–	85.02	–
Total Recovery	78.94	–	88.75	–

For analysis of parent compound and metabolites, milk, muscle, kidney and liver were conventionally extracted with mixtures of acetonitrile/water. An additional extraction step with acetone was performed for milk (furanone label). Fat was extracted with mixtures of n-heptane and acetonitrile/ water followed by a partition procedure yielding an acetonitrile/ water phase. After purification and concentration, the resulting conventional extracts of milk, muscle, fat, kidney and liver represented between 73.3 and 95.0% TRR for the furanone label and 92.9 and 99.8% TRR for the pyridinylmethyl label. Solids of kidney and liver were exhaustively extracted with mixtures of acetonitrile/water, 0.1 N hydrochloric acid and 0.1 N sodium hydroxide using microwave assistance (pyridinylmethyl label), but the exhaustive extracts were not further investigated.

The characterisation and identification of residues in tissues and milk are summarized in Tables 33 and 34.

Table 33 Distribution of parent and metabolites in the tissues and milk of a lactating goat after dosing with [furanone-4-^{14}C]-flupyradifurone

Extract/Identification	Milk (24 to 102 h)		Muscle		Fat		Kidney		Liver	
Overall TRR [mg/kg]	1.046		0.539		0.265		1.472		1.746	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Conventional extraction										
ACN/water extract	90.7	0.948	95.0	0.512	88.4	0.234	89.1	1.311	73.3	1.280
BYI 02960 (parent)	23.9	0.250	88.1	0.475	80.5	0.213	50.5	0.744	59.8	1.045
Lactose	66.8	0.698	–	–	–	–	–	–	–	–
Polar metabolites	–	–	4.4	0.024	5.0	0.013	10.0	0.148	12.1	0.211
Unknown	–	–	0.7	0.004	–	–	–	–	1.4	0.024
OH-gluA (isomer 1)	–	–	–	–	–	–	2.2	0.032	–	–
OH-gluA (isomer 2)	–	–	–	–	–	–	2.2	0.032	–	–
OH-gluA (isomer 3)	–	–	–	–	–	–	4.7	0.069	–	–
OH-gluA (isomer 4)	–	–	–	–	–	–	3.5	0.052	–	–
Des-difluoroethyl	–	–	–	–	–	–	1.3	0.019	–	–
OH	–	–	1.8	0.01	2.9	0.008	14.6	0.215	–	–
Identified in conv. extract	90.7	0.948	89.9	0.484	83.4	0.221	79.0	1.163	59.8	1.045
Characterised in conv. extract	n.d.	n.d.	5.1	0.028	5.0	0.013	10.0	0.148	13.5	0.211
MeOH/DCM-fraction of the clean-up by SPE distillate	0.6 0.1	0.007 0.001	0.2 n.d.	0.001 n.d.	0.2 –	0.001 –	0.4 0.3	0.006 0.005	0.6 –	0.011 –
n-heptane phase	n.a.	n.a.	n.a.	n.a.	5.4	0.014	n.a.	n.a.	n.a.	n.a.
Exhaustive extraction										
ACN/water extract	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	2.6	0.039	3.1	0.053
0.1 N hydrochloric acid	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	4.0	0.059	11.1	0.193
0.1 N sodium hydroxide	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	3.6	0.052	12.0	0.209
Total identified	90.7	0.948	89.9	0.484	83.4	0.221	79.0	1.163	59.8	1.045
Total characterised	n.d.	n.d.	5.1	0.028	5.0	0.013	10.0	0.148	13.5	0.235
Not analysed residues	0.7	0.008	0.2	0.001	5.6	0.015	11.0	0.161	26.8	0.466
Total extracted	91.4	0.956	95.2	0.513	94.1	0.249	100.0	1.472	100.0	1.746
Solids	8.6	0.090	4.8	0.026	5.9	0.016	n.d.	n.d.	n.d.	n.d.
Accountability	100	1.046	100	0.539	100	0.265	100	1.472	100	1.746

Table 34 Distribution of parent and metabolites in the tissues and milk of a lactating goat after dosing with [Pyridinylmethyl-^{14}C]-BYI 02960 (flupyradifurone)

Extract/ Identification	Milk (24 to 102 h)		Muscle		Fat		Kidney		Liver	
Overall TRR [mg/kg]	0.186		0.356		0.106		1.869		1.215	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
ACN/water extract	99.3	0.184	99.4	0.353	99.2	0.105	98.8	1.847	92.8	1.128
BYI 02960 (parent)	88.8	0.165	98	0.349	99.2	0.105	34.8	0.650	84.6	1.028
cysteinyl-nicotinic acid	–	–	–	–	–	–	6.1	0.114	4.8	0.058
hippuric acid	9.1	0.017	–	–	–	–	9.5	0.178	0.8	0.01
methylthio-glyoxylic acid	1.5	0.003	1.3	0.005	–	–	–	–	–	–

Extract/ Identification	Milk (24 to 102 h)		Muscle		Fat		Kidney		Liver	
Overall TRR [mg/kg]	0.186		0.356		0.106		1.869		1.215	
(BYI 02960-) Equivalents	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
OH-gluA (isomer 1)	–	–	–	–	–	–	6.0	0.112	–	–
OH-gluA (isomer 2)	–	–	–	–	–	–	9.3	0.175	1.4	0.016
OH-gluA (isomer 3)	–	–	–	–	–	–	8.4	0.158	–	–
OH-gluA (isomer 4)	–	–	–	–	–	–	7.5	0.141	–	–
AMCP-Difluoroethanamine	–	–	–	–	–	–	1.1	0.02	1.2	0.015
OH	–	–	–	–	–	–	16.0	0.299	–	–
Total identified	99.3	0.184	99.4	0.353	99.2	0.105	98.8	1.847	92.8	1.127
MeOH/DCM-fraction of the clean-up by SPE	–	–	0.1	< 0.001	0.6	0.001	0.1	0.001	0.1	0.001
n-heptane phase	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.
acetone extract	0.1	< 0.001	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Not analysed residues	0.1	< 0.001	0.1	< 0.001	0.6	0.001	0.1	0.001	0.1	0.001
Total extracted	99.5	0.185	99.5	0.354	99.8	0.106	98.9	1.848	92.9	1.129
Solids	0.5	0.001	0.5	0.002	0.2	< 0.001	1.1	0.021	7.1	0.086
Accountability	100	0.186	100	0.356	100	0.106	100	1.869	100	1.215

For the furanone label, parent was the only significant residue in muscle, fat, and liver (59.8–88.1% TRR). In addition to parent, lactose was found in milk (66.8% TRR, 0.698 mg/kg equiv.). Some metabolism was observed in kidney; however, the parent was still the predominant residue (50.5% TRR). In addition to parent, the other major component of the radioactivity in the kidney was BYI 02960-OH (hydroxylation in position 5 of the furanone ring), which amounted to 14.6% of the TRR.

For the pyridinylmethyl label, parent was by far the dominating constituent of the residue in milk, organs and tissues. Its concentration amounted to 88.8% of the TRR for milk, 98.0% of the TRR for muscle, 99.2% of the TRR for fat, 34.8% of the TRR for kidney and 84.6% of the TRR for liver. Extensive metabolism was detected in the kidney, where most metabolites was found. Apart from parent, the major component of radioactivity in the kidney was BYI 02960-OH (hydroxylation in position 5 of the furanone ring), which amounted to 16.0% of the TRR. Four glucuronic acid conjugates of the hydroxylated parent compound were also detected at 6.0–9.3% TRR while hippuric acid was observed at 9.5% TRR.

A metabolic pathway for flupyradifurone in goats was proposed. The pathway reflects the following observed reactions:

- hydroxylation of parent followed by conjugation with glucuronic acid forming two diastereomeric conjugates of BYI 02960-OH (BYI 02960-OH-gluA, isomer 2 and 3) with the hydroxylation and conjugation being in the 5-position of the furanone ring, one isomer (BYI 02960-OH-gluA, isomer 4) with hydroxylation and conjugation in the difluoroethyl side chain and one isomer (BYI 02960-OH-gluA, isomer 1) with hydroxylation and conjugation in an unknown position.
- oxidative cleavage of the pyridinylmethyl bridge forming BYI 02960-6-CNA followed by conjugation with glycine to form BYI 02960-hippuric acid.
- substitution of the chlorine atom of parent with glutathione followed by degradation resulting in the conjugate BYI 02960-cysteine, oxidative cleavage of the pyridinylmethyl bridge of BYI 02960-cysteine forming BYI 02960-cysteinyl-nicotinic acid, further degradation of BYI 02960-cysteine in the cysteine group and the furanone ring forming BYI 02960-methylthio-glyoxylic acid.
- cleavage of the furanone ring forming BYI 02960-AMCP-difluoroethanamine.
- cleavage of the difluoroethyl group forming BYI 02960-des-difluoroethyl.

- cleavage and subsequent degradation of the furanone ring forming small carbon units (C-1- or C-2-fragments), entering the carbon pool of endogenous compounds and finally being used for example for the biosynthesis of lactose.

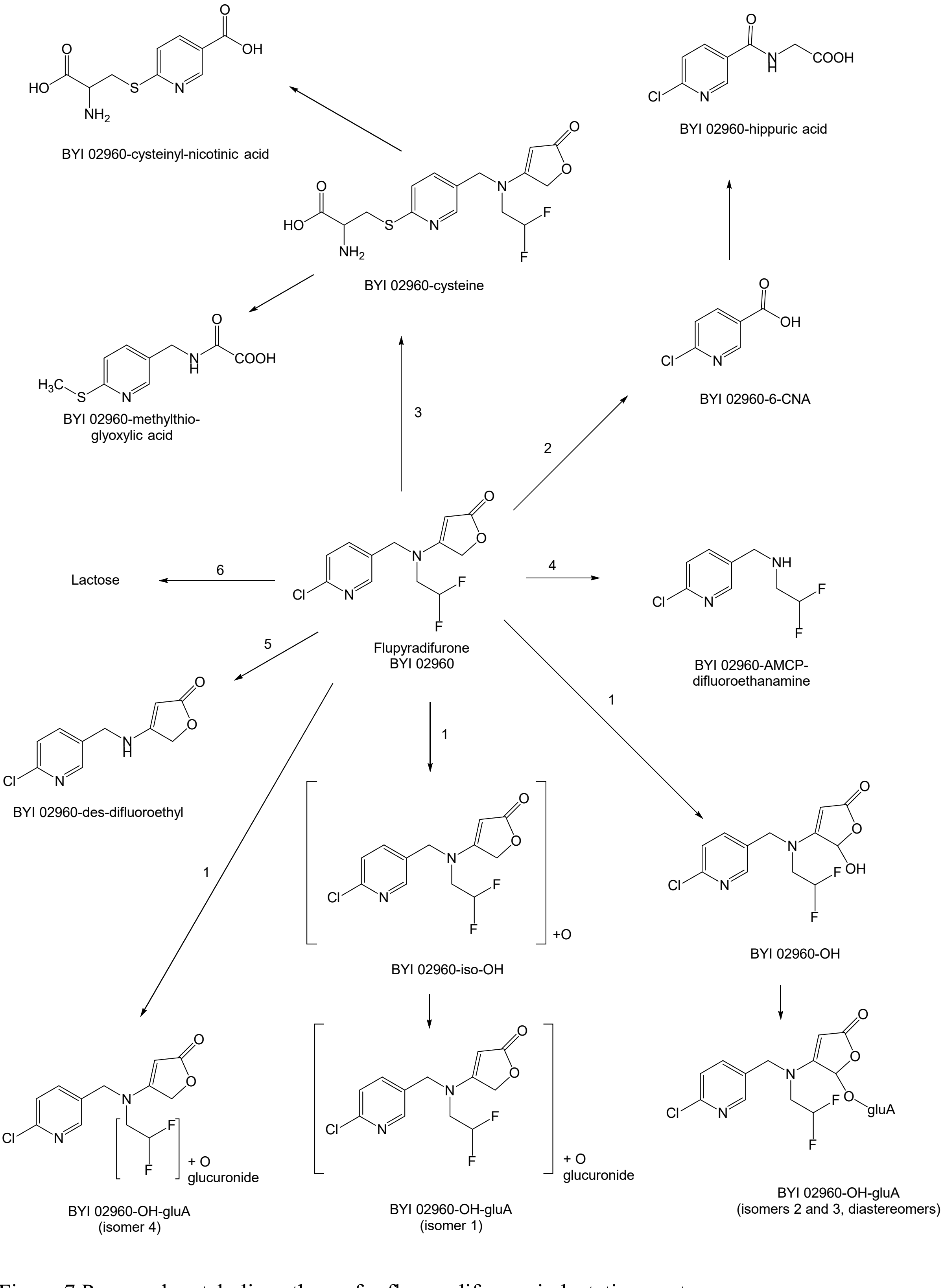


Figure 7 Proposed metabolic pathway for flupyradifurone in lactating goats

Laying hens

Two studies on the metabolism of flupyradifurone in laying hens were conducted with the test compound labelled either in the [furanone-4-^{14}C]- or the [pyridinylmethyl-^{14}C]- position. Six hens (White leghorns, 6–8 months, 1.57 kg mean body weight through the study for the furanone label and 1.62 kg mean body weight through the study for the pyridinylmethyl label) were dosed orally once daily in the morning for 14 consecutive days with an aqueous 0.5% Tragacanth® suspension of 1.02 mg of the active substance per kg body weight or 1.05 mg of the active substance per kg body weight which corresponded to 17.13 mg ai/kg dry feed/day for the furanone label (Bongartz and Koester 2012b, MEF-11/200) and 16.18 mg ai/kg dry feed/day for the pyridinylmethyl label (Bongartz and Koester 2012a, MEF-11/199). The animals were sacrificed at approximately six hours after the last administration. Total radioactive residues (TRR) were determined daily in the eggs and excreta, and at sacrifice in the dissected organs and tissues (muscle, fat, liver, kidney, skin and eggs from ovary/oviduct). Eggs, muscle, fat, liver and excreta were extracted and analysed for parent compound and metabolites.

The concentration of radioactivity in eggs ranged from 0.024 mg/kg equiv. at Day 1 to 1.198 mg/kg equiv. at sacrifice (furanone label) and 0.016 mg/kg equiv. at Day 1 to 0.119 mg/kg equiv. at sacrifice (pyridinylmethyl label). Following a linear increase, a plateau level of approximately 1.035 mg/kg equiv. was reached nine days after the first administration (furanone label) and 0.08 mg/kg equiv. was reached six days after the first administration (pyridinylmethyl label). In the organs and tissues, the highest radioactivity concentrations were determined in liver (2.178 mg/kg equiv.) and kidney (1.083 mg/kg equiv.) corresponding to 0.37 and 0.05% of the total dose respectively (furanone label) and in kidney (1.073 mg/kg equiv.) and liver (0.435 mg/kg equiv.) corresponding to 0.05 and 0.08% of the total dose respectively.

The overall recovery of the administered dose was 82.16% of the total dose for the furanone label and 96.11% of the total dose for the pyridinylmethyl label radioactivity was expected to still be present in the gastro-intestinal tract at sacrifice, due to the short period between the last administration and sacrifice.

Table 35 Concentration of radioactive residues in poultry matrices after dosing with [pyridinylmethyl-^{14}C]-flupyradifurone or [furanone-4-^{14}C]-flupyradifurone

	[furanone-4-^{14}C]-flupyradifurone		[pyridinylmethyl-^{14}C]-flupyradifurone	
Matrices	Percent of total dose administered	TRR (mg/kg)	Percent of total dose administered	TRR (mg/kg)
Liver	0.37	2.178	0.08	0.435
Kidney	0.05	1.083	0.05	1.073
Eggs from ovary/oviduct	0.46	2.774	0.01	0.147
Skeletal muscle, total	0.50	0.183	0.19	0.070
Body skin, total	0.07	0.257	0.02	0.094
Body fat, total	0.34	0.427	0.02	0.021
Total organs/tissues	1.80	–	0.37	–
Eggs (cumulative after 14 days)	2.35	0.757	0.24	0.081
Excreta (cumulative after 14 days)	78.01	–	95.51	–
Total recovery	82.16	–	96.11	–

For analysis of parent compound and metabolites, eggs (Days 2–7 and 8–13.25), muscle, fat and liver were conventionally extracted with acetonitrile and heptane followed by a partition procedure (furanone label). In the case of eggs, muscle and liver for the furanone label, the extraction procedure was continued with a mixture of acetonitrile/water. For the pyridinylmethyl label eggs (Days 3–13.25), muscle and liver were conventionally extracted with mixtures of acetonitrile/water and pure acetonitrile while fat was extracted with acetonitrile and n-heptane followed by a partition procedure yielding an acetonitrile and n-heptane phase. Post-extraction solids of eggs, muscle and liver were exhaustively extracted with mixtures of acetonitrile/water followed by acetonitrile/water/formic acid using microwave assistance (furanone label) while for the pyridinylmethyl label, post extraction solids of liver only were exhaustively extracted with acetonitrile/water using microwave assistance.

The characterisation and identification of the residues in eggs, muscle, fat and liver are summarized in Table 36.

Table 36 Identification and characterisation of the TRR in hen matrices after dosing with [furanone-4-^{14}C]-flupyradifurone

Extract/Identification	Eggs (day 2 to 7)		Eggs (Day 8 to 13.25)		Muscle		Fat		Liver	
TRR [mg/kg]	0.540		1.048		0.183		0.427		2.178	
(BYI 02960-) Equivalents	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg
Conventional extraction										
Conventional extract	65.0	0.351	66.8	0.701	38.6	0.071	98.5	0.421	72.1	1.57
BYI 02960 (parent)	2.3	0.013	1.6	0.016	13.6	0.005	–	–	0.5	0.01
n-heptane phase (identified as fatty acids)	52.0	0.281	58.3	0.611	8.1	0.015	95.9	0.41	51.5	1.121
des-difluoroethyl-OH-SA	0.1	0.001	n.d.	n.d.	0.5	0.001	–	–	0.2	0.004
des-difluoroethyl	1.2	0.006	0.6	0.007	2.6	0.005	–	–	0.8	0.017
OH-SA	0.6	0.003	0.5	0.005	n.d.	n.d.	–	–	5.1	0.112
OH	2.3	0.013	1.6	0.016	2.4	0.004	–	–	0.8	0.018
Identified in the conventional extract	58.5	0.317	62.6	0.655	16.5	0.03	95.9	0.41	58.9	1.282
Number of characterised fractions and metabolites	4		2		9		1		15	
Amount of the largest characterised fraction or metabolite	2.7	0.015	3.6	0.038	6.6	0.012	2.6	0.011	2.3	0.05
Characterised in the conventional extract	6.4	0.035	4.3	0.045	22.0	0.040	2.6	0.011	13.2	0.288
Losses during the conventional extraction	10.7	0.058	10.7	0.112	10.3	0.019	n.d.	n.d.	n.d.	n.d.
Exhaustive extraction										
—neutral ACN/water extract	7.0	0.038	3.6	0.038	8.1	0.015	n.a.		9.6	0.209
—1st acidic ACN/water extract	8.3	0.045	10.0	0.105	39.5	0.072	n.a.		12.1	0.264
—2nd acidic ACN/water extract	1.1	0.006	3.3	0.035	[a]		n.a.		n.a.	
Number of characterised fractions and metabolites	6		6		5				12	
Amount of the largest characterised fraction or metabolite	6.5	0.035	7.1	0.075	35.0	0.064			4.8	0.104
Characterised in the exhaustive extracts	16.5	0.089	16.9	0.177	47.7	0.087	n.a.		21.7	0.473
Losses during the conventional extraction	6.8	0.037	4.1	0.043	n.d.	n.d.	n.a.		4.5	0.098
Total identified	58.5	0.316	62.5	0.656	16.5	0.030	95.9	0.410	58.9	1.282
Total characterised	22.9	0.124	21.2	0.223	69.7	0.128	2.6	0.011	34.9	0.761
Total extracted	81.5	0.440	83.8	0.878	86.2	0.158	98.5	0.421	93.8	2.043
Total losses	17.5	0.094	14.7	0.155	10.3	0.019	n.d.	n.d.	4.5	0.098
Solids	1.1	0.006	1.5	0.016	3.5	0.006	1.5	0.006	1.7	0.036
Accountability	100	0.540	100	1.048	100	0.183	100	0.427	100	2.178

[a] Both acidic ACN/water extracts of muscle were combined before concentration.

Table 37 Identification and characterisation of the TRR in hen matrices after dosing with [pyridinylmethyl-^{14}C]-flupyradifurone

Extract/Identification	Eggs (Days 3–13.25)		Muscle		Fat		Liver	
TRR [mg/kg]	0.084		0.070		0.021		0.435	
(BYI 02960-) Equivalents	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg
Conventional extraction	96.1	0.081	92.6	0.064	79.7	0.017	74.6	0.324
n-heptane phase	n.a.		n.a.		n.a.		n.a.	
ACN/water phase	96.1	0.081	92.6	0.064	79.7	0.017	74.6	0.324
BYI 02960 (parent)	19.8	0.017	9.8	0.007	15.3	0.003	0.9	0.004
lactato-mercaptyl-nicotinic acid	4.0	0.003	3.6	0.002	–	–	15.5	0.068
acetyl-cysteinyl-nicotinic acid	–	–	–	–	–	–	0.3	0.001
6-CNA	7.2	0.006	8.8	0.006	1.8	< 0.001	6.4	0.028
des-difluoroethyl-OH-SA	–	–	2.1	0.001	5.6	0.001	3.1	0.014
acetyl-AMCP	23.1	0.019	40.2	0.028	28.5	0.006	6.3	0.027
des-difluoroethyl	8.9	0.007	9.9	0.007	5.0	0.001	1.8	0.008
AMCP-difluoroethanamine-SA	–	–	–	–	–	–	0.3	0.001
OH-SA	5.1	0.004	1.8	0.001	16.2	0.003	22.5	0.098
OH	18.0	0.015	8.1	0.006	5.5	0.001	1.5	0.007
Identified in conventional extract	86.2	0.072	84.2	0.059	77.9	0.016	58.6	0.255
Number of characterised metabolites	2		2		1		10	
Amount of the largest metabolite	8.5	0.007	6.6	0.005	1.8	< 0.001	6.3	0.027
Characterised in conventional extract	9.9	0.008	8.4	0.006	1.8	< 0.001	16.0	0.070
Exhaustive extraction	n.a.		n.a.		n.a.		19.8	0.086
Number of characterised metabolites							11	
Amount of the largest metabolite							6.1	0.027
Total identified	86.2	0.072	84.2	0.059	77.9	0.016	58.6	0.255
Total characterised	9.9	0.008	8.4	0.006	1.8	< 0.001	35.8	0.156
Total extracted	96.1	0.081	92.6	0.064	79.7	0.017	94.4	0.411
Solids	3.9	0.003	7.4	0.005	20.3	0.004	5.5	0.024
Accountability	100	0.084	100	0.070	100	0.021	100	0.435

For the furanone label the main part of the radioactivity in eggs (Days 2 to 7, 52.0% and Days 8 to sacrifice, 58.3%) was detected in the n-heptane phase and identified as fatty acids. The parent, BYI 02960-OH, BYI 02960-OH-SA, BYI 02960-des-difluoroethyl and BYI 02960-des-difluoroethyl-OH-SA were identified in low amounts (less than 2%). Residues in the n-heptane phase of muscle were identified as fatty acids (8.1% of the TRR). Very low amounts (less than 2%) of parent, BYI 02960-OH, BYI 02960-des-difluoroethyl and BYI 02960-des-difluoroethyl-OH-SA were identified in the acetonitrile/water extract. Metabolites in the acidic exhaustive extract were characterised by their extraction behaviour, and represented the major portion of residues in the muscle sample (39.5% of the TRR). An HPLC analysis could not be performed, due to the high matrix burden in the extract. The major part of the radioactive residues in fat (95.9% of the TRR) was identified as fatty acids after saponification. The fatty acids were presumably formed from smaller carbon units (C-1- or C-2-fragments), which entered the carbon pool of endogenous compounds after cleavage and subsequent degradation of the furanone ring. More than half of the residues in liver (51.5% of the TRR) was also identified as fatty acids. Parent, BYI 02960-OH, BYI 02960-OH-SA, BYI 02960-des-difluoroethyl and BYI 02960-des-difluoroethyl-OH-SA were identified in the acetonitrile/water extract. The predominant metabolite was BYI 02960-OH-SA, which amounted to 5.1% of the TRR.

For the pyridinylmethyl label major metabolites in eggs were parent (19.8% of the TRR), BYI 02960-acetyl-AMCP (23.1% of the TRR) and BYI 02960-OH (18.0% of the TRR). The main metabolite in muscle was BYI 02960-acetyl-AMCP, which amounted to 40.2% of the TRR. Parent, BYI 02960-6-CNA, BYI 02960-des-difluoroethyl and BYI 02960-OH were quantified in amounts between 8.1–9.9% of the TRR. The main metabolites in the polar phase of fat were parent (15.3% of the TRR), BYI 02960-acetyl-AMCP (28.5% of the TRR) and BYI 02960-OH-SA (16.2%of TRR). Most the TRR in liver (approx. 75%) was conventionally extracted. The main metabolites in the conventional extract were BYI 02960-OH-SA (22.5% of the TRR), BYI 02960-lactato-mercaptyl-nicotinic acid (15.5% of the TRR), BYI 02960-6-CNA (6.4% of the TRR) and BYI 02960-acetyl-

AMCP (6.3% of the TRR). Parent was detected in negligible amounts of 0.004 mg/kg equiv. Metabolites in the exhaustive extract of liver amounted to 19.8% of the TRR. All unknown metabolites in the exhaustive extract of liver were only characterised by extraction and chromatographic behaviour (35.8% of the TRR; 0.156 mg/kg equiv.).

A metabolic pathway for flupyradifurone in laying hens was proposed.

The pathway reflects the following observed reactions:

- hydroxylation in position 5 of the furanone ring forming BYI 02960-hydroxy followed by conjugation with sulfuric acid to BYI 02960-OH-SA.
- oxidative cleavage of the pyridinylmethyl moiety forming BYI 02960-6-CNA and substitution of the chloro group of BYI 02960-6-CNA with glutathione followed by degradation resulting in the conjugates BYI 02960-acetyl-cysteinyl-nicotinic acid and BYI 02960-lactato-mercaptyl-nicotinic acid.
- cleavage of the difluoroethyl group forming BYI 02960-des-difluoroethyl followed by hydroxylation and conjugation with sulphuric acid to BYI 02960-des-difluoroethyl-OH-SA.
- cleavage of the furanone ring and conjugation with sulphonic acid forming BYI 02960-AMCP-difluoroethanamine-SA.
- oxidative degradation of the furanone ring forming BYI 02960-acetic acid.
- cleavage of the pyridinylmethyl moiety forming an alcohol conjugated with serine (BYI 02960-CHMP-serinate).
- cleavage of the furanone ring and the difluoroethyl group forming an amine followed by acetylation to BYI 02960-acetyl-AMCP.
- cleavage and subsequent total degradation of the furanone ring forming smaller carbon units (C-1- or C-2-fragments), entering the carbon pool of endogenous compounds and then being used for the biosynthesis of fatty acids.

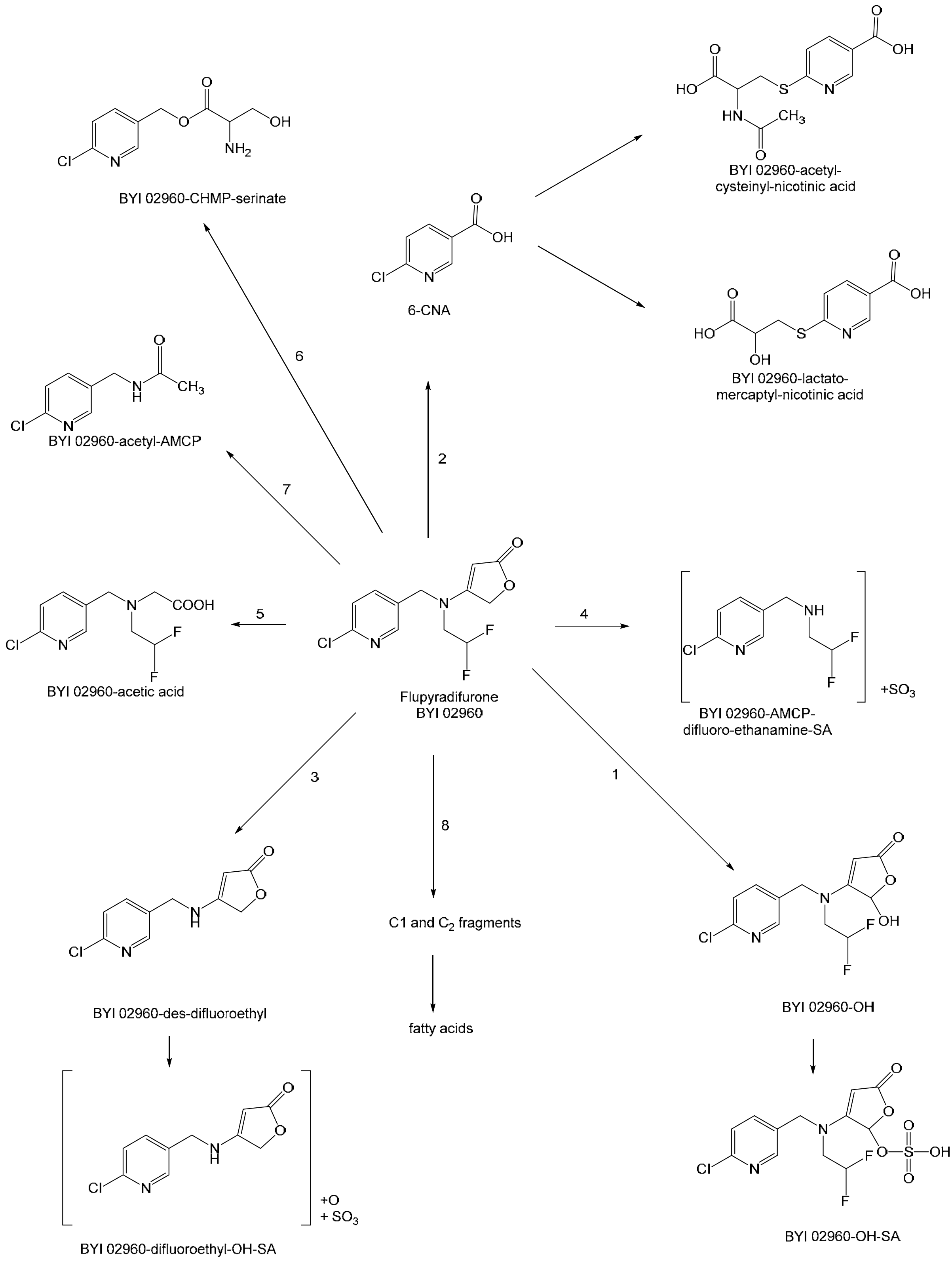


Figure 8 Proposed metabolic pathway for flupyradifurone in laying hens

Summary of animal metabolism

The Meeting received animal metabolism studies with flupyradifurone in rats, hens and goats.

While parent was the main compound detected in excreta of rats (up to 50% of radioactivity in males and 70% in females), flupyradifurone was metabolized to eight identified metabolites and up to 19 unidentified metabolites involving hydroxylation, conjugation and cleavage reactions.

Metabolism in poultry was far more extensive than in ruminants. Parent BYI 02960 was generally a minor component in hen matrices, except for fat (15% of the TRR) and eggs (20% of the TRR) for the pyridinylmethyl label only. Fatty acids were the major metabolic product with the furanone label for eggs, fat, and liver, indicating extensive metabolism and re-incorporation of the radiolabel into natural products. A similar path was seen in ruminant milk. With the pyridinylmethyl label, acetyl-AMCP, from cleavage of both the furanone and difluroethyl groups, was the major residue in eggs (23% TRR), and the major residue in liver was BYI 02960 OH-SA (23% TRR) from hydroxylation of the furanone.

Metabolism in ruminants was very limited. BYI 02960 was the major portion of the residue in fat, muscle, and liver (60–88% TRR for the furanone label and 85–99% TRR for the pyridinylmethyl label). The parent was also the major component in milk with the pyridinylmethyl label (89% TRR), but lactose from total breakdown and reincorporation of the radiolabelled carbon was the major component in milk for the furanone label (67% TRR). Significant metabolism was demonstrated only in kidney. The major metabolites were BYI 02960 OH (15–16% of the TRR) and BYI 02960 OH-glucuronides (up to 9% of the TRR) from hydroxylation followed by conjugation with glucuronic acid.

Difluoroacetic acid would not have been detected in the hen and goat metabolism studies as a labelled residue due to the positions of the radiolabels. No studies were performed with [ethyl-1-^{14}C]-BYI 02960 which would have enabled detection of difluoroacetic acid.

Environmental Fate in Soil

The Meeting received information on soil photolysis, aerobic and anaerobic soil metabolism, field dissipation, adsorption / desorption behaviour in different soils, confined rotational crops and field crop rotation. Only those studies relevant to the current evaluation are reported here.

Soil Photolysis

The phototransformation of [pyridinylmethyl-^{14}C]- and [furanone-^{14}C]- flupyradifurone was studied on a loam soil from Guadalupe, California, USA (pH 6.5, organic carbon 0.6%) for eight days at 20 ± 1 °C (Menke and Unold 2011a, MEF-10/351). The test item dissolved in water/methanol (1:1 v/v) and then further diluted in water, was directly applied as drops to the surface of the soil aliquots at an application rate of approximately 400 g/ha. The treated samples were continuously exposed to artificial irradiation (xenon lamp with < 290 nm cut-off filter, 1082 Wm^{-2} and 1116 Wm^{-2} for [pyridinylmethyl-^{14}C]- label and [furanone-^{14}C]- flupyradifurone experiments respectively. The dark controls which were set up were stored at 22 ± 1 °C. Sampling times were 0, 0.2, 1, 4, 5, 7 and 8 days after treatment (DAT) for both experiments. Samples from systems containing air-dried soil were only taken at the end of the study period.

In the [pyridinylmethyl-^{14}C]- flupyradifurone study, the concentration of the flupyradifurone was determined to be 99% applied radioactivity (AR) at Day 0 and 94% by the end of the study (Day 8). From Day 4 onwards, some very minor transformation products were detected (maximum 1.8% AR, 7 days after treatment). No significant degradation of flupyradifurone occurred in the dark controls with 98% of AR present at the end of the study. No transformation products were detected.

In the [furanone-^{14}C]- flupyradifurone study, the concentration of the flupyradifurone was also determined to be 99% AR at Day 0 and 94% by Day 8. From Day 1 onwards, some very minor transformation products were detected (maximum 1.3% AR, 8 days after treatment). In the dark controls flupyradifurone accounted for 97% of AR present at the end of the study. No transformation products were detected.

No significant differences could be detected between humid and dry soil samples.

Overall degradation of flupyradifurone was very slow. Experimental DT_{50} values of flupyradifurone in the irradiated samples were comparable (99.6 days in the [pyridinylmethyl-^{14}C]-flupyradifurone and 109.3 days in the [furanone-^{14}C]- flupyradifurone study). Corresponding values in the dark controls were > 1000 days and 419.2 days ([pyridinylmethyl-^{14}C]- flupyradifurone and [furanone-^{14}C]- flupyradifurone respectively).

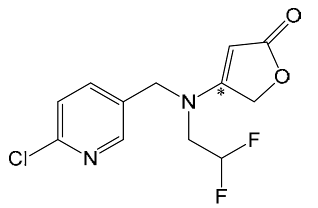
The results show that light has little influence on the behaviour and degradation of flupyradifurone in soil and that a major phototransformation product is not expected.

Aerobic soil metabolism

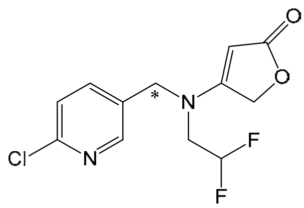
Degradation of flupyradifurone was studied under laboratory aerobic conditions in numerous soils from the USA, Europe and Brazil at temperatures of 20 ± 2 °C. Application rates of radiolabelled flupyradifurone to soils were 200 g/ha (Menke 2011, MEF-07/334), 400–410 g/ha (Menke and Unold 2011b, MEF-10/804; Ripperberger 2012, MERVP037-2; Menke and Unold 2011c, MEF-10/858; Menke and Unold 2011d, MEF-10/880; Shepherd 2012, MERVP038-1) and 1500 g/ha (De Souza 2012, 2301-BS120-342-11).

Test systems consisted of Erlenmeyer flasks equipped with traps for the collection of CO_2 and volatile organic compounds. Soil samples were taken and extracted at various sampling times after treatment. The duration of these studies was 117–120 days.

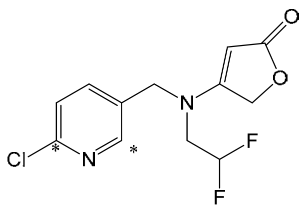
The following four labels were used for these experiments.



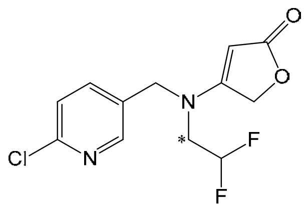
[Furanone-4-^{14}C]
BYI 02960



[Pyridinylmethyl-^{14}C]
BYI 02960



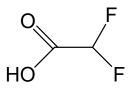
[Pyridine-2,6-^{14}C]
BYI 02960



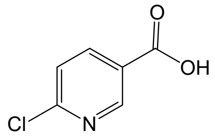
[Ethyl-1-^{14}C]
BYI 02960

* indicates position of radiolabel

In addition, studies were performed with the following metabolites.



Difluoroacetic acid
(DFA)



6-chloronicotininc acid
(6-CNA)

Brief summaries of the metabolism / degradation studies are given below. Best fit DT_{50} and DT_{90} values for various soils determined using various kinetic models are shown in Tables 38–40.

Menke 2011, MEF-07/334 (4 European soils, [pyridinylmethyl-^{14}C] BYI 02960)

The mineralisation of BYI 02960 was high. At the end of the study between 29.4 and 58.6% AR of $^{14}CO_2$ was generated indicating that relatively quick mineralisation of degradates occurred once metabolisation of BYI 02960 was initiated. Volatile organic compounds were very low (≤ 0.1% AR) at all sampling dates. Three very minor metabolites were detected (all < 3% AR) and quantitated together with the test item. Non-extractable residues increased from 1.0–4.1% AR at Day 0 after treatment, to 12.5–16.8% AR at study end.

Menke 2011b, MEF-10/804 (4 European soils, [furanone-4-^{14}C] BYI 02960)

The mineralisation of BYI 02960 was high. At the end of the study between 18.0 and 38.9% AR of $^{14}CO_2$ was generated. Volatile organic compounds were very low (≤ 0.1% AR) at all sampling dates.

Only very minor transformation products (all < 2% AR) were detected in the extracts. Three very minor metabolites were detected in all four soils and were characterised by their retention times. One (maximum of 1.8% AR) was identified as BYI 02960-chloro by spectroscopic methods. Another metabolite (detected once in one soil at a maximum of 0.4% AR) was tentatively assigned as BYI 02960-des-difluoroethyl *via* co-chromatography. Non-extractable residues increased from 2.4–4.6% AR at Day 0 after treatment, to 27.8–34.1% AR at study end.

Ripperger 2012, MERVP037-2 (2 USA soils, [furanone-4-^{14}C] BYI 02960)

BYI 02960 mineralises rapidly under aerobic conditions to CO_2 (12.3% Springfield NE, 36.1% Sanger, CA) by study end. Volatile organic compounds were very low (< 0.1% AR) at all sampling dates. Non-extractable residues increased from 0.2 and 0.8% AR at Day 0 after treatment, to 16.4 and 30.6% AR at study end.

Menke and Unold 2011c, MEF-10/858 (3 European soils, [ethyl-1-^{14}C] BYI 02960)

The mineralisation of BYI 02960 was high. At the end of the study $^{14}CO_2$ accounted for between 25.9 and 42.3% AR. Volatile organic compounds were not detected in significant amounts (< 0.1% AR). Only one major transformation product (maximum values of 22.0–33.9% AR at 45–48 days) was detected in the extracts of all three soils, which was identified as difluoroacetic acid (DFA) *via* HPLC-MS/MS and accurate mass determination. Levels had declined to 16.3–23.8% AR at the end of the study. In addition, one minor transformation product was detected in all three soils at maximum values of 0.4–1.8% AR (90–118 days). Non-extractable residues increased from 2.7–3.2% AR at Day 0 after treatment, to 14.3–17.9% AR at study end.

Menke and Unold 2011d, MEF-10/880 (1 European soil, [pyridine-2, 6-^{14}C] BYI 02960)

BYI 02960 mineralises rapidly under aerobic conditions to CO_2 (57.4% by study end). Volatile organic compounds were not detected in significant amounts (< 0.1% AR). Only very minor transformation products (all ≤ 2.5% AR) were detected in the extracts. One of these, (maximum at 7 days, 2.5% AR), was identified as 6-CNA by co-chromatography and was below the detection limit at the end of the study. Non-extractable residues increased from 3.3% AR at Day 0 after treatment, to 16.2% AR at study end.

Shepherd 2012, MERVP038-1 (2 USA soils, [pyridine-2, 6-^{14}C] BYI 02960)

BYI 02960 mineralises rapidly under aerobic conditions to CO_2 (20.2 and 36.1% by study end). Volatile organic compounds were not detected in significant amounts (< 0.1% AR). Only one major degradate, identified as 6-CNA, was observed (in the sandy loam at a maximum 17.1% AR, 60 days which declined to 8.2% AR at the end of the study). Non-extractable residues increased from 0.1 and 0.7% AR at Day 0 after treatment, to 11.3 and 25.5% AR at study end.

De Souza 2012, 2301-BS120-342-11 (4 Brazilian soils, [pyridine-2, 6-^{14}C] BYI 02960)

BYI 02960 mineralises rapidly under aerobic conditions to CO_2 (2.9–17.5% by study end). Volatile organic compounds were not detected in significant amounts (≤ 0.1% AR). Only one major degradate, identified as 6-CNA, was observed at a maximum 21.5% AR, after 64 days in the Osório soil. It was observed at a maximum of 2–13% AR in the other three soils. Non-extractable residues increased from a maximum of 2.2% AR at Day 0 after treatment, to 11.9–24.2% AR at study end.

Degradation of the flupyradifurone metabolite, 6-chloronicotinic acid (6-CNA), was studied under laboratory aerobic conditions in three soils from England at temperatures of 20 ± 2 °C (Lowden, Oddy and Jones 1997, 11257). The application rates of radiolabelled 6-chloronicotinic acid to soils was 200 g/ha. The duration of the study was 119 days. The major metabolite detected was carbon dioxide (84–92% of the applied dose at the end of the study). Two minor transient metabolites were detected at a maximum of 3% in all soils. The very rapid breakdown of 6-CNA to CO_2 and non-persistent minor metabolites means that it will not persist in aerobic soils.

Table 38 Best fit DT_{50} and DT_{90} values for flupyradifurone (parent) in aerobic soil (20 °C)

SOIL	DT_{50} [days]	DT_{90} [days]	χ^2 Error%	Model	Reference
[Pyridinylmethyl-^{14}C]-Flupyradifurone					
Monheim, Germany [sandy loam, pH 6.4, OC 1.2%]	63.4	443.3	1.17	DFOP	Menke 2011, MEF-07/334 (Dissipation study) Sur and Dorn 2012a, MEF-11/619 (Kinetic modelling analysis)
	169.1		1.17	DFOP	
Burscheid, Germany [silt loam, pH 6.5, OC 1.8%]	52.4	209.3	0.59	DFOP	
	54.4		2.31	SFO	
Burscheid, Germany [loam, pH 5.4, OC 2.3%]	120.0	489.7	1.24	DFOP	
	157.5		1.24	DFOP	
Blankemheim, Germany [clay loam, pH 7.4, OC 4.6%]	56.4	265.1	1.69	DFOP	
	60.1		3.34	SFO	
[Pyridine-2,6-^{14}C]-Flupyradifurone					
Burscheid, Germany [silt loam, pH 6.5, OC 2.4%]	33.0	221.3	2.0	DFOP	Menke and Unold 2011d, MEF-10/880
	90.0			DFOP (pseudo SFO)	Sur and Dorn 2012d, MEF-11/838 (Kinetic modelling analysis)
Springfield, NE, USA [silt loam, pH 6.7, OC 2.3%]	242	898	0.6	DFOP	Shepherd 2011, MERVP038-1
Sanger, CA, USA [sandy loam, pH 7.3, OC 0.6%]	55.3	> 1000	1.7	DFOP	
Piracicaba, Brazil [clay, pH 6.0, OC 2.8%]	203	> 365			de Souza 2012, MERVP038-1
Nova Prata, Brazil [clay, pH 5.2, OC 1.8%]	142	> 365			
Osório, Brazil [sandy loam, pH 4.9, OC 0.2%]	127	> 365			
Viamão, Brazil [silty clay loam, pH 4.1, OC 7.8%]	371	> 365			
[Furanone-4-^{14}C]-Flupyradifurone					
Springfield, NE, USA [silt loam, pH 6.7, OC 2.3%]	374	> 1000	1.1	FOMC	Ripperger 2011,

SOIL	DT_{50} [days]	DT_{90} [days]	χ^2 Error%	Model	Reference
Sanger, CA, USA [sandy loam, pH 7.3, OC 0.6%]	58.3	273	1.1	DFOP	MERVP037-2
	57.2	455	1.1	FOMC	
Monheim, Germany [sandy loam, pH 6.1, OC 2%]	62.2	390.6	1.55	DFOP	Menke and Unold 2011b, MEF-10/804 (Dissipation study) Sur and Dorn 2012b, MEF-11/620 (Kinetic modelling analysis)
	141.5		1.55	DFOP	
Burscheid, Germany [silt loam, pH 6.5, OC 2.5%]	33.2	229.5	1.71	DFOP	
	40.5		5.30	SFO	
Burscheid, Germany [silt loam, pH 4.8, OC 3.3%]	98.3	462.5	2.03	DFOP	
	157.5		2.03	DFOP	
Blankemheim, Germany [silty clay, pH 7.1, OC 4.1%]	49.3	303.1	2.26	DFOP	
	55.1		3.87	SFO	
[Ethyl-1-^{14}C]-Flupyradifurone					
Blankemheim, Germany [clay loam, pH 7.1, OC 5.1%]	33.9	649.6	1.9	DFOP	Menke and Unold 2011c, MEF-10/858 Sur and Dorn 2012c, MEF-11/855 (Kinetic modelling analysis)
	38.6			SFO	
Monheim, Germany [loamy sand, pH 6.2, OC 1.9%]	62.0	538.1	1.6	DFOP	
	210.2			DFOP	
Burscheid, Germany [silt loam, pH 6.5, OC 2.4%]	34.1	329.8	2.3	DFOP	
	43.0			SFO	

OC = Organic Carbon, SFO = Single Phase First Order, FOMC = First Order Multi Compartment, DFOP = Double First Order in Parallel, DFOS = Double First Order in Series

Table 39 Best fit DT_{50} and DT_{90} values for difluoroacetic acid in aerobic soil

SOIL	DT_{50} (day)	DT_{90} (day)	Model used	Reference
Difluoroacetic acid				
Blankemheim, Germany [clay loam, pH 7.1, OC 5.1%]	44.9	149.0	SFO	Sur and Dorn 2012c, MEF-11/855 (Kinetic modelling analysis)
	32.0		SFO	
Monheim, Germany [loamy sand, pH 6.2, OC 1.9%]	73.6	244.5	SFO	
	73.6		SFO	
Burscheid, Germany [silt loam, pH 6.5, OC 2.4%]	67.4	223.9	SFO	
	37.8		SFO	

OC = Organic Carbon, SFO = Single Phase First Order, FOMC = First Order Multi Compartment

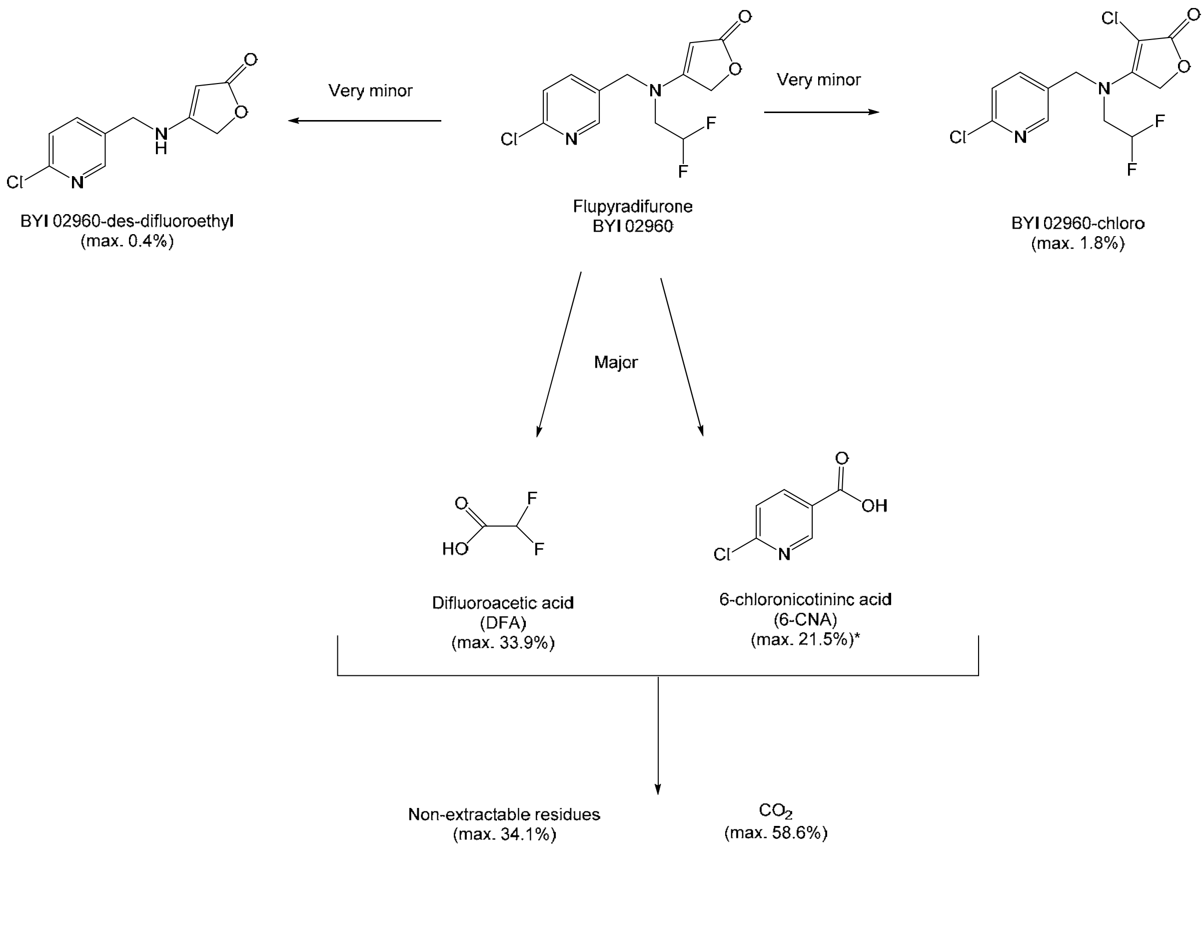
Table 40 Best fit DT_{50} and DT_{90} values for 6-chloronicotinic acid in aerobic soil

SOIL	DT_{50} (day)	DT_{90} (day)	χ^2 Error%	Model used	Reference
[^{14}C] 6-chloronicotinic acid					
Manningtree, Essex, UK [sandy loam, pH 5.8, OC 3.1%]	3.5	8.3			Lowden, Oddy and Jones 1997, 11257 Sur and Dorn 2012e, MEF-11/837 (Kinetic modelling analysis)
	2.9	9.7		SFO	
Royston, Hertfordshire, UK [silty clay loam, pH 7.4, OC 3.8%]	2.9	6.6			
	2.2	7.4		SFO	
Ongar, Essex, UK [clay loam, pH 7.0, OC 1.7%]	6.5	13.3			
	5.3	17.5		SFO	
6-chloronicotinic acid					

SOIL	DT_{50} (day)	DT_{90} (day)	χ^2 Error%	Model used	Reference
Sanger, CA, USA [sandy loam, pH 7.3, OC 0.6%]	36.6	121	13.8	DFOP/SFO	Shepherd 2011, MERVP038-1
Burscheid, Germany [silt loam, pH 6.5, OC 2.4%]	3.1	10.4		SFO	Sur and Dorn 2012d, MEF-11/838 (Kinetic modelling analysis)
	3.0			SFO	

OC = Organic Carbon, SFO = Single Phase First Order, DFOP = Double First Order in Parallel

The route of flupyradifurone degradation in soil was studied using the four different radiolabel positions. The data gathered in the aerobic soil metabolism studies demonstrated that flupyradifurone is degraded in soil. Considering the results from laboratory soil metabolism studies, the major routes of degradation of flupyradifurone under aerobic conditions are cleavage of the difluoroethyl group producing difluoroacetic acid, cleavage of the molecule at the pyridinylmethylene bridge, with subsequent oxidation to 6-chloronicotinic acid and mineralisation to CO_2 and formation of non-extractable residues.



Note: The degradates observed and the maximum values are those for all label positions

* Max 17.1% in USA soils and 21.5% in Brazilian soils. Brazilian soil studies were conducted at higher rate of 1500 g a.i./ha

Figure 9 Proposed metabolic pathway for the aerobic degradation in soil of flupyradifurone

Field dissipation studies

The degradation behaviour (field dissipation) of flupyradifurone was investigated at various sites in Europe, Canada and the USA.

Europe

An SL formulation was applied to bare soil in a single application at an application rate of 1.25 L/ha and using a target water volume of 300 L/ha (knapsack/boom sprayer) corresponding to 250 g flupyradifurone/ha at six sites; Monheim (Germany), Great Chishill (United Kingdom), Burscheid (Germany), Albaro (Italy), Vilobi d'Onyar (Spain) and Burscheid/ Hanscheider Hof (Germany) (Heinemann 2011b, 09-2702). Soil specimens were taken at intervals up to 540 days post-application and down to a maximum depth of 100 cm. All soil specimens were frozen until analysis. Soil specimens were analysed for flupyradifurone and the metabolite BYI 02960-DFA using Method 01074/M001. The LOQ was 0.005 mg/kg for each analyte and the LOD 0.0015 mg/kg.

Monheim (Germany)—Flupyradifurone was observed in the 0–10 cm soil depth at 237 g/ha at 0 DALA (95% of the nominal application rate), which declined to 35.6 g/ha at Day 545 (15% of the applied amount). Under field conditions at Monheim, flupyradifurone had a DT_{50} of 41.0 days and a DT_{90} of 749 days.

Great Chishill (United Kingdom)—Flupyradifurone was observed in the 0–10 cm soil depth at 236 g/ha at 0 DALA, which declined to 70.2 g/ha at Day 552 (29.8% of the applied amount). Under field conditions at Great Chishill, flupyradifurone had a DT_{50} of 251 days and a DT_{90} of > 1000 days.

Burscheid (Germany)—Flupyradifurone was observed in the 0–10 cm soil depth at 245 g/ha at 0 DALA, which declined to 17.9 g/ha at Day 540 (7.3% of the applied amount). Under field conditions at Burscheid, flupyradifurone had a DT_{50} of 42.8 days and a DT_{90} of 484 days.

Albaro (Italy)—Flupyradifurone was observed in the 0–10 cm soil depth at 268 g/ha at 0 DALA, which declined to 7.8 g/ha at Day 547 (2.9% of the applied amount). Under field conditions at Burscheid, flupyradifurone had a DT_{50} of 8.3 days and a DT_{90} of 279 days.

Vilobi d'Onyar (Spain)—Flupyradifurone was observed in the 0–10 cm soil depth at 240 g/ha at 0 DALA, which declined to 8.8 g/ha at Day 533 (3.7% of the applied amount). Under field conditions at Vilobi d'Onyar, flupyradifurone had a DT_{50} of 22.6 days and a DT_{90} of 215 days.

Burscheid/ Hanscheider Hof (Germany)—Flupyradifurone was observed in the 0–10 cm soil depth at 229 g/ha at 0 DALA, which declined to 20.5 g/ha at Day 540 (9.0% of the applied amount). Under field conditions at Burscheid/ Hanscheider Hof, flupyradifurone had a DT_{50} of 39.0 days and a DT_{90} of 579 days.

In summary, the dissipation of flupyradifurone showed a biphasic behaviour. After treatment it dissipated in a very fast first step within one month, followed by a second slower step until the end of the study. Residues remained in the upper 0–20 cm soil layer and only very small amounts (< LOQ) were translocated to a maximum depth of 30 cm, except for at Vilobi d'Onyar, Spain where flupyradifurone residues, just greater than the LOQ, were found at Day 13 to a depth of 30 cm.

At study completion flupyradifurone residues in soil corresponded to 2.9–29.8% of the applied amount. Flupyradifurone dissipated from soil at all test sites with DT_{50} values ranging from 8.3 to 251 days.

The metabolite DFA appeared in the upper soil layer 0–20 cm in amounts of up to 17.4 g/ha which always decreased towards the end of the study (Table 42).

Table 41 Dissipation rates of flupyradifurone in the European terrestrial field dissipation study

Site [parameters at 0–30 cm]	DT_{50} [days]	DT_{90} [days]	χ^2 Error%	Model	Reference
Monheim, Germany [sandy loam, pH 6.3, OC 1.2%, av. temp. range 1–23 °C]	41.0	749	7.5	DFOP	Heinemann 2011 09-2702
Great Chishill, United Kingdom [clay loam, pH 5.8, OC 2.2%, av. temp. range 1–18 °C]	251	> 1000	7.5	DFOP	

Site [parameters at 0–30 cm]	DT_{50} [days]	DT_{90} [days]	χ^2 Error%	Model	Reference
Burscheid, Germany [silt loam, pH 6.3, OC 0.9%, av. temp. range –3–19 °C]	42.8	484	6.3	DFOP	
Albaro, Italy [loam, pH 7.4, OC 1.3%, av. temp. range 2–26 °C]	8.3	279	7.1	DFOP	
Viloba d'Onyar, Spain [loam, pH 5.9, OC 0.7%, av. temp. range 5–24 °C]	22.6	215	6.6	DFOP	
Burscheid/Hanscheider Hof, Germany [loam, pH 5.5, OC 1.5%, av. temp. range –3–19 °C]	39.0	579	11.3	DFOP	

OC = Organic Carbon, DFOP = Double First Order in Parallel

Table 42 Mean sum values (g/ha) of flupyradifurone and DFA in the European terrestrial field dissipation study

	Monheim, Germany	0 days	6	12	29	60	90	123	190	354	432	545 days
BYI02960	0–100 cm	237	230	170	120	106	105	92.1	71.8	64.7	39.4	35.6
DFA	0–100 cm	104	3.64	7.00	6.10	8.80	12.4	11.2	10.8	7.80	7.10	5.40
	Great Chishill, United Kingdom	0 days	7	14	30	68	96	128	204	394	478	552 days
BYI02960	0–100 cm	236	162	155	183	177	144	136	105	106	89.5	70.2
DFA	0–100 cm	0	0	1.11	4.02	6.68	7.90	8.30	7.38	5.50	5.70	5.50
	Burscheid, Germany	0 days	6	12	29	60	91	124	197	357	439	540 days
BYI02960	0–100 cm	245	208	172	143	112	109	69.9	59.3	54.3	25.2	17.9
DFA	0–100 cm	0	1.37	4.25	4.79	10.9	11.4	8.80	10.9	10.7	11.1	7.90
	Albaro, Italy	0 days	7	14	28	60	91	119	177	360	441	547 days
BYI02960	0–100 cm	268	142	112	105	60.8	77.5	50.6	50.1	20.6	11.6	7.83
DFA	0–100 cm	0	1.30	6.50	9.19	10.0	9.25	7.89	9.16	3.99	1.40	0
	Viloba d'Onyar, Spain	0 days	7	13	29	63	91	111	181	357	440	533 days
BYI02960	0–100 cm	240	146	128	119	92.1	57.6	49.1	32.5	22	10.7	8.80
DFA	0–100 cm	1.14	5.33	5.75	7.46	6.91	7.10	5.40	4.90	1.20	0	0
	Burscheid/Hanscheider Hof, Germany	0 days	6	12	29	60	92	124	197	357	439	540 days
BYI02960	0–100 cm	229	254	178	126	96.3	63.8	63.8	53.6	39.6	31.4	20.5
DFA	0–100 cm	0	2.22	6.89	8.09	17.4	8.75	10.4	8.95	7.88	6.61	2.17

Canada

An SL formulation was applied to bare soil in single application at an application rate of 1.25 L/ha and using a target water volume of 300 L/ha (tractor mounted sprayer) corresponding to a nominal rate of 600 g flupyradifurone/ha at three sites; Odessa, Saskatchewan, Clarksburg, Ontario and New Glasgow, Prince Edward Island (Harbin 2012, MERVP055). The actual application rates were 598–639 g flupyradifurone/ha. Soil specimens were taken from control and treated plots to a depth of 15 cm immediately after application (0 DAT). Additional samples were taken from the treated plots at nominal sampling times of 3, 7, 14, 30, 60, 120, 330, 390 and 450 DAT at depths of 0 to 105 cm. All soil specimens were frozen until analysis. Soil specimens were analysed for flupyradifurone and the significant metabolites BYI 02960-DFA and 6-CNA using Method 01074/M001. The LOQ was 0.005 mg/kg for each analyte.

Odessa, Saskatchewan—Flupyradifurone was observed in the 0–15 cm soil depth at a 0.217 mg/kg at 0 DAT (66% of theoretical applied rate of 600 g ai/ha or 0.330 mg/kg), which declined to 0.104 mg/kg at Day 484 (48% of the 0-day amount). Under field conditions at Odessa, flupyradifurone had a DT_{50} of 234 days and a DT_{75} of > 1000 days.

Clarksburg, Ontario—Flupyradifurone was observed in the 0–15 cm soil depth at 0.275 mg/kg at 0 DAT, (74% of theoretical applied rate of 600 g ai/ha or 0.372 mg/kg) which declined to 0.0344 mg/kg at Day 450 (13% of the 0-day amount). Under field conditions at Clarksburg, flupyradifurone had a DT_{50} of 94.9 days and a DT_{75} of 352 days.

New Glasgow, Prince Edward Island—Flupyradifurone was observed in the 0–15 cm soil depth at 0.186 mg/kg at 0 DAT, (47% of theoretical applied rate of 600 g ai/ha or 0.394 mg/kg) which declined to 0.0379 mg/kg at Day 450 (20% of the 0-day amount). Under field conditions at Glasgow, flupyradifurone had a DT_{50} of 83.0 days and a DT_{75} of 334 days.

At all three sites, average residues of flupyradifurone greater than the LOQ of 0.005 mg/kg were detected in the 15–30 cm soil depth by a nominal 7 DAT and were observed intermittently at concentrations ranging from 0.0051–0.0153 mg/kg through to 401 DAT. No residues of flupyradifurone were observed at depths greater than 30 cm.

DFA residues greater than the LOQ of 0.005 mg/kg were detected in the 0–15 cm soil depth at the Ontario and Prince Edward Island sites with a maximum DFA residue of 0.019 mg/kg found at the Ontario site at 59 DAT. No DFA residues greater than the LOQ were observed at the Saskatchewan site. No DFA residues greater than the LOQ were observed at any of the sites at depths greater than 15 cm.

No 6-CNA residues greater than the LOQ were observed at any site.

In summary, at the Ontario and Prince Edward Island sites, based on DT_{75} values of less than a year and ≤ 20% of the initial flupyradifurone remaining at the end of the monitoring period, there is some potential for flupyradifurone to carry over into the following year. At the Saskatchewan site, where the DT_{75} was greater than 1000 days and 48% of the initial flupyradifurone remained at the end of the monitoring period, there is probably potential for flupyradifurone carry over into the following year.

In all three Canadian soils, flupyradifurone and its metabolites were found primarily in the 0–15 cm sampling level. Flupyradifurone degraded to DFA, to a lesser extent to 6-CNA and to bound residues, followed by mineralisation to CO_2.

Table 43 Dissipation rates of flupyradifurone in the Canadian terrestrial field dissipation study

Site [parameters at 0–15 cm]	DT_{50} [days]	DT_{75} [days]	χ^2 Error%	Model	Reference
Odessa, Saskatchewan [loam, pH 7.6, OC 1.4%]	234	> 1000	8.4	DFOP	Harbin 2012 MERVP055
Clarksburg, Ontario [sandy loam, pH 7.0, OC 2.3%]	94.9	352	10.0	DFOP	
New Glasgow, Prince Edward Island [sandy loam, pH N/A, OC 2.2%]	83.0	334	10.1	FOMC	

OC = Organic Carbon, FOMC = First Order Multi Compartment, DFOP = Double First Order in Parallel

Table 44 Average concentrations (ng/g) of flupyradifurone, DFA and 6-CNA found in four subplots (mg/kg, dry weight) in the Canadian terrestrial field dissipation study

	Ontario	–3 days	0	3	7	14	30	59	120	337	381	450 days
BYI02960	0–15 cm		275.2	245.5	240.8	173.7	173.8	127.0	159.1	63.1	66.1	34.4
	15–30 cm				5.1		(2.2)	(2.5)	6.5	5.7	(3.7)	(1.8)
DFA	0–15 cm					(4.4)	8.7	10.9	7.2	(4.5)	7.8	5.2
	15–30 cm								(1.9)			
6-CNA	0–15 cm				(3.8)	(3.3)	(3.7)					
	Prince Edward Island	–5 days	0	3	7	14	29	62	125	345	392	455 days
BYI02960	0–15 cm		186.4	220.2	219.1	160.9	141.2	115.6	86.4	57.7	49.1	37.9
	15–30 cm			(1.6)	7.7	7.5	(3.0)		(3.2)	(3.8)		
DFA	0–15 cm						6.7	8.0	(3.2)			(2.3)
	15–30 cm							(2.0)	(2.7)			

	Ontario	–3 days	0	3	7	14	30	59	120	337	381	450 days
6-CNA	0–15 cm				(4.8)	(3.9)						
	Saskatchewan	–4 days	0	3	7	14	30	60	120	330	390	450 days
BYI02960	0–15 cm		216.7	204.0	238.5	213.5	186.8	137.1	117.8	121.2	105.2	103.6
	15–30 cm				15.3	(2.5)	10.4		(2.5)	5.9	5.5	(2.2)

Results in parentheses are below the method LOQ

USA

Field dissipation studies were conducted at Tulare County, California (Lenz M. 2012a, MERVY001), Jackson County, Florida (Lenz M. 2012b, MERVY002) and Blaine County, Idaho (Lenz M. 2012c, MERVP028). In each study an SL formulation was applied to bare soil in a single application at a target rate of 600 g flupyradifurone/ha.

Sampling occurred at various times up to 535 DAT (California), 363 DAT (Florida) and 364 DAT (Idaho). Each soil sampling (four cores from a pre-determined randomly selected sub-section from each sub-plot) consisted of two soil cores, a 0–15 cm core and a 15–120 cm core. Only a 0–15 cm core was collected at 0-day. The 15–120 cm cores were segmented into approximately 15 cm sections. Segments from the same depth from a subplot were composited into one sample per depth. All soil specimens were frozen until analysis.

Soil specimens were analysed for flupyradifurone and the significant metabolites difluoroacetic acid (DFA) and 6-chloronicotinic acid (6-CNA) using LC-MS/MS. The LOQ was 0.005 mg/kg for each analyte.

Tulare County, California—Flupyradifurone was observed in the 0–15 cm soil depth at a maximum mean concentration of 0.2006 mg/kg at 0 DAT, which declined to 0.0267 mg/kg at Day 535 (13% of the 0-day amount). Major transformation products were 6-chloronicotinic acid and difluoroacetic acid. The maximum mean concentration of DFA found in the total soil core was 0.0146 mg/kg or 21.9% of the applied dose as parent equivalents. The maximum total mean concentration of 6-CNA was 0.0069 mg/kg or 6.3% of the applied amount as parent equivalents. Flupyradifurone residues were found to a soil depth of 30 cm while residues of 6-chloronicotinic acid and difluoroacetic acid were found greater than LOQ at a soil depth of 15 and 30 cm respectively. Under field conditions at Tulare County, flupyradifurone had a SFO DT_{50} of 125 days and a DT_{75} of 248 days and a DFOP DT_{50} of 57 days and DT_{75} of 327 days.

Jackson County, Florida—Flupyradifurone was observed in the 0–15 cm soil depth at a mean concentration of 0.3045 mg/kg at 0 DAT, which declined to 0.0544 mg/kg at Day 363 (18% of the 0-day amount). Major transformation products were 6-chloronicotinic acid and difluoroacetic acid. The maximum mean concentration of difluoroacetic acid found in the total soil core was 0.0052 mg/kg or 5.1% of the applied amount as parent equivalents. The maximum total mean concentration of 6-chloronicotinic acid was 0.0082 mg/kg or 4.9% of the applied amount as parent equivalents. Flupyradifurone residues were found to a soil depth of 15 cm (one sample at the method LOD from 15–30 cm). Chloronicotinic acid and difluoroacetic acid were observed at a soil depth of 15 cm, with sporadic detections of difluoroacetic acid below the LOQ to a depth of 60 cm. Under field conditions at Jackson County, flupyradifurone had a SFO DT_{50} of 77.1 days and a DT_{75} of 154 days and a FOMC DT_{50} of 9.3 days and a DT_{75} of 122 days.

Blaine County, Idaho—Flupyradifurone was observed in the 0–15 cm soil depth at a mean concentration of 0.4165 mg/kg at 0 DAT, which declined to 0.0864 mg/kg at Day 364 (21% of the 0-day amount). Major transformation products were 6-chloronicotinic acid and difluoroacetic acid. The maximum mean concentration of difluoroacetic acid found in the total soil core was 0.018 mg/kg or 7.8% of the applied amount as parent equivalents. The maximum total mean concentration of 6-chloronicotinic acid was 0.0134 mg/kg or 5.9% of the applied amount as parent equivalents. Flupyradifurone residues were found greater than the method LOD to a soil depth of 30 cm.

Chloronicotinic acid and difluoroacetic acid were observed at greater than the method LOD at a soil depth of 60 cm and 90 cm respectively, and greater than the LOQ at a soil depth of 15 and 30 cm respectively. Under field conditions at Blaine County, flupyradifurone had a SFO DT_{50} of 152 days and a DT_{75} of 301 days and a DFOP T_{50} of 49 days and a DT_{75} of 272 days.

All residues of flupyradifurone, DFA and 6-CNA above the LOQs of 0.005 mg/kg were detected in the top 0–30 cm of soil.

The field dissipation studies showed that the major observed routes of dissipation for flupyradifurone are biodegradation to 6-chloronicotinic acid and difluoroacetic acid and non-extractable residues followed by mineralisation to carbon dioxide.

Table 45 Dissipation rates of flupyradifurone in the USA terrestrial field dissipation studies

Site [parameters at 0–15 cm]	DT_{50} [days]	DT_{75} [days]	χ^2 Error%	Model	Reference
Tulare County, California [loam, pH 8.2, OC 0.9%]	57	327	4.3	DFOP	Lenz 2012a MERVY001
Jackson County, Florida [loamy sand, pH 6.5, OC 0.8%]	9.3	122	7.1	FOMC	Lenz 2012b MERVY002
Blaine County, Idaho [loam, pH 8.0, OC 1.4%]	49	272	8.0	DFOP	Lenz 2012c MERVP028

OC = Organic Carbon, DFOP = Double First Order in Parallel, FOMC = First Order Multi Compartment

Table 46 Average concentrations (ng/g) of flupyradifurone, DFA and 6-CNA found in four subplots (mg/kg, dry weight) in the USA terrestrial field dissipation studies

	California	–4 days	0	3	7	14	30	62	90	119	181	272	366	535 days
Parent	0–15 cm		200.6	190.5	186.1	147.3	131.6	93.7	86.5	75.2	69.0	56.5	53.9	26.7
	15–30 cm								(2.1)	(2.2)	5.4			
DFA	0–15 cm			(1.7)	(3.6)	5.4	12.4	8.4	5.0	6.4	(4.6)		(2.7)	(2.9)
	15–30 cm							6.2	(3.6)	(2.6)	(2.8)			
	30–45 cm						(2.2)				(2.6)			
	45–60 cm												(1.7)	
6–CNA	0-15 cm			(3.0)	6.9	5.6								
	Florida	–1 days	0	3	7	14	30	59	92	121	189	275	363 days	
Parent	0–15 cm		304.5	185.7	180.6	144.1	109.1	69.6	84.0	81.3	76.0	59.1	54.4	
DFA [a]	0–15 cm			(3.0)	(4.6)	5.2	(3.5)		(2.5)					
6-CNA	0–15 cm			5.3	8.2	7.1	(2.2)							
	Idaho	–1 days	0	3	9	14	30	61	89	124	156	280	364 days	
Parent	0–15 cm		416.5	264.6	277.1	232.6	193.6	199.8	168.1	144.1	126.8	119.6	86.4	
	0–30 cm									7.4	5.4	(4.4)	(4.0)	
DFA	0–15 cm				(2.5)	(4.3)	5.0	(3.7)						
	15–30 cm							(4.4)	(3.4)	(1.6)				
	30–45 cm							(2.7)	(2.9)					
	45–60 cm								(2.2)					
	60–75 cm													
	75–90cm									(1.7)				
6-	0–15 cm			(4.1)	10.8	13.4	10.8							

	California	–4 days	0	3	7	14	30	62	90	119	181	272	366	535 days
CNA														

Results in parentheses are below the method LOQ

[a] Control reading of 1.7 ng/g

Field accumulation in rotational crops

Field rotational crop studies for flupyradifurone were conducted in several European countries.

In 2010–11, four trials (one each in Germany, the Netherlands, Spain, and southern France) were conducted to determine the residues of flupyradifurone in rotated field crops (following crops) after spray application of an SL formulation containing 200 g flupyradifurone /L (Schoening and Bauer 2012, 10-2503). A single application was made at a nominal rate of 1 L/ha, corresponding to 200 g ai/ha. Water rates were 300–400 L/ha. Applications were either made to bare soil or to lettuce (primary crop or target crop). In the latter case, the crop was then harvested from the field, remaining plants were incorporated into the soil, and rotational crops were planted.

At various intervals, crops were planted back onto the test area to simulate a crop failure ("rotation 1", plant-back interval [PBI] 25–33 days), a second use of the plot in the same year ("rotation 2", PBI 60–200 days), or use of the same plot in the succeeding year ("rotation 3", PBI 260–330 days). In each rotation, a root crop (carrots or turnips), a leafy crop (lettuce), or a cereal (barley) were planted or sown at each interval.

Samples of the rotational crops were taken at their respective harvest times, as well as at one earlier interval (immature RACs for lettuce and root crops, or fodder ["green material"] for barley). Samples were stored frozen until analysis which was within 540 days. Samples were analysed for parent, difluoroacetic acid (DFA), BYI 02960-difluoroethylaminofuranone (DFEAF) and 6-CNA by LC/MS/MS method 01304. The LOQs were 0.01 mg/kg for parent and DFEAF and 0.02 mg/kg for DFA (except for barley straw (0.05 mg/kg) as parent and 0.005 mg/kg for 6-CNA as itself. Mean recoveries from fortified control samples were within acceptable limits (71–108%). Residues found in samples from treated plots are summarized below in Tables 48–50.

Table 47 Results from European rotational field trials for root crops

Target Crop, Country	Rotational Crop, Variety (rotation information)	Portion analysed	PBI	DALT (days)	Residues (mg/kg) expressed as parent				CNA (mg/kg)
					BYI 02960 (parent)	difluoro-acetic acid (DFA)	BYI 02960-difluoro-ethylamino-furanone (DFEAF)	Parent + DFA + CNA	
Soil, Germany	Carrot, Cestas F1 (Rotation 1)	root	25	95 109	< 0.01 < 0.01	0.03 0.03	< 0.01 < 0.01	0.04 0.04	< 0.005 < 0.005
Lettuce, Germany	Carrot, Cestas F1 (Rotation 2)	root	70	150 164	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	< 0.03 < 0.03	< 0.005 < 0.005
Lettuce, Germany	Carrot, Cestas (Rotation 3)	root	284	396 410	< 0.01 < 0.01	0.03 0.04	< 0.01 < 0.01	0.04 0.05	< 0.005 < 0.005
Soil, Netherlands	Carrot, Nerja (Rotation 1)	root	25	92 106	< 0.01 < 0.01	0.03 0.05	< 0.01 < 0.01	0.04 0.06	< 0.005 < 0.005
Lettuce, Netherlands	Carrot, Nerja (Rotation 2)	root	61	151 165	< 0.01 < 0.01	0.03 0.03	< 0.01 < 0.01	0.04 0.04	< 0.005 < 0.005
Lettuce, Netherlands	Carrot, Nerja (Rotation 3)	root	328	406 420	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	< 0.03 < 0.03	< 0.005 < 0.005
Soil, France	Turnip, edible	body	25	82 96	< 0.01 < 0.01	0.12 0.10	< 0.01 < 0.01	0.13 0.11	< 0.005 < 0.005

Target Crop, Country	Rotational Crop, Variety (rotation information)	Portion analysed	PBI	DALT (days)	Residues (mg/kg) expressed as parent				CNA (mg/kg)
					BYI 02960 (parent)	difluoro-acetic acid (DFA)	BYI 02960-difluoro-ethylamino-furanone (DFEAF)	Parent + DFA + CNA	
	Aramis (Rotation 1)	leaf	25	82 96	0.03 0.02	0.20 0.07	< 0.01 < 0.01	0.23 0.09	< 0.005 < 0.005
Lettuce, France	Turnip, edible Aramis (Rotation 2)	body	70	127 141	< 0.01 < 0.01	0.05 0.04	< 0.01 < 0.01	0.06 0.05	< 0.005 < 0.005
		leaf	70	127 141	0.02 < 0.01	0.08 0.03	< 0.01 < 0.01	0.10 0.04	< 0.005 < 0.005
Lettuce, France	Turnip, edible Aramis (Rotation 3)	body	314	380 394	< 0.01 < 0.01	0.04 0.03	< 0.01 < 0.01	0.05 0.04	< 0.005 < 0.005
		leaf	314	380 394	0.03 0.04	0.11 0.16	< 0.01 < 0.01	0.14 0.20	< 0.005 < 0.005
Soil, Spain	Carrot, Coral Nantesa (Rotation 1)	root	30	100 114	< 0.01 < 0.01	0.05 0.06	< 0.01 < 0.01	0.06 0.07	< 0.005 < 0.005
Lettuce, Spain	Carrot, Coral Nantesa (Rotation 2)	root	145	314 328	< 0.01 < 0.01	0.02 0.03	< 0.01 < 0.01	0.03 0.04	< 0.005 < 0.005
Lettuce, Spain	Carrot, Coral Nantesa (Rotation 3)	root	279	349 363	< 0.01 < 0.01	< 0.02 0.03	< 0.01 < 0.01	< 0.03 0.04	< 0.005 < 0.005

Table 48 Results from European rotational field trials for leafy vegetables

Plot No Country	Rotational Crop, Variety (rotation information)	Portion analysed	PBI	DALT (days)	Residues (mg/kg) expressed as parent				CNA (mg/kg)
					BYI 02960 (parent)	difluoro-acetic acid (DFA)	BYI 02960-difluoro-ethylamino-furanone (DFEAF)	Parent + DFA + CNA	
Soil, Germany	Lettuce, Argentines, Butterhead variety (Rotation 1) PBI 25 days	Head	25	63 77	0.01 < 0.01	0.06 0.05	< 0.01 < 0.01	0.07 0.06	< 0.005 < 0.005
Lettuce, Germany	Lettuce, Argentines, Butterhead variety (Rotation 2) PBI 77 days	Head	77	124 138	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	< 0.03 < 0.03	< 0.005 < 0.005
Lettuce, Germany	Lettuce, Aleppo, Loose leaf variety (Rotation 3) PBI 320 days	Head	320	354 368	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	< 0.03 < 0.03	< 0.005 < 0.005

Plot No Country	Rotational Crop, Variety (rotation information)	Portion analysed	PBI	DALT (days)	Residues (mg/kg) expressed as parent				CNA (mg/kg)
					BYI 02960 (parent)	difluoro-acetic acid (DFA)	BYI 02960-difluoro-ethylamino-furanone (DFEAF)	Parent + DFA + CNA	
Soil, Netherlands	Lettuce, Lucan, Butterhead variety (Rotation 1)	Head	25	46 60	0.03 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	0.05 < 0.03	< 0.005 < 0.005
Lettuce, Netherlands	Lettuce, Lucan, Butterhead variety (Rotation 2)	head	64	95 109	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	< 0.03 < 0.03	< 0.005 < 0.005
Lettuce, Netherlands	Lettuce, Lucan, Butterhead variety (Rotation 3)	head	329	358 372	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	< 0.03 < 0.03	< 0.005 < 0.005
Soil, France	Lettuce, Pitice, Loose leaf (Rotation 1)	head	28	55 69	0.08 0.03	0.12 0.11	< 0.01 < 0.01	0.20 0.14	< 0.005 < 0.005
Lettuce, France	Lettuce, Pitice, Loose leaf (Rotation 2)	head	70	107 121	0.03 0.01	0.08 0.09	< 0.01 < 0.01	0.11 0.10	< 0.005 < 0.005
Lettuce, France	Lettuce, Pitice, Loose leaf (Rotation 3)	head	314	357 371	0.02 < 0.01	0.08 0.05	< 0.01 < 0.01	0.10 0.06	< 0.005 < 0.005
Soil, Spain	Lettuce, Murai, (leafy variety) (Rotation 1)	head	30	72 85	0.03 0.01	0.03 0.03	< 0.01 < 0.01	0.06 0.04	< 0.005 < 0.005
Lettuce, Spain	Lettuce, Pelican, Butterhead (Rotation 2)	head	145	291 305	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	< 0.03 < 0.03	< 0.005 < 0.005
Lettuce, Spain	Lettuce, Murai (leafy variety) (Rotation 3)	head	279	321 334	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	< 0.03 < 0.03	< 0.005 < 0.005

Table 49 Results from European rotational field trials for cereals

Plot No	Rotational	Portion	DALT	Residues (mg/kg) expressed as parent	CNA

Country	Crop, Variety (rotation information)	analysed	(days)	BYI 02960 (parent)	difluoro-acetic acid (DFA)	BYI 02960-difluoro-ethylamino-furanone (DFEAF)	Parent + DFA (+ CNA for grain)	(mg/kg)
Soil, Germany	Barley, Simba (Rotation 1) PBI 25 days	green material	56	0.01	0.11	< 0.01	0.12	< 0.005
		grain	116	< 0.01	0.33	< 0.01	0.34	< 0.005
		straw	116	0.02	0.13	< 0.01	0.15	< 0.005
Lettuce, Germany	Barley, Leibnitz (Rotation 2) PBI 116 days	green material	305	< 0.01	0.03	< 0.01	0.04	< 0.005
		grain	406	< 0.01	0.10	< 0.01	0.11	< 0.005
		straw	406	< 0.01	< 0.05	< 0.01	< 0.06	< 0.005
Lettuce, Germany	Barley, Simba (Rotation 3) PBI 284 days	green material	333	< 0.01	< 0.02	< 0.01	< 0.03	< 0.005
		grain	420	< 0.01	0.10	< 0.01	0.11	< 0.005
		straw	420	< 0.01	< 0.05	< 0.01	< 0.06	< 0.005
Soil, Netherlands	Barley, Cervoise (Rotation 1) PBI 33 days	green material	226	< 0.01	0.04	< 0.01	0.05	< 0.005
		straw	329	< 0.01	< 0.05	< 0.01	< 0.06	< 0.005
Lettuce, Netherlands	Barley, Cervoise (Rotation 2) PBI 137 days	green material	330	< 0.01	0.03	< 0.01	0.04	< 0.005
		grain	446	< 0.01	0.04	< 0.01	0.05	< 0.005
		straw	446	< 0.01	< 0.05	< 0.01	< 0.06	< 0.005
Lettuce, Netherlands	Barley, Tripple (Rotation 3) PBI 295 days	green material	350	< 0.01	0.02	< 0.01	0.03	< 0.005
		grain	459	< 0.01	0.06	< 0.01	0.07	< 0.005
		straw	459	< 0.01	< 0.05	< 0.01	< 0.06	< 0.005
Soil, France	Barley, Attraction (Rotation 1) PBI 27 days	green material	126	0.02	0.37	< 0.01	0.39	< 0.005
		grain	218	< 0.01	0.63	< 0.01	0.64	< 0.005
		straw	218	0.04	0.34	< 0.01	0.38	0.01
Lettuce, France	Barley, Attraction (Rotation 2) PBI 195 days	green material	294	0.01	0.10	< 0.01	0.11	< 0.005
		grain	386	< 0.01	0.26	< 0.01	0.27	< 0.005
		straw	386	0.02	0.10	< 0.01	0.12	0.01
Lettuce, France	Barley, Attraction (Rotation 3) PBI 290 days	green material	324	0.03	0.15	< 0.01	0.18	< 0.005
		grain	419	< 0.01	0.37	< 0.01	0.38	< 0.005
		straw	419	< 0.01	0.17	< 0.01	0.18	0.006
Soil, Spain	Barley, Graphic (Rotation 1) PBI 27 days	green material	133	< 0.01	0.03	< 0.01	0.04	< 0.005
		grain	233	< 0.01	0.09	< 0.01	0.10	< 0.005
		straw	233	0.01	0.05	< 0.01	0.06	0.02

Plot No Country	Rotational Crop, Variety (rotation information)	Portion analysed	DALT (days)	Residues (mg/kg) expressed as parent				CNA (mg/kg)
				BYI 02960 (parent)	difluoro-acetic acid (DFA)	BYI 02960-difluoro-ethylamino-furanone (DFEAF)	Parent + DFA (+ CNA for grain)	
Lettuce, Spain	Barley, Graphic (Rotation 2) PBI 143 days	green material	249	< 0.01	0.03	< 0.01	0.04	< 0.005
		grain	349	< 0.01	0.09	< 0.01	0.10	< 0.005
		straw	349	< 0.01	< 0.05	< 0.01	< 0.06	0.009
Lettuce, Spain	Barley, Graphic (Rotation 3) PBI 266 days	green material	306	< 0.01	0.02	< 0.01	0.03	< 0.005
		grain	363	< 0.01	0.09	< 0.01	0.10	< 0.005
		straw	363	< 0.01	< 0.05	< 0.01	< 0.06	< 0.005

Parent flupyradifurone was absent (< 0.01 mg/kg) in barley grain at a PBI of about 30 days and in turnip root and carrot at a PBI of about 25 days. Parent was found in turnip tops (0.04 mg/kg) at a PBI of about 300 days. It was generally absent (< 0.01 mg/kg) in head lettuce at all PBIs, but found in leaf lettuce at PBIs up to 314 days (up to 0.08 mg/kg) and was non-quantifiable to 0.04 mg/kg in barley forage and barley straw at all PBIs.

Difluoroethyl-amino-furanone (DFEAF) was non-quantifiable in all commodities at all PBIs. DFA was quantifiable in most commodities at all PBIs (up to 0.63 mg/kg in cereal grain and up to 0.12 mg/kg in lettuce and root crops).

Residues of 6-CNA were not detected (< 0.005 mg/kg) in samples of carrot, turnip, lettuce, barley grain and barley green material from the European field rotational trials. Low residues of 6-CNA were detected in some barley straw samples.

A number small (single crop, single rotation) field rotational studies were conducted in northern and southern Europe in 2011 with potatoes (Schulte 2012a, 11-2550), leeks (Schulte 2012b, 11-2551), cucumber (Schulte 2012c, 11-2552), onion (Schulte 2012d, 11-2553), French bean (Schulte 2012e, 11-2555), pea (Schulte 2012f, 11-2556) and winter rape (Schulte 2012g, 11-2554). Two applications of flupyradifurone were made to bare soil, each at 125 g ai/ha using an SL formulation containing 200 g/L BYI 02960. The interval between applications was approximately 10 days. Rotational crops were planted approximately 30 days after the last application. Samples were taken at typical harvest maturity; potatoes at BBCH 45–49, leeks at BBCH 48–49, cucumbers at BBCH 71–79, onions at BBCH 47–49, French beans at BBCH 73–79, peas seeds at BBCH 89 and rape seeds at BBCH 89.

Samples were analysed for parent compound and its metabolites DFA, DFEAF and 6-CNA using Method 01304. Results are summarized below.

Table 50 Additional European field rotational crop studies

Trial No. Crop, Variety, Location, Year	Application rate (g ai/ha)	No. applications, (Interval, days)	Spray volume (L/ha)	PBI (days)	DALA	Residues (mg/kg) expressed as parent				CNA (mg/kg)
						Parent	DFA	DFEAF	Parent + DFA + CNA	
11-2550-01 Potato, Resy, France, 2011	125	2 (10)	300	31	122	< 0.01	0.19	< 0.01	0.20	< 0.005
11-2550-02 Potato,	125	2 (10)	400	33	98	< 0.01	0.028	< 0.01	0.038	< 0.005

Trial No. Crop, Variety, Location, Year	Application rate (g ai/ha)	No. applications, (Interval, days)	Spray volume (L/ha)	PBI (days)	DALA	Residues (mg/kg) expressed as parent				CNA (mg/kg)
						Parent	DFA	DFEAF	Parent + DFA + CNA	
Riviera, Netherlands, 2011										
11-2550-03 Potato, Red Pontiac, Spain, 2011	125, 136	2 (9)	300, 327	25	126	< 0.01	0.25	< 0.01	0.26	< 0.005
11-2550-04 Potato, Arinda, Italy, 2011	125	2 (10)	300	30	137	< 0.01	0.036	< 0.01	0.046	< 0.005
11-2551-01 Leek, Bleu de Solaize, France, 2011	125	2 (10)	300	26	103	< 0.01	0.23	< 0.01	0.24	< 0.005
11-2551-02 Leek, Prelin, Germany, 2011	125	2 (10)	300	33	130	< 0.01	0.095	< 0.01	0.105	< 0.005
11-2551-03 Leek, Atal Temprano, Spain, 2011	125	2 (10)	300	28	97	< 0.01	0.020	< 0.01	0.03	< 0.005
11-2551-04 Leek, Libertas, Italy, 2011	125	2 (13)	400	31	112	< 0.01	0.039	< 0.01	0.049	< 0.005
11-2552-01 Cucumber, Raider, France, 2011	125	2 (10)	300	29	79	< 0.01	0.28	< 0.01	0.29	< 0.005
11-2552-01 Cucumber, Melody, Germany, 2011	125	2 (10)	300	25	73	< 0.01	0.32	< 0.01	0.33	< 0.005
11-2552-03 Cucumber, Dahser Hibrido ciclo corto, Spain, 2011	125	2 (10)	300	28	83	< 0.01	0.047	< 0.01	0.057	< 0.005
11-2552-04 Cucumber, Marketmore , Italy, 2011	125	2 (10)	400	28	69	< 0.01	0.41	< 0.01	0.42	< 0.005
11-2553-01 Onion, Paile des vertus, France, 2011	125	2 (10)	300	30	175	< 0.01	0.16	< 0.01	0.17	< 0.005
11-2553-02 Onion, Sherpa F1,	125	2 (10)	300	33	133	< 0.01	0.071	< 0.01	0.081	< 0.005

Trial No. Crop, Variety, Location, Year	Application rate (g ai/ha)	No. applications, (Interval, days)	Spray volume (L/ha)	PBI (days)	DALA	Residues (mg/kg) expressed as parent				CNA (mg/kg)
						Parent	DFA	DFEAF	Parent + DFA + CNA	
Germany, 2011										
11-2553-03 Onion, Dorata di Parma, Italy, 2011	125	2 (11)	300	25	116	< 0.01	0.058	< 0.01	0.068	< 0.005
11-2553-04 Onion, Figueras, Spain, 2011	132, 125	2 (10)	317, 300	28	118	< 0.01	< 0.02	< 0.01	< 0.03	< 0.005
11-2555-01 French bean, Banga, France, 2011	125	2 (10)	300	29	93	< 0.01	1.1	< 0.01	1.1	0.014
11-2555-02 French bean, Saxa, Germany, 2011	125	2 (10)	300	25	105	< 0.01	0.57	< 0.01	0.58	< 0.005
11-2555-03 French bean, OR Amo, Italy, 2011	125	2 (10)	400	30	85	< 0.01	0.38	< 0.01	0.39	< 0.005
11-2555-04 French bean, Cleo Temprana, Spain, 2011	125	2 (10)	300	28	90	< 0.01	0.26	< 0.01	0.30	0.016
11-2556-01 Pea, field, Montana, France, 2011	125	2 (11)	300	25	136	< 0.01	2.3	< 0.01	2.3	0.007
11-2556-02 Pea, field, Mascara, Germany, 2011	125	2 (10)	300	28	144	< 0.01	0.99 (*c0.11*)	< 0.01	1.0	0.01
11-2556-03 Pea, field, Lambado, Italy, 2011	125	2 (12)	400	30	100	< 0.01	0.65 (*c0.031*)	< 0.01	0.68	0.008
11-2556-04 Pea, field, Lincoln, Spain, 2011	125	2 (10)	300	35	132	< 0.01	2.1	< 0.01	2.1	0.017
11-2554-01 Winter rape, Dynastie hybrid 00, France, 2011	125	2 (10)	300	41	369	< 0.01	0.090	< 0.01	0.10	< 0.005
11-2554-02 Winter rape, ES-Astrid, Germany, 2011	125	2 (10)	300	34	361	< 0.01	0.15	< 0.01	0.16	< 0.005
11-2554-03	125	2 (10)	400	27	307	< 0.01	0.05	< 0.01	0.06	< 0.005

Trial No. Crop, Variety, Location, Year	Application rate (g ai/ha)	No. applications, (Interval, days)	Spray volume (L/ha)	PBI (days)	DALA	Residues (mg/kg) expressed as parent				CNA (mg/kg)
						Parent	DFA	DFEAF	Parent + DFA + CNA	
Winter rape, Primus Winter variety, Italy, 2011										
11-2554-04 Winter rape, Pacific winter, Spain, 2011	125	2 (9)	300	39	290	< 0.01	0.072	< 0.01	0.082	< 0.005

In the European single crop, single rotation crop studies residues of parent were all < 0.01 mg/kg (potatoes n = 4, cucumber n = 4, leek n = 4, French bean n = 4, onion n = 4, pea n = 4 and rape seed n = 4).

Difluoroethyl-amino-furanone (DFEAF) was non-quantifiable in all commodities while DFA was quantifiable in most commodities.

Residues of 6-CNA were < 0.005 mg/kg (measured as 6-CNA) in all samples except bean pods which had residues of < 0.005 (2), 0.014 and 0.016 mg/kg and pea, dry which had residues of 0.007, 0.008, 0.01 and 0.017 mg/kg.

A summary of residues of parent in the field rotational studies is tabulated below.

Table 51 Flupyradifurone parent residues in the field rotational residues studies

Rotational Crop	No. of trials	Application Rate (g ai/ha)	Maximum flupyradifurone residues (mg/kg) after application to bare soil or lettuce (primary crop)		
			25–41 day PBI (bare soil)	61–145 day PBI (primary crop)	266–320 day PBI (primary crop)
Barley forage	4	200	0.02	0.01	0.03
Barley grain	4	200	< 0.01	< 0.01	< 0.01
Barley straw	4	200	0.04	0.02	< 0.01
Carrot and turnip roots	4	200	< 0.01	< 0.01	< 0.01
Lettuce	4	200	0.08	0.03	0.02
Potato	4	2 × 125	< 0.01		
Cucumber	4	2 × 125	< 0.01		
Leek	4	2 × 125	< 0.01		
French bean	4	2 × 125	< 0.01		
Onion	4	2 × 125	< 0.01		
Pea, field (dry)	4	2 × 125	< 0.01		
Winter rape (seed)	4	2 × 125	< 0.01		

PBI = plant back interval

A summary of total residues (flupyradifurone + DFA + CNA) in the field rotational studies is tabulated below.

Table 52 Flupyradifurone residues (flupyradifurone + DFA + CNA) in the field rotational residues studies

Rotational Crop	No. of trials	Application Rate (g ai/ha)	Maximum, mean and median flupyradifurone + DFA + CNA residues (mg/kg) after application to bare soil or lettuce (primary crop) [converted to residues at seasonal maximum application rate of 409 g ai/ha]		
			25–41 Day PBI (bare soil)	61–145 Day PBI (primary crop)	266–320 Day PBI (primary crop)

			Maximum	Mean	Median	Maximum	Mean	Median	Maximum	Mean	Median
Barley forage	4	200	0.39 [0.80]	0.15 [0.31]	0.085 [0.17]	0.11 [0.22]	0.058 [0.12]	0.04 [0.08]	0.18 [0.37]	0.068 [0.14]	0.03 [0.06]
Barley grain	4	200	0.64 [1.31]	0.36 [0.74]	0.34 [0.70]	0.27 [0.55]	0.13 [0.27]	0.105 [0.21]	0.38 [0.78]	0.17 [0.34]	0.105 [0.21]
Barley straw	4	200	0.38 [0.78]	0.16 [0.33]	0.105 [0.21]	0.12 [0.25]	0.075 [0.15]	< 0.06 [< 0.12]	0.18 [0.37]	0.09 [0.18]	< 0.06 [< 0.12]
Carrot and turnip roots	4	200	0.13 [0.27]	0.069 [0.14]	0.06 [0.12]	0.06 [0.12]	0.04 [0.08]	0.04 [0.08]	0.05 [0.10]	0.039 [0.080]	0.04 [0.08]
Lettuce	4	200	0.20 [0.41]	0.081 [0.17]	0.06 [0.12]	0.11 [0.22]	0.049 [0.10]	< 0.03 [< 0.06]	0.10 [0.20]	0.043 [0.088]	< 0.03 [< 0.06]
Potato	4	2 × 125	0.26 [0.43]	0.14 [0.23]	0.12 [0.20]						
Cucumber	4	2 × 125	0.42 [0.69]	0.27 [0.44]	0.31 [0.51]						
Leek	4	2 × 125	0.24 [0.39]	0.11 [0.18]	0.077 [0.13]						
French bean	4	2 × 125	1.1 [1.80]	0.59 [0.98]	0.49 [0.80]						
Onion	4	2 × 125	0.17 [0.28]	0.087 [0.14]	0.075 [0.12]						
Pea, field (dry)	4	2 × 125	2.3 [3.77]	1.52 [2.49]	1.55 [2.56]						
Winter rape (seed)	4	2 × 125	0.16 [0.26]	0.10 [0.16]	0.091 [0.15]						

PBI = plant back interval

Overall highest, mean and median residue for barley forage are 0.39 (0.80 at 409 g ai/ha), 0.092 (0.19) and 0.04 (0.082) mg/kg respectively

Overall highest, mean and median residue for barley grain are 0.64 (1.3 at 409 g ai/ha), 0.21 (0.43) and 0.11 (0.22) mg/kg respectively

Overall highest, mean and median residue for barley straw are 0.38 (0.78 at 409 g ai/ha), 0.11 (0.22) and < 0.06 (< 0.12) mg/kg respectively

Overall highest, mean and median residue for carrot and turnip roots are 0.13 (0.27 at 409 g ai/ha), 0.049 (0.10) and 0.04 (0.08) mg/kg respectively

Overall highest, mean and median residue for lettuce are 0.20 (0.41 at 409 g ai/ha), 0.058 (0.12) and < 0.03 (< 0.06) mg/kg respectively

The field rotational crop studies suggest that residues of flupyradifurone may occur in rotational crops.

RESIDUE ANALYSIS

Analytical methods

Details of analytical methods including validation data were supplied for the determination of flupyradifurone and key metabolites in plant and animal matrices, soil and water and are considered satisfactory.

Table 53 A summary of all analytical methods for plants and animals

Matrix	Analyte	Method No.	Detection system	LOQ	Reference
Plant	Flupyradifurone DFA DFEAF 6-CNA (data collection)	01304 (RV-001-P10-02 Amendment	HPLC-MS/MS	Dried beans, wheat forage, orange fruit, soya bean seeds, tomato fruit and wheat grain LOQ = 0.01 mg/kg for parent, DFEAF and 6-CNA in all matrices	Li 2011 with amendment by Schöning 2012, RARVP013

Matrix	Analyte	Method No.	Detection system	LOQ	Reference
		No. 1)		LOQ = 0.02 mg/kg for DFA in orange fruit and tomato fruit and 0.05 mg/kg in all other matrices	Justus 2011, MEF-11/793 (Extraction efficiency testing)
Plant	Flupyradifurone DFA DFEAF 6-CNA (data collection)	01212	HPLC-MS/MS	Tomato fruit, grapes, kidney bean dry seed, barley grain and summer rape seed LOQ = 0.01 mg/kg for parent, furanone and 6-CNA in all matrices LOQ = 0.02 mg/kg for DFA	Rosati 2012, MR-10/174
Plant	Flupyradifurone DFA (enforcement)	01330	HPLC-MS/MS	Lettuce head, rape seed, orange fruit and wheat grain LOQ = 0.01 mg/kg for parent and 0.02 mg/kg for DFA Hop cone LOQ = 0.05 mg/kg for parent and 0.10 mg/kg for DFA	Schulte and Bauer 2012a, MR-011/096 Konrad 2012a, 2011/0134/01 (ILV of Method 01330)
Plant	Flupyradifurone DFA (enforcement)	01330 Modification M001	HPLC-MS/MS	Rape seed LOQ = 0.01 mg/kg for parent and 0.02 mg/kg for DFA	Schulte and Teubner 2012a, MR-12/054 Konrad 2012c, 2012/0082/01 (ILV of Method 01330/M001)
Animal	Flupyradifurone DFA BYI 02960-acetyl-AMCP BYI 02960-OH (data collection)	RARVP040 (RV-004-A11-04)	HPLC-MS/MS	All poultry and bovine matrices LOQ = 0.01 mg/kg for parent, BYI 02960-acetyl-AMCP and BYI 02960-OH LOQ = 0.01 mg/kg for DFA in poultry matrices and 0.02 mg/kg in bovine matrices (except whey = 0.05 mg/kg)	Wade and Netzband 2012, RARVP041, Moore and Harbin 2012, RARVP050
Animal	Flupyradifurone DFA (enforcement)	01214	HPLC-MS/MS	Muscle, liver, kidney, egg, fat and milk LOQ = 0.01 mg/kg for parent and 0.02 mg/kg for DFA	Schulte and Bauer 2012b, MR-011/144 Konrad 2012b, 2011/0164/01 (ILV of Method 01214)

Plant commodities

Method RV-001-P10-02

Method RV-001-P10-02 also called 01304 (data collection): Method 01304 for the determination of flupyradifurone in plant matrices by means of HPLC with tandem mass spectrometry (HPLC-MS/MS) was reported by Li in 2011 and with amendment by Schöning in 2012 (RARVP013). This was the data generation method used in the field trials.

Flupyradifurone and its metabolites DFA, DFEAF and 6-CNA are extracted twice from plant material with acetonitrile/water (4/1, v/v) with 2.2 mL/L formic acid. After rinsing and diluting with the same extraction solvent mixture, aliquots of the extracts are purified through a C-$_{18}$ solid-phase extraction column, then amended with a mixture of stable, isotopically labelled internal standards. The final solution is analysed by HPLC-MS/MS. Two MRM transitions for quantitation and confirmation are monitored for BYI 02960 (*m/z* 289/126 or 90) and DFEAF (*m/z* 162/94 or 98). An HPLC-MS/MS method is highly specific, but the confirmatory ions were tested, and due to repeatability issues with BYI 02960 at the LOQ in some matrices, a second column system (Gemini C_{18}, instead of HILIC as used in the primary method) was employed for confirmatory purposes with that compound. This column is also used as a confirmatory measurement of 6-CNA. For DFA, no second MRM transition is available. A Restek Allure Organic Acids HPLC column is therefore employed as a different separation system (as opposed to a HILIC column for the primary determination).

The accuracy of the method was assessed based on the determined recovery rates. The materials tested included bean (dry seed), wheat (grain & forage), orange (fruit), tomato (fruit), and soya bean (seed). Samples were fortified with BYI 02960 and 6-CNA at concentrations of 0.01 and 1.0 mg/kg, DFEAF at concentrations of 0.01, 0.013 and 1.0 or 1.3 mg/kg, and DFA at 0.05 and 1.0 mg/kg (for orange and tomato fruit samples, at 0.02 mg/kg as well). Some other spiking levels were employed. Metabolite levels were expressed in parent equivalents. Mean recoveries per fortification level for BYI 02960, DFEAF, DFA and 6-CNA for all matrices were in a range of 75–110%, using the primary conditions.

Confirmatory procedures for BYI 02960 and DFEAF called for the use of the same chromatographic system, but using a second MRM transition. For these two compounds, mean recoveries ranged from 81–107% for all matrices. However, as the repeatability was not within specification (relative standard deviation [RSDs] > 20%) for BYI 02960 in two matrices (orange fruit and wheat grain, at the LOQ), a second confirmatory method was developed, utilising a different column system (Gemini C_{18}, instead of HILIC as used in the primary method). Using this system, using both MRM transitions, mean recovery rates ranged from 85–104% in all matrices, always with acceptable RSD values.

For DFA, the confirmatory procedure based on chromatography *via* an alternative column (Restek Allure Organic Acids, instead of HILIC as used in the primary method) gave mean recovery values ranging from 77–105%, with a single exception. For tomato fruit, the mean recovery at the 50×LOQ spiking level (1.0 mg/kg) was 111%. This value was acceptable, since it was very close to 110%, the RSD was very low (1.0%) and the overall recovery over both fortification levels was in the acceptable range. For 6-CNA the confirmatory procedure using a Gemini column gave mean recovery values ranging from 82–106%.

The LOQ for flupyradifurone, DFEAF and 6-CNA, defined as the lowest validated fortification level, was 0.01 mg/kg in all matrices tested. For DFA, the LOQ was 0.02 mg/kg in orange fruit and tomato fruit and 0.05 mg/kg in all other matrices tested. All metabolite levels are expressed in parent equivalents.

Method linearity was validated over the range 0.25 to 312.5 ng/L for all four analytes (flupyradifurone, DFEAF, DFA and 6-CNA). The coefficients of determination of the $1/x^2$ weighted linearity curves were all > 0.99.

Table 54 Recoveries for method 01304: flupyradifurone in plants

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Method: Hilic Column Transition 289 → 126			Hilic Column, Confirmatory Transition 289 → 90		
				Range [%]	Mean ± SD [%]	RSD [%]	Range [%]	Mean ± SD [%]	RSD [%]
				HPLC-MS/MS			HPLC-MS/MS		
Bean, dried bean	Flupyradifurone	6	0.01	85–108	93 ± 8.1	8.6	80–109	95 ± 10.9	11.7
		5	1.0	90–130	109 ± 14.6	13.3	102–137	113 ± 14.2	12.6
Wheat, forage	Flupyradifurone	5	0.01	86–104	97 ± 7.5	7.7	80–108	90 ± 11.6	12.9
		5	1.0	86–99	95 ± 5.5	6.0	96–108	99 ± 4.9	5.1
Orange, fruit	Flupyradifurone	5	0.01	88–109	99 ± 9.9	9.9	47–137	87 ± 32.2	37.3
		5	1.0	87–109	100 ± 8.4	8.5	83–103	94 ± 7.6	8.3
Soya bean, seed	Flupyradifurone	5	0.01	75–105	87 ± 11.3	13.1	87–120	107 ± 13.9	12.9
		5	1.0	74–103	94 ± 11.9	12.5	90–111	101 ± 8.1	8.2
Tomato, fruit	Flupyradifurone	5	0.01	82–105	97 ± 9.8	9.9	73–119	93 ± 18.2	19.4
		5	0.1	86–108	97 ± 9.8	10.0	95–120	104 ± 11.4	11.1
		5	1.0	81–96	88 ± 5.8	6.5	96–111	105 ± 5.6	5.4
Wheat, grain	Flupyradifurone	5	0.01	74–100	87 ± 9.4	10.7	65–157	102 ± 35.6	34.9
		5	1.0	73–98	88 ± 10.0	11.6	76–119	94 ± 16.7	17.7

Table 55 Recoveries for method 01304: flupyradifurone in plants

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	2nd Confirmatory Method: Gemini Column			2nd Confirmatory Method: Gemini Column		
				Primary Transition 289 → 126			Secondary Transition 289 → 90		
				Range [%]	Mean ± SD [%]	RSD [%]	Range [%]	Mean ± SD [%]	RSD [%]
				HPLC-MS/MS			HPLC-MS/MS		
Bean, dried bean	Flupyradifurone	11	0.01	90–106	96 ± 5.0	5.3	88–108	96 ± 6.6	6.8
		5	1.0	92–114	103 ± 9.7	9.6	93–115	104 ± 10.4	10.0
Wheat, forage	Flupyradifurone	5	0.01	87–95	93 ± 3.2	3.5	88–100	94 ± 4.9	5.3
		5	1.0	81–95	87 ± 5.3	5.9	80–93	85 ± 4.9	5.7
Orange, fruit	Flupyradifurone	5	0.01	91–103	96 ± 4.9	5.3	87–103	96 ± 6.7	6.2
		5	1.0	91–98	96 ± 2.6	2.8	92–98	95 ± 2.4	2.4
Soya bean, seed	Flupyradifurone	5	0.01	98–110	102 ± 5.2	5.1	90–98	93 ± 3.4	3.6
		5	1.0	90–96	93 ± 2.8	3.3	90–94	92 ± 1.8	1.8
Tomato, fruit	Flupyradifurone	5	0.01	79–114	95 ± 13.0	13.7	77–117	93 ± 15.7	16.9
		5	0.10	87–104	95 ± 6.3	6.4	86–97	92 ± 4.5	4.8
Wheat, grain	Flupyradifurone	5	0.01	86–101	92 ± 5.9	6.6	82–105	96 ± 9.6	10.1
		5	1.0	83–99	93 ± 5.8	6.5	84–98	93 ± 5.6	5.8

Table 56 Recoveries for method 01304: DFA in plants

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Method: Hilic Column			Restek Allure Organic Acids Column, Confirmatory		
				Transition 95 → 51			Transition 95 → 51		
				Range [%]	Mean ± SD [%]	RSD [%]	Range [%]	Mean ± SD [%]	RSD [%]
				HPLC-MS/MS			HPLC-MS/MS		
Bean, dried bean	DFA	6/5	0.05	73–80	75 ± 2.9	3.9	74–80	77 ± 3.1	3.9
		5	1.0	88–184	97 ± 13.6	12.6	75–84	79 ± 3.7	4.3
Wheat, forage	DFA	5	0.05	85–92	89 ± 3.2	3.5	98–107	102 ± 3.2	3.3
		5	1.0	96–103	99 ± 2.6	2.8	101–106	105 ± 2.0	2.0
Orange, fruit	DFA	5	0.02	96–111	104 ± 6.4	5.8	92–119	101 ± 10.9	10.7
		5/–	0.05	87–100	93 ± 5.2	5.6	–	–	–
		5	1.0	99–101	100 ± 1.0	0.8	100–105	101 ± 1.9	2.0
Soya bean, seed	DFA	5	0.05	71–79	76 ± 2.8	3.9	78–95	84 ± 6.5	7.4
		5	1.0	73–78	76 ± 2.1	2.5	80–88	84 ± 3.6	4.5
Tomato, fruit	DFA	5	0.02	97–108	102 ± 4.9	5.0	96–118	103 ± 8.6	8.5
		5/–	0.05	81–103	90 ± 8.2	9.1	–	–	–
		5/–	0.1	90–103	97 ± 4.8	5.0	–	–	–
		5	1.0	91–99	95 ± 3.0	3.2	110–113	111 ± 0.9	1.0
Wheat, grain	DFA	5	0.05	86–96	89 ± 4.4	4.7	78–96	87 ± 6.7	7.9
		5	1.0	85–94	90 ± 3.2	3.7	85–97	91 ± 4.9	5.4

Table 57 Recoveries for method 01304: DFEAF in plants

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary			Confirmatory		
				Transition 162 → 94			Transition 162 → 98		
				Range [%]	Mean ± SD [%]	RSD [%]	Range [%]	Mean ± SD [%]	RSD [%]
				HPLC-MS/MS			HPLC-MS/MS		
Bean, dried bean	DFEAF	5	0.01	79–88	84 ± 3.9	4.4	74–100	81 ± 10.5	13.4
		6	0.013	87–110	96 ± 8.8	9.1	88–119	101 ± 11.1	11.1
		5	1.0	90–135	109 ± 18.1	16.5	92–109	101 ± 7.3	7.1
Wheat, forage	DFEAF	5	0.01	83–120	95 ± 14.6	15.5	90–116	100 ± 10.6	10.3
		5	0.013	91–103	96 ± 4.7	5.1	82–95	88 ± 5.1	5.8
		5	1.0	82–100	90 ± 7.8	8.3	89–110	94 ± 8.7	9.2
Orange, fruit	DFEAF	5	0.01	96–125	110 ± 12.5	11.4	88–106	99 ± 7.2	7.3
		5	0.013	82–99	93 ± 7.3	7.7	83–94	90 ± 4.4	4.6

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Transition 162 → 94			Confirmatory Transition 162 → 98		
				Range [%]	Mean ± SD [%]	RSD [%]	Range [%]	Mean ± SD [%]	RSD [%]
		5	1.3	93–106	100 ± 6.0	6.0	91–99	96 ± 3.1	3.4
Soya bean, seed	DFEAF	5	0.01	89–103	94 ± 5.9	6.2	75–89	81 ± 6.7	8.3
		5	0.013	81–112	99 ± 12.5	12.5	89–97	94 ± 3.5	3.4
		5	1.3	91–103	96 ± 4.8	4.8	86–93	89 ± 3.0	3.2
Tomato, fruit	DFEAF	5	0.01	94–136	106 ± 17.4	16.2	74–114	96 ± 14.9	15.3
		5	0.013	78–105	90 ± 10.1	11.2	78–106	95 ± 10.5	11.3
		5	0.13	89–107	97 ± 7.3	7.6	92–108	102 ± 6.0	5.9
		5	1.3	94–97	96 ± 1.2	1.2	89–95	92 ± 2.0	2.6
Wheat, grain	DFEAF	5	0.01	79–116	99 ± 13.4	13.8	84–101	92 ± 7.1	7.5
		5	0.013	94–118	105 ± 10.4	10.1	80–116	93 ± 16.8	17.9
		5	1.3	91–109	98 ± 7.8	8.0	90–114	102 ± 10.3	10.0

Table 58 Method recoveries for method 01304: 6-CNA in plants

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Method (Hilic Column)			Secondary Method (Gemini Column)		
				Range [%]	Mean ± SD [%]	RSD [%]	Range [%]	Mean ± SD [%]	RSD [%]
				HPLC-MS/MS			HPLC-MS/MS		
Bean, dried bean	6-CNA	6/11	0.01	74–102	91 ± 9.2	10.3	81–110	97 ± 10.2	10.6
		5	1.0	88–108	97 ± 8.8	9.0	77–100	88 ± 10.0	11.3
Wheat, forage	6-CNA	5	0.01	83–96	90 ± 4.6	5.2	82–108	96 ± 9.7	10.2
		5	1.0	90–101	94 ± 4.9	5.0	82–92	88 ± 3.7	4.4
Orange, fruit	6-CNA	5	0.01	93–115	107 ± 9.3	8.7	95–117	103 ± 9.9	9.2
		5	1.0	94–111	100 ± 6.8	6.8	91–97	94 ± 3.0	3.1
Soya bean, seed	6-CNA	5	0.01	80–111	98 ± 13.9	14.4	98–119	106 ± 8.4	7.9
		5	1.0	80–90	84 ± 3.8	4.7	79–86	82 ± 2.9	3.5
Tomato, fruit	6-CNA	5	0.01	77–107	90 ± 11.3	12.9	76–90	85 ± 5.6	6.6
		5	0.1	85–129	101 ± 17.4	17.2	85–98	94 ± 5.0	5.5
		5/–	1.0	89–104	95 ± 5.9	5.9	–	–	
Wheat, grain	6-CNA	5	0.01	77–106	94 ± 11.2	12.2	80–88	85 ± 3.0	3.5
		5	1.0	84–104	95 ± 7.4	7.9	85–94	90 ± 4.2	4.7

The stability of residues of BYI 02960, 6-CNA, DFA and DFEAF in the initial extract (before clean-up) and the final extract was checked after approximately 20 days for the tested sample materials at the 1.0 mg/kg level and was found to be acceptable. The stability of the calibration standards, spiking solutions and internal standard solutions was evaluated after storage in the refrigerator for 185 days. The stored solutions were compared with freshly prepared solutions. All the solutions were stable for at least 185 days when stored refrigerated at 4 °C ± 3 °C.

The extraction efficiency of the residue method 01304 for the determination of the relevant residues of BYI 02960 in plant matrices, consisting of the parent compound and its metabolites DFEAF and DFA, was assured by choosing the same extraction procedures as used in the plant metabolism studies. An extraction efficiency study was conducted (Justus 2011g, MEF-11/793). Aged radioactive residues in samples from plant metabolism and confined rotational crop studies were analysed using method 01304 (Li & Schöning 2012, RARVP013), and the recoveries of the extracted residues were then compared to those in the respective metabolism studies. The sample materials were tomato fruit, cotton seed, potato tuber and wheat straw. The contribution of the metabolite DFA to the extraction efficiency was determined based on tomato fruit samples originating from the metabolism study with the [ethyl-1-^{14}C]-label, the only plant metabolism study conducted with this label, which metabolizes to ^{14}C-DFA.

Relative amounts of the analytes relevant to a residue definition, BYI 02960, DFA and DFEAF, were then compared between the original metabolism study extracts and those made using method 01304. The determined extraction efficiency values in tomato, cotton, potato, and wheat matrices were 100.7%, 160.2%, 104.0%, and 88.4%, respectively. The high value of 160% for cotton seeds was postulated to be due to losses of radioactivity observed during extraction and purification steps in the cotton metabolism study, but not observed in the extraction efficiency study.

These results demonstrate that residue method 01304 is suitable for the extraction and quantification of the total residues of BYI 02960 for risk assessment, consisting of parent compound and DFA from plant matrices. Satisfactory extraction was also proven for metabolite DFEAF, which was a potential candidate for the residue definition for risk assessment. This conclusion applies for other methods based on the same extraction procedures as well.

A summary of the extraction efficiency results is given in Table 59.

Table 59 Recovery results of extraction efficiency testing of method 01304—Recoveries from representative matrices taken from the plant metabolism studies

Crop and matrix	Values determined in/with						
	Metabolism studies			Method 01304			
	TRR [a] [mg/kg]	Components of TTR [b] [mg/kg]	TTR/TRR [%]	TRR [a] [mg/kg]	Components of TTR [b] [mg/kg]	TTR/TRR [%]	Extraction efficiency [c] [%]
tomato fruit [d]	0.201	0.198	98.8	0.191	0.190	99.5	100.7
cotton seed [e]	0.068	0.016	23.4	0.068	0.026	37.5	160.2
potato tuber [f]	0.171	0.102	59.8	0.191	0.119	62.2	104.0
wheat grain [g]	6.290	2.833	45.1	6.139	2.448	39.9	88.4

[a] TRR = total radioactive residues

[b] TTR = total toxic residue, i.e. the total residue of BYI 02960, comprising parent compound, DFA, and DFEAF

[c] Extraction efficiency = ratio of (TTR extracted with method 01304) ÷ (TTR extracted in metabolism study) as%

[d] Metabolism study reported under no. MEF-11/498

[e] Metabolism study reported under no. MEF-11/393

[f] Metabolism study reported under no. MEF-10/769

[g] Metabolism study reported under no. MEF-11/365

Similarly, the extraction efficiency of the residue method 01304 for the determination of the 6-CNA in plant matrices, was also investigated in the same study (Justus 2011, MEF-11/793).

For tomato fruits, cotton seeds and wheat straw, the TRR values obtained in the current extraction efficiency study were in good agreement with those obtained in the metabolism and confined rotational crops studies. For potato tubers, a significantly lower TRR value was obtained (0.49 mg/kg vs 0.115 mg/kg). This was thought to be due to inhomogeneous distribution of the radioactivity in the pieces of the cut potato tubers.

Table 60 Recovery results of extraction efficiency testing of method 01304—Recoveries of 6-CNA from representative matrices taken from the plant metabolism studies

Crop and matrix	Values determined in/with						
	Metabolism studies			Method 01304			
	TRR [a] [mg/kg]	6-CNA [mg/kg]	6-CNA/TRR [%]	TRR [a] [mg/kg]	6-CNA [mg/kg]	6-CNA/TRR [%]	Extraction efficiency [b] [%]
tomato fruit [c]	0.130	0.017	13.2	0.141	0.018	12.5	95.0
cotton seed [d]	0.068	0.003	5.0	0.068	0.011	16.4	330.5
potato tuber [e]	0.115	0.021	18.4	0.049	0.013	26.9	146.0
wheat grain [f]	9.015	0.458	5.1	9.396	0.395	4.2	82.4

[a] TRR = total radioactive residues

[b] Extraction efficiency = ratio of (6-CNA extracted with method 01304) ÷ (6-CNA extracted in metabolism study) as %

[c] Metabolism study reported under no. MEF-11/498

[d] Metabolism study reported under no. MEF-11/393

[e] Metabolism study reported under no. MEF-10/769

[f] Metabolism study reported under no. MEF-11/365

The determined extraction efficiency values in tomato, cotton, potato and wheat matrices for the metabolite 6-CNA, were 95.0%, 330.5%, 146.0%, and 82.4%, respectively. Again the high value of 330% for cotton seeds was postulated to be due to losses of radioactivity observed during extraction and purification steps in the cotton metabolism study, but not observed in the extraction efficiency study. It was also thought that another possible reason was the addition of formic acid into solvent mixture of the residue method, which supported the extraction of the acidic metabolite 6-CNA by conversion into the non-ionic form. This was also suggested as the reason for high extraction efficiency for potato tubers.

Until extraction, the samples in these studies were stored over a period of 15–31 months. Comparison of the profiles recorded in the extraction efficiency study with the profiles from the metabolism and confined rotational crop studies showed no significant change of the residues. It was concluded that results were not influenced by degradation of the representative components through the period of storage after the original metabolism studies.

Method 01212

Method 01212 (data collection): A validated data collection method (01212) has been reported for the determination of flupyradifurone and its metabolites DFA, DFEAF and 6-CNA in/on plant materials using HPLC-MS/MS (Rosati 2012, MR-10/174).

The residues of flupyradifurone and its metabolites are extracted twice from plant material with acetonitrile/water (4/1, v/v) with 2.2 mL/L formic acid. Aliquots of the extracts are purified over a cationic resin (AG-50W-X8), then amended with a mixture of stable, isotopically labelled internal standards. The final solution is analysed by HPLC-MS/MS using either a Hilic (for BYI 02960, DFEAF or 6-CNA) or Hypercarb (DFA) column. As this is a data collection method, only one MRM transition is monitored for BYI 02960 (*m/z* 289/126), DFEAF (*m/z* 162/94), DFA (*m/z* 95/51) and 6-CNA (*m/z* 156/112) in each matrix tested. The HPLC-MS/MS method is highly specific and fully satisfactory for data collection.

The accuracy of the method was assessed based on the determined recovery rates in different matrices. Samples of bean (dry seed), barley (grain), grape (bunch), tomato (fruit), and rape (seed) were fortified with BYI 02960, DFEAF and 6-CNA at concentrations of 0.01 and 0.10 mg/kg while DFA was spiked at 0.02 and 0.20 mg/kg. (Metabolites were expressed in parent equivalents.) Mean recoveries per fortification level for BYI 02960, DFEAF, and DFA for all matrices were in a range of 70–110%, with three exceptions. In two cases, mean DFEAF recoveries were outside of this range (tomato fruit, LOQ, 111%; and barley grain, LOQ, 114%), and in another, mean DFA values were higher (tomato fruit, LOQ, 111%). These values were nevertheless considered to be acceptable, based on the fact that they were very close to 110% and, in all cases, the RSD was satisfactory (8.3–13.1%). For 6-CNA the mean of recoveries were within 70–110% except for tomato fruit (LOQ, 119%) explained by the mean value of estimated apparent residue in control samples (20% of the LOQ) and the fact that no correction was made to recoveries and summer rape seed (LOQ, 117%) which had a satisfactory RSD of 13.5%.

The correlation between the injected amount of substance and the detector response was linear in the range from 0.05 to 50 µg/L for BYI 02960, DFEAF and 6-CNA and from 0.15 to 0.50 µg/L for DFA. The correlation coefficients (r) were > 0.99 in all cases. The LOQ for BYI 02960, DFEAF and 6-CNA was 0.01 mg/kg in all matrices tested. (All metabolite values are expressed in parent equivalents.) For DFA, the LOQ was 0.02 mg/kg in all matrices. For BYI 02960, DFEAF, DFA and 6-CNA, RSD values were 0.7–14.9% across all matrices.

Table 61 Recoveries for method 01212: flupyradifurone in plants

Matrix	Analyte [a]	No. of tests	Spiking level [mg/kg]	Transition 289 → 126		
				Range [%]	Mean	RSD [%]

				HPLC-MS/MS		
Tomato, fruit	Flupyradifurone	5	0.01	94–125	104	12.0
		5	0.10	99–118	109	7.3
Grape, bunch of grapes	Flupyradifurone	5	0.01	97–107	101	4.8
		5	0.10	100–113	106	5.3
Kidney bean, dry seed	Flupyradifurone	5	0.01	96–117	105	8.2
		5	0.10	95–108	110	8.1
Barley, grain	Flupyradifurone	5	0.01	85–121	101	13.9
		5	0.10	95–104	98	3.8
Summer rape, seed	Flupyradifurone	5	0.01	88–112	103	8.9
		5	0.10	97–107	101	4.6

[a] Expressed as BYI 02960

Table 62 Recoveries for method 01212: DFA in plants

Matrix	Analyte [a]	No. of tests	Spiking level [mg/kg]	Transition 95 → 51		
				Range [%]	Mean	RSD [%]
				HPLC-MS/MS		
Tomato, fruit	DFA	5	0.02	93–127	111	13.1
		5	0.20	103–109	106	2.3
Grape, bunch of grapes	DFA	5	0.02	96–104	101	3.4
		5	0.20	97–104	101	2.7
Kidney bean, dry seed	DFA	5	0.02	83–103	92	8.6
		5	0.20	94–96	95	0.7
Barley, grain	DFA	5	0.02	90–110	98	8.0
		5	0.20	76–90	84	6.7
Summer rape, seed	DFA	5	0.02	68–84	74	8.8
		5	0.20	68–71	70	1.9

[a] Expressed as BYI 02960

Table 63 Recoveries for method 01212: DFEAF in plants

Matrix	Analyte [a]	No. of tests	Spiking level [mg/kg]	Transition 162 → 94		
				Range [%]	Mean	RSD [%]
				HPLC-MS/MS		
Tomato, fruit	DFEAF	5	0.01	97–120	111	8.3
		5	0.10	90–106	100	6.0
Grape, bunch of grapes	DFEAF	5	0.01	97–113	101	6.7
		5	0.10	96–113	104	5.8
Kidney bean, dry seed	DFEAF	5	0.01	88–111	99	10.2
		5	0.10	94–122	110	9.4
Barley, grain	DFEAF	5	0.01	106–133	114	10.4
		5	0.10	87–104	95	6.5
Summer rape, seed	DFEAF	5	0.01	75–109	93	14.5
		5	0.10	99–112	106	4.9

[a] Expressed as BYI 02960

Table 64 Recoveries for method 01212: 6-CNA in plants

Matrix	Analyte [a]	No. of tests	Spiking level [mg/kg]	Transition 156 → 112		
				Range [%]	Mean	RSD [%]
				HPLC-MS/MS		
Tomato, fruit	6-CNA	5	0.01	113–139	119	9.5
		5	0.10	103–115	109	4.0

Matrix	Analyte [a]	No. of tests	Spiking level [mg/kg]	Transition 156 → 112		
				Range [%]	Mean	RSD [%]
				HPLC-MS/MS		
Grape, bunch of grapes	6-CNA	5	0.01	102–112	107	4.0
		5	0.10	98–109	104	4.7
Kidney bean, dry seed	6-CNA	5	0.01	74–104	93	14.0
		5	0.10	90–125	110	12.6
Barley, grain	6-CNA	5	0.01	92–130	107	14.9
		5	0.10	98–101	100	1.3
Summer rape, seed	6-CNA	5	0.01	102–137	117	13.5
		5	0.10	101–122	110	7.5

[a] Expressed as BYI 02960

Method 01330 and its modification M001

Method 01330 and its modification M001 (enforcement method): A residue analytical method, 01330, was developed as an enforcement method for the determination of the residues of BYI 02960 (parent compound) and its metabolite DFA in/on plant matrices using HPLC-MS/MS (Schulte and Bauer 2012a, MR-011/096).

Residues are extracted twice from plant material with acetonitrile/water (4/1, v/v) with 2.2 mL/L formic acid. After dilution, two aliquots of the raw extract were filtered for separate measurement of BYI 02960 and DFA. The solutions are analysed by HPLC-MS/MS using either a C_{18}-column (BYI 02960) or a ZIC®-HILIC SeQuantTM or a Hypercarb column (DFA); residues were quantified against matrix-matched standards. Two MRM transitions for quantitation and confirmation are monitored for BYI 02960 (*m/z* 289/126 or 90) in each matrix tested. For DFA, only one MRM transition (*m/z* 95/51) is available. Thus, a Hypercarb column is employed as a different separation system (as opposed to HILIC for the primary determination). The confirmatory methods were fully validated.

The accuracy of the method was assessed on the basis of the determined recovery rates in various matrices. Samples of lettuce (head), rape (seed), orange (fruit), wheat (grain) and hop (cone), were fortified with flupyradifurone at concentrations of 0.01 and 0.10 mg/kg (0.05 and 0.5 mg/kg in hops) while DFA was spiked at 0.02 and 0.20 mg/kg (0.10 and 1.0 mg/kg for hops), expressed in parent equivalents. Mean recoveries per fortification level for the primary method for both analytes and all matrices were in a range of 88–106%, with one exception at 62–65% (DFA, rape seed). Using the confirmatory conditions, mean values per fortification level were 70–108%, with one exception at 64% (DFA, rape seed, 0.20 mg/kg). To improve the low recoveries for rape seed, method 01330 was modified slightly (Method 01330/M001).

The correlation between the injected amount of substance and the detector response was linear for standards in matrix in the range from 0.125 to 500 µg/L. The correlation coefficients of were > 0.99 in both cases. Linearity was proven for the confirmatory method over the same concentration range with correlation coefficients of > 0.99.

The LOQ for BYI 02960, defined as the lowest validated fortification level, was 0.01 mg/kg in all matrices tested, except hop cones, in which it was 0.05 mg/kg. For DFA, the LOQ was 0.02 mg/kg in all matrices except hops, where it was 0.10 mg/kg.

Table 65 Recoveries for method 01330: flupyradifurone in plants

Matrix	Analyte [a]	No. of tests	Spiking level [mg/kg]	Quantification			Confirmatory		
				Transition 289 → 126			Transition 289 → 90		
				Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]
				HPLC-MS/MS			HPLC-MS/MS		
Lettuce,	Flupyradifurone	5	0.01	84–105	97	8.8	85–107	99	9.0

Matrix	Analyte [a]	No. of tests	Spiking level [mg/kg]	Quantification			Confirmatory		
				Transition 289 → 126			Transition 289 → 90		
				Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]
				HPLC-MS/MS			HPLC-MS/MS		
head		5	0.10	88–99	95	5.1	87–98	94	5.0
Rape, seed	Flupyradifurone	5	0.01	91–99	95	3.8	91–100	96	4.0
		5	0.10	92–98	94	2.8	89–97	92	3.4
Orange, fruit	Flupyradifurone	5	0.01	88–97	95	4.1	91–97	96	2.7
		5	0.10	99–106	102	2.8	98–105	101	3.2
Wheat, grain	Flupyradifurone	5	0.01	100–109	105	3.1	100–108	104	3.4
		5	0.10	104–109	106	1.8	100–105	102	1.8
Hop, cone	Flupyradifurone	5	0.05	86–94	91	2.4	84–94	90	3.4
		5	0.50	92–94	93	1.1	88–92	90	1.8

[a] Expressed as BYI 02960

Table 66 Recoveries for method 01330: DFA in plants

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Method: Hilic Column			Hypercarb Column, Confirmatory		
				Transition 95 → 51			Transition 95 → 51		
				Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]
				HPLC-MS/MS			HPLC-MS/MS		
Lettuce, head	DFA	5	0.02	80–104	94	10.5	78–100	91	9.4
		5	0.20	88–101	96	6.1	90–101	97	4.9
Rape, seed	DFA	5	0.02	61–71	65	6.5	66–74	70	5.0
		5	0.20	56–74	62	11.6	60–71	64	7.8
Orange, fruit	DFA	5	0.02	84–104	97	8.6	89–101	97	5.1
		5	0.20	95–99	97	1.7	98–103	101	2.1
Wheat, grain	DFA	5	0.02	83–104	91	8.9	84–90	87	3.1
		5	0.20	82–96	88	5.7	87–93	91	2.6
Hop, cone	DFA	5	0.10	96–105	101	4.2	100–113	108	5.5
		5	1.0	97–105	102	3.2	94–102	98	3.5

An independent laboratory validation was conducted for method 01330 (Konrad 2012a, 2011/0134/01). Samples of lettuce head, orange fruit and wheat grain were fortified with BYI 02960 parent compound and DFA at the nominal fortification levels of 0.01 and 0.10 mg/kg.

Analysis of samples was performed per method 01330 (Schulte & Bauer 2012, RARVP012). Two MRM transitions were measured for BYI 02960, one for quantification and the second for confirmation. For all matrices, for both fortification levels, and for both MRM transitions monitored, the mean recoveries were between 75% and 103%, with relative standard deviations of < 10%. For DFA, one MRM transition was determined using two different HPLC conditions. For all matrices, for both fortification levels, and for both the primary and the confirmatory HPLC procedure, the mean recoveries were between 73 and 100%, except for wheat grain at the 10× LOQ, where the value was 68%. RSDs were < 10% in all cases. A summary of the independent laboratory validation results is in Table 67.

Table 67 Independent laboratory validation recoveries for method 01330: flupyradifurone in plants

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Quantification			Confirmatory		
				Transition 289 → 126			Transition 289 → 90		
				Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]
				HPLC-MS/MS			HPLC-MS/MS		
Lettuce, head	Flupyradifurone	5	0.01	89–92	90	1.4	88–94	92	2.5
		5	0.1	89–94	91	2.0	92–96	94	1.5
Wheat, grain	Flupyradifurone	5	0.01	77–92	86	6.7	67–81	75	7.0
		5	0.1	80–95	91	6.8	84–98	93	6.3

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Quantification			Confirmatory		
				Transition 289 → 126			Transition 289 → 90		
				Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]
				HPLC-MS/MS			HPLC-MS/MS		
Orange, fruit	Flupyradifurone	5	0.01	89–96	92	3.0	92–103	98	4.5
		5	0.1	96–102	99	2.4	99–104	103	1.9

Table 68 Independent laboratory validation recoveries for method 01330: DFA in plants

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Method: Hilic Column			Hypercarb Column, Confirmatory		
				Transition 95 → 51			Transition 95 → 51		
				Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]
				HPLC-MS/MS			HPLC–MS/MS		
Lettuce, head	DFA	5	0.01	92–97	94	2.1	72–81	78	4.3
		5	0.1	96–100	99	1.8	84–91	88	2.9
Wheat, grain	DFA	5	0.01	85–89	87	2.1	84–98	90	6.2
		5	0.1	60–72	68	7.2	71–76	73	2.7
Orange, fruit	DFA	5	0.01	87–106	93	8.3	81–96	89	6.4
		5	0.1	98–103	100	2.5	86–91	88	2.8

The modification M001 of the residue analytical method, 01330, describes the determination of the residues of BYI 02960 (parent compound) and its metabolite DFA in/on rape seed using HPLC-MS/MS (Schulte and Teubner 2012, MR-12/054).

BYI 02960 and DFA are extracted from rape seeds three times, using a mixture of acetonitrile/water (4/1, v/v) with 2.2 mL/L formic acid. After dilution, two aliquots of the raw extract are filtered for separate measurement of BYI 02960 and DFA. Quantitation was done by reverse-phase HPLC and electrospray-MS/MS detection against matrix-matched standards. Two MRM transitions for quantitation and confirmation are monitored for BYI 02960 (*m/z* 289/126 or 90) in rape seed. For DFA, an MRM transition of *m/z* 95/51 is used. No second MRM transition is available, so a Hypercarb column was employed as a different separation system (as opposed to HILIC for the primary determination). Matrix effects are observed for all samples of rape seed.

The accuracy of the method was assessed based on the determined recovery rates. Samples were fortified with BYI 02960 at concentrations of 0.01 and 0.10 mg/kg, while DFA was spiked at 0.02 mg/kg and 0.20 mg/kg, expressed in parent equivalents. Mean recoveries were acceptable. The results are summarized below in Tables 69 and 70.

The correlation between the injected amount of substance and the detector response was linear for standards in matrix in the range from 0.125 to 50 µg/L for BYI 02960 (both mass transitions) and 0.250 to 50 µg/L for DFA (HILIC column) and 0.25 to 25 µg/L (Hypercarb column), using at least five different concentration levels, for both compounds. The correlation coefficients were > 0.99 in all cases. The LOQ was 0.01 mg/kg in rape seed for BYI 02960 and 0.02 mg/kg for DFA.

Table 69 Recoveries for method 01330/M001: flupyradifurone in rape seed

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Quantification			Confirmatory		
				Transition 289 → 126			Transition 289 → 90		
				Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]
				HPLC-MS/MS			HPLC-MS/MS		
Rape, seed	Flupyradifurone	5	0.01	97–106	101	3.7	103–107	105	1.7
		5	0.10	91–99	97	3.7	95–102	100	3.0

Table 70 Recoveries for method 01330/M001: DFA in rape seed

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Method: Hilic Column			Hypercarb Column Confirmatory		
				Transition 95 → 51			Transition 95 → 51		
				Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]
				HPLC-MS/MS			HPLC-MS/MS		
Rape, seed	DFA	5	0.02	80–85	84	2.5	73–82	79	4.7
		5	0.20	71–77	73	3.4	80–99	88	8.8

An independent laboratory validation was conducted for method 01330/M001 (Konrad 2012c, 2012/0082/01). Samples of rape seed were fortified with BYI 02960 parent compound at the nominal fortification levels of 0.01 and 0.10 mg/kg, and at nominal fortification levels of 0.02 and 0.20 mg/kg DFA (*i.e.* the LOQ and the 10-fold LOQ for both analytes).

Analysis of samples was performed per method 01330/M001. Two MRM transitions were measured for BYI 02960, one for quantification and the second for confirmation. For both fortification levels, and for both MRM transitions monitored, the mean recoveries ranged from 81–93%, with relative standard deviations of 2.4–4.7%. For DFA, one MRM transition was determined using two different HPLC conditions. For both fortification levels, and for both the primary and the confirmatory HPLC procedure, the mean recoveries were between 90–103%, with relative standard deviations of 3.0–8.6%. Matrix effects were observed so it was decided to use matrix-matched standards for all matrices to compensate all possible matrix effects. A summary of the independent laboratory validation tests is given in Tables 71 and 72.

Table 71 Independent laboratory validation recoveries for method 01330/M001: flupyradifurone in rape seed

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Quantification			Confirmatory		
				Transition 289 → 126			Transition 289 → 90		
				Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]
				HPLC-MS/MS			HPLC-MS/MS		
Rape, seed	Flupyradifurone	5	0.01	76–85	81	4.7	77–85	82	3.9
		5	0.10	89–94	93	2.4	88–95	92	2.8

Table 72 Independent laboratory validation recoveries for method 01330/M001: DFA in rape seed

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Method: Hilic Column			Hypercarb Column Confirmatory		
				Transition 95 → 51			Transition 95 → 51		
				Range [%]	Mean [%]	RSD [%]	Range [%]	Mean [%]	RSD [%]
				HPLC-MS/MS			HPLC-MS/MS		
Rape, seed	DFA	5	0.02	96–108	103	5.1	90–110	97	8.6
		5	0.20	95–102	99	3.0	86–93	90	3.3

Animal commodities

Method RV-004-A11-04

Method RV-004-A11-04: A residue analytical method, RV-004-A11-04, was developed as a data collection method for the determination of the residues of BYI 02960 (parent compound), and its metabolites DFA, BYI 02960-acetyl-AMCP and BYI 02960-OH in/on animal matrices. The method validation is reported in feeding study reports RARVP050 (cattle, Moore & Harbin 2012) and RARVP041 (poultry, Wade & Netzband 2012), both of which also contain the full method description in a report appendix.

The residues are extracted twice from animal material by diluting liquid matrices (milk, whey, cream, urine) or blending tissue matrices with acetonitrile/water (4/1, v/v) with 2.2 mL/L formic acid.

Milk and fat samples require the addition of pentane and centrifugation. Aliquots of the extracts are purified through a C-$_{18}$ solid-phase extraction column, then amended with a mixture of stable, isotopically labelled internal standards. The final solution is analysed by HPLC-MS/MS.

As this is a data collection method, only one MRM transition was required. Nevertheless, two were monitored for BYI 02960 (*m/z* 289/126 or 90), BYI 02960-acetyl-AMCP (*m/z* 185/107 or 143), and BYI 02960-OH (*m/z* 305/126 or 90) in each matrix tested. Generally, the HPLC-MS/MS method is highly specific and fully satisfactory for data collection. Nevertheless, additional confirmatory procedures were developed for parent BYI 02960 and BYI 02960-OH, as pre-testing showed that the confirmatory ion's sensitivity might be too low in some matrices. Thus, a Gemini C-$_{18}$ column was employed (as opposed to HILIC in the primary method). However, as all validation experiments were successful using HILIC, these alternative confirmatory procedures were not validated further. For DFA, only one MRM transition was available (*m/z* 95/51), so an alternative chromatographic system was chosen for confirmation (Restek Organic Acids column as opposed to HILIC).

The accuracy of the method was assessed on the basis of the determined recovery rates. Samples were fortified with BYI 02960, BYI 02960-acetyl-AMCP, and BYI 02960-OH at concentrations of 0.01 mg/kg in all matrices as well as 4.0 mg/kg in chicken matrices and 0.10 mg/kg and various other levels in bovine matrices and with DFA at 0.01 and 4.0 or 14.0 mg/kg in chicken matrices and at 0.02 and other levels in bovine matrices. Metabolite levels were expressed in parent equivalents. Mean recoveries and RSD values per fortification level for BYI 02960 parent and metabolites were generally acceptable.

The correlation between the injected amount of substance and the detector response was linear in the range from 0.5 to 250 ng/mL, using at least five different concentration levels, for all compounds. The correlation coefficients of the 1/x weighted linear regression were > 0.99 in all cases.

The LOQ for BYI 02960, BYI 02960-acetyl-AMCP, and BYI 02960-OH was 0.01 mg/kg in all matrices tested. For DFA, the LOQ was 0.01 mg/kg in all chicken matrices and 0.02 mg/kg in cattle matrices, except whey, where it was 0.05 mg/kg.

The extraction efficiency of the residue method for the determination of the relevant residues of BYI 02960 in animal matrices, consisting of the parent compound and its metabolites DFA, BYI 02960-acetyl-AMCP, and BYI 02960-OH, was assured by choosing the same extraction procedures as used in the plant metabolism studies, except that formic acid (2.2 mL/L) was added to the extraction solution.

Nevertheless, an extraction efficiency examination was conducted as part of the chicken and cattle feeding studies. Aged residues in respective samples were analysed using procedures described in method RV-004-A11-04 and, in parallel, using the procedures described in the metabolism studies. The sample materials were eggs, fat, liver, and muscle in the poultry report, and kidney and milk in the ruminant report.

Following addition of appropriate isotopically labelled internal standards and clean-up on a C-$_{18}$ Bond Elut column, the samples were analysed by HPLC-MS/MS. The animal residue method RV-004-A11-04 could extract and measure relevant aged residues from all tested matrices, with extraction efficiency ranging from 81–90% for the poultry matrices and 90 and 105% for cattle kidney and milk based on total residues (parent and three metabolites).

Table 73 Recovery results of method RV-004-A11-04: flupyradifurone in animal matrices

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Transition 289 → 126			Confirmatory Transition 289 → 90		
				range [%]	mean [%]	RSD [%]	range [%]	mean [%]	RSD [%]
Chicken egg	Flupyradifurone	17/7	0.01	76–118	96	14.2	80–102	89	9.4
		3	4.0	81–100	92	10.7	73–95	87	13.8
Chicken fat	Flupyradifurone	12/7	0.01	75–105	91	10.1	73–130	96	25.6
		3	4.0	104–110	107	2.9	103–106	104	1.7
Chicken liver	Flupyradifurone	12/7	0.01	84–121	101	11.4	74–120	105	15.0
		3	4.0	100–112	104	6.1	96–108	101	6.2

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Transition 289 → 126			Confirmatory Transition 289 → 90		
				range [%]	mean [%]	RSD [%]	range [%]	mean [%]	RSD [%]
Chicken muscle	Flupyradifurone	12/7	0.01	81–119	105	13.5	81–123	106	13.1
		3	4.0	108–112	109	2.1	102–107	104	2.4
Bovine milk	Flupyradifurone	7	0.01	102–110	108	2.5	99–112	106	4.7
		1	0.025	107	–	–	–	–	–
		5	0.05	97–105	102	3.4	–	–	–
		3	0.10	103–109	105	–	–	106	2.0
		1	0.25	103	–	–	–	–	–
		3	2.0	100–103	102	1.5	–	–	–
Bovine cream	Flupyradifurone	7	0.01	105–118	110	4.0	104–123	111	6.3
		3	0.10	107–113	110	2.8	104–112	109	4.0
		3	1.0	109–111	110	1.0	108–115	111	3.2
Bovine whey	Flupyradifurone	7	0.01	98–105	101	2.8	102–112	108	3.7
		7	0.025	101–106	103	2.0	–	–	–
		3	0.10	101–102	102	0.6	99–103	101	2.1
		3	0.25	103–105	104	1.0	–	–	–
		3	1.0	100–104	102	2.0	–	–	–
Bovine fat	Flupyradifurone	7	0.01	96–107	103	3.6	99–115	107	5.9
		2	0.05	93, 100	97	–	–	–	–
		3	0.1	97–101	99	2.1	96–103	100	3.5
		3	1.5	92–99	95	3.7	–	–	–
Bovine kidney	Flupyradifurone	7	0.01	91–99	96	2.9	92–103	96	4.1
		3	0.05	87–98	93	5.9	–	–	–
		3	0.10	94–96	95	1.2	92–96	94	2.2
		3	6.0	90–97	93	3.9	–	–	–
Bovine liver	Flupyradifurone	7	0.01	84–98	90	5.3	85–98	92	5.6
		3	0.05	96–98	97	1.2	–	–	–
		3	0.10	87–91	89	2.3	88–90	89	1.3
		3	4.0	90–93	91	1.7	–	–	–
Bovine muscle	Flupyradifurone	7	0.01	92–100	96	3.5	100–112	105	3.7
		2	0.05	95, 99	97	–	–	–	–
		3	0.10	93–98	95	3.0	91–97	93	3.4
		3	2.0	89–92	90	1.7	–	–	–

Table 74 Recovery results of method RV-004-A11-04: BYI 02960-acetyl-AMCP in animal matrices

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Transition 185 → 107			Confirmatory Transition 185 → 143		
				range [%]	mean [%]	RSD [%]	range [%]	mean [%]	RSD [%]
Chicken egg	BYI 02960-acetyl-AMCP	16	0.01	70–120	93	12.7	75–99	89	11.1
		3	4.0	81–93	88	7.1	85–97	89	7.5
Chicken fat	BYI 02960-acetyl-AMCP	12	0.01	77–119	94	12.2	82–114	96	13.6
		3	4.0	108–111	109	1.4	106–111	109	2.3
Chicken liver	BYI 02960-acetyl-AMCP	12	0.01	89–116	106	10.0	91–120	111	10.9
		3	4.0	99–115	105	8.6	82–91	86	5.3
Chicken muscle	BYI 02960-acetyl-AMCP	12	0.01	88–35	111	11.7	98–119	111	6.5
		3	4.0	101–108	105	3.6	102–110	104	5.1
Bovine milk	BYI 02960-acetyl-AMCP	7	0.01	101–109	105	2.4	102–107	105	1.7
		1	0.025	112	–	–	–	–	–
		5	0.05	102–109	105	2.9	–	–	–
		3	0.10	107–112	109	2.4	110–111	110	0.5
		1	0.25	103	–	–	–	–	–
		3	2.0	98–99	99	0.6	–	–	–
Bovine cream	BYI 02960-acetyl-AMCP	7	0.01	93–113	102	7.5	99–114	108	5.4
		3	0.10	111–114	112	1.5	110–113	112	1.4
		3	1.0	109–115	113	2.9	108–112	111	2.1
Bovine whey	BYI 02960-acetyl-AMCP	7	0.01	99–106	102	2.3	99–105	102	2.0
		3	0.10	102–103	102	0.6	102	102	2.0

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Transition 185 → 107			Confirmatory Transition 185 → 143		
				range [%]	mean [%]	RSD [%]	range [%]	mean [%]	RSD [%]
Bovine fat	BYI 02960-acetyl-AMCP	7	0.01	94–100	97	2.0		96	3.3
		2	0.05	97, 98	98	–	–	–	–
		3	0.1	96–101	98	2.6	–	–	0.6
		3	1.5	101–106	103	2.6	–	–	–
Bovine kidney	BYI 02960-acetyl-AMCP	7	0.01	96–105	100	3.0	92-106	99	5.3
		3	0.05	90-100	95	5.3	–	–	–
		3	0.10	97–99	98	1.0	95–97	96	1.0
		3	6.0	91–95	93	2.2	–	–	–
Bovine liver	BYI 02960-acetyl-AMCP	7	0.01	80–104	92	9.5	82–101	92	7.1
		3	0.05	95–109	102	6.9	–	–	–
		3	0.10	92–98	94	3.4	93–96	95	1.6
		3	6.0	93–94	94	0.6			
Bovine muscle	BYI 02960-acetyl-AMCP	7	0.01	89–101	97	4.7	88–103	95	6.2
		2	0.05	95–103	99	–	–	–	–
		3	0.10	91–103	96	6.7	91–102	95	6.4
		3	2.0	95–96	96	0.6	–	–	–

Table 75 Recovery results of method RV-004-A11-04: DFA in animal matrices

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Method (HILIC column)			Confirmatory Transition (RESTEK organic acids column)		
				range [%]	mean [%]	RSD [%]	range [%]	mean [%]	RSD [%]
Chicken egg	DFA	17	0.01	68–112	82	15.9	74–101	88	10.7
		3	4.0	70–75	72	3.7	67–77	7.1	7.2
Chicken fat	DFA	12	0.01	74–97	87	9.4	74–101	81	8.4
		3	4.0	92–100	96	4.2	67–77	93	2.2
Chicken liver	DFA	12	0.01	75–103	86	9.9	83–105	94	7.9
		3	4.0	82–91	86	5.3	84–95	90	6.1
Chicken muscle	DFA	12	0.01	82–118	95	10.8	79–117	98	14.2
		3	4.0	80–86	83	3.6	80–81	81	0.7
Bovine milk	DFA	7	0.02	76–92	85	6.3	78–95	90	7.3
		7	0.05	77–93	86	8.5	–	–	–
		3	0.20	91–94	92	1.7	88–91	89	1.9
		3	0.40	85–93	88	5.3	–	–	–
Bovine cream	DFA	7	0.02	85–103	95	7.1	78–92	86	5.7
		3	0.20	101–106	103	2.6	97–98	98	0.6
Bovine whey	DFA	7	0.05	87–96	93	3.9	85–97	92	4.2
		3	0.50	94–102	98	4.1	93–99	96	3.2
Bovine fat	DFA	7	0.02	81–96	89	6.0	87–100	92	5.3
		2	0.05	89, 93	91	–	–	–	–
		3	0.60	88–90	89	1.1	85–90	87	2.5
Bovine kidney	DFA	7	0.02	66–79	72	6.7	76–89	82	5.7
		2	0.05	69, 72	71	–	–	–	–
		3	0.80	79–84	82	3.5	81–85	83	2.5
Bovine liver	DFA	7	0.02	68–97	79	12.7	70–77	74	4.0
		2	0.05	64, 72	68	–	–	–	–
		3	0.60	79–89	83	6.7	76–86	80	6.4
Bovine muscle	DFA	7	0.02	66–78	74	5.7	61–73	66	6.5
		3	0.05	70–78	73	6.0	–	–	–
		3	0.50	65–73	68	6.1	67–73	70	4.3

Table 76 Recovery results of method RV-004-A11-04: BYI 02960-OH in animal matrices

Matrix	Analyte	No. of tests	Spiking level	Primary Transition 305 → 126	Confirmatory Transition 305 → 90

			[mg/kg]	range [%]	mean [%]	RSD [%]	range [%]	mean [%]	RSD [%]
Chicken egg	BYI 02960-OH	17	0.01	72–113	94	12.0			
		3	4.0	89–102	96	6.8			
Chicken fat	BYI 02960-OH	12	0.01	86–106	94	7.3			
		3	4.0	113–118	116	2.3			
Chicken liver	BYI 02960-OH	12	0.01	71–120	88	15.6			
		3	4.0	106–108	107	0.9			
Chicken muscle	BYI 02960-OH	12	0.01	81–120	106	10.7			
		3	4.0	108–112	110	1.8			
Bovine milk	BYI 02960-OH	7	0.01	105–115	110	2.9	96–112	106	5.1
		1	0.025	106	–	–	–	–	–
		5	0.05	100–106	103	2.7	–	–	–
		3	0.10	105–110	108	2.3	102–111	107	4.3
		1	0.25	105	–	–	–	–	–
		3	2.0	101–105	102	2.3	–	–	–
Bovine cream	BYI 02960-OH	7	0.01	107–124	114	4.9	108–121	114	4.8
		3	0.10	110–113	111	1.4	110–114	111	2.1
		3	1.0	111–114	113	1.5	107–114	110	3.2
Bovine whey	BYI 02960-OH	7	0.01	100–104	103	1.5	95–133	116	11.3
		7	0.025	104–112	108	2.3	–	–	–
		3	0.10	100–102	101	1.0	111–118	115	3.1
		3	0.25	106–112	108	3.0	–	–	–
Bovine fat	BYI 02960-OH	7	0.01	101–109	106	2.5	94–111	101	5.7
		2	0.05	98, 105	102	–	–	–	–
		3	0.10	97–106	101	4.4	97–103	101	3.4
		3	1.5	94–98	96	2.2	–	–	–
Bovine kidney	BYI 02960-OH	7	0.01	90–105	97	5.8	83–97	92	4.6
		3	0.05	93–97	95	2.1	–	–	–
		3	0.10	98	98	0.0	100–101	100	0.6
		3	6.0	94–100	96	3.3	–	–	–
Bovine liver	BYI 02960-OH	7	0.01	87–95	90	3.2	85–97	92	3.9
		3	0.05	93–100	97	3.7	–	–	–
		3	0.10	91–92	91	0.6	88–94	92	3.5
		3	4.0	95–98	97	1.6	–	–	–
Bovine muscle	BYI 02960-OH	7	0.01	93–106	99	4.3	78–102	91	10.8
		2	0.05	96, 117	107	–	–	–	–
		3	0.10	94–101	97	3.7	92–107	98	8.1
		3	2.0	92	92	0.0	–	–	–

Method 01214

Method 01214 (enforcement method): Method 01214 was developed for the determination of the residues of BYI 02960 (parent compound), and its metabolite DFA in/on animal matrices (Schulte and Bauer 2012b, MR-011/144).

The residues are extracted twice with acetonitrile/water (4/1, v/v), with the addition of n-heptane in the cases of fat and milk. The materials tested included bovine muscle, liver, kidney, and milk; and chicken fat and egg. After the addition of formic acid and dilution, two aliquots of the raw extract were filtered for separate measurement of the parent BYI 02960 and its metabolite DFA. The solution was analysed by HPLC-MS/MS; residues were quantified against matrix-matched standards.

Two MRM transitions for quantitation and confirmation were monitored for BYI 02960 (m/z 289/126 or 90) in each matrix tested. For DFA, no second MRM transition is available. A Hypercarb column was employed as a different separation system (as opposed to HILIC for the primary determination). The confirmatory methods were fully validated; hence the quantitation and confirmation methods can be used interchangeably.

The accuracy of the method was assessed on the basis of the determined recovery rates. Samples were fortified with BYI 02960 at concentrations of 0.01 and 0.10 mg/kg while DFA was spiked at 0.02 and 0.20 mg/kg, expressed in parent equivalents. Mean recoveries per fortification level for the primary method for both analytes and all matrices were in a range of 90-108%, with one exception at 112% (BYI 02960, muscle, 0.01 mg/kg). Using the confirmatory conditions, mean values per fortification level were 91–108% in all matrices except for BYI 02960, muscle, at 0.01 mg/kg (112%). In both cases of higher recovery, the RSD was low (6.7 and 7.2%), so that these higher values were acceptable. The results are summarized below in Tables 77 and 78.

The correlation between the injected amount of substance and the detector response was linear for standards in matrix in the range from 0.125 to 100 µg/L, using at least five different concentration levels, for both compounds. The correlation coefficients of the 1/x weighted linear regression were > 0.99 in both cases. Linearity was proven for the confirmatory method as well, over the same concentration range and, again, with correlation coefficients of > 0.99. The LOQ for BYI 02960 was 0.01 mg/kg in all matrices tested. For DFA, the LOQ was 0.02 mg/kg.

Table 77 Recovery results of method 01214: flupyradifurone in animal matrices

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Transition 289 → 126			Confirmatory Transition 289 → 90		
				range [%]	mean [%]	RSD [%]	range [%]	mean [%]	RSD [%]
Bovine muscle	Flupyradifurone	5	0.01	100–119	112	6.7	98–119	112	7.2
		5	0.10	105–112	108	3.0	105–112	107	2.8
Bovine liver	Flupyradifurone	5	0.01	77–107	99	12.6	76–108	99	13.3
		5	0.10	96–101	99	1.9	96–104	100	3.2
Bovine kidney	Flupyradifurone	5	0.01	100–105	104	2.0	104–106	105	0.9
		5	0.10	83–102	94	7.7	82–100	93	7.4
Chicken fat	Flupyradifurone	5	0.01	101–113	107	4.2	103–109	105	2.2
		5	0.10	103–114	107	4.4	102–111	107	3.7
Bovine milk	Flupyradifurone	5	0.01	103–109	104	2.5	99–109	104	3.7
		5	0.10	93–109	103	5.9	93–109	103	6.3
Chicken egg	Flupyradifurone	5	0.01	96–99	97	1.1	93–104	96	4.7
		5	0.10	84–102	93	7.4	87–102	95	6.5

Table 78 Recovery results of method 01214: DFA in animal matrices

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Method Hilic Column			Confirmatory Method Hypercarb column		
				range [%]	mean [%]	RSD [%]	range [%]	mean [%]	RSD [%]
Bovine muscle	DFA	5	0.02	104–107	106	1.1	105–112	108	2.1
		5	0.20	97–105	100	3.1	103–107	105	1.6
Bovine liver	DFA	5	0.02	72–105	95	13.9	72–103	95	13.6
		5	0.20	93–105	101	4.7	88–94	91	2.5
Bovine kidney	DFA	5	0.02	103–108	106	1.8	96–104	100	3.4
		5	0.20	94–101	97	3.0	88–102	94	5.9
Chicken fat	DFA	5	0.02	99–107	105	3.1	105–109	106	1.6
		5	0.20	101–111	104	3.7	99–105	102	2.8
Bovine milk	DFA	5	0.02	101–110	107	3.3	101–105	103	1.5
		5	0.20	92–108	102	5.9	93–104	101	4.5
Chicken egg	DFA	5	0.02	89–98	92	4.1	98–102	100	1.5
		5	0.20	81–99	90	8.1	86–98	94	5.0

An independent laboratory validation was performed for method 01214 (Konrad 2012b, 2011/0164/01). Samples of all matrices covered by the main method itself were fortified with BYI 02960 parent compound at the nominal fortification levels of 0.01 and 0.10 mg/kg, *i.e.* the LOQ and the 10-fold LOQ and with DFA at 0.02 and 0.20 mg/kg.

Analysis of samples was performed per method 01214 (Schulte & Bauer, 2012; 3.1.2.2/01). Two MRM transitions were measured for BYI 02960. For all matrices, for both fortification levels, and for both MRM transitions monitored, the mean recoveries were between 87% and 101%, with relative standard deviations of < 15%. For DFA one MRM transition was determined using two different HPLC conditions. For all matrices, for both fortification levels, and for both the primary and the confirmatory HPLC procedure, the mean recoveries were between 73 and 99%. RSDs were < 15% in all cases.

Table 79 Independent laboratory validation recoveries for method 01214: flupyradifurone in animal matrices

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Transition 289 → 126			Confirmatory Transition 289 → 90		
				range [%]	mean [%]	RSD [%]	range [%]	mean [%]	RSD [%]
Bovine muscle	Flupyradifurone	5	0.01	93–98	96	2.3	88–97	94	3.9
		5	0.10	90–96	94	2.5	92–98	96	2.6
Bovine liver	Flupyradifurone	5	0.01	97–100	98	0.9	95–101	98	2.5
		5	0.10	96–101	98	1.8	96–102	99	2.6
Bovine kidney	Flupyradifurone	5	0.01	92–94	93	0.8	93–98	95	2.2
		5	0.10	88–92	90	1.9	90–97	94	2.5
Bovine fat	Flupyradifurone	5	0.01	90–100	96	4.2	93–100	97	2.6
		5	0.10	89–97	93	3.6	92–99	95	3.5
Bovine milk	Flupyradifurone	5	0.01	98–101	100	1.0	96–102	99	2.8
		5	0.10	91–97	95	2.2	94–100	97	2.3
Chicken egg	Flupyradifurone	5	0.01	91–109	98	8.3	90–108	99	9.4
		5	0.10	84–90	87	3.1	89–92	90	1.7

Table 80 Independent laboratory validation recoveries for method 01214: DFA in animal matrices

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Primary Method Hilic Column			Confirmatory Method Hypercarb column		
				range [%]	mean [%]	RSD [%]	range [%]	mean [%]	RSD [%]
Bovine muscle	DFA	5	0.02	95–100	98	2.1	72–74	73	1.3
		5	0.20	83–91	87	3.4	79–88	84	3.8
Bovine liver	DFA	5	0.02	79–105	89	11.1	72–96	79	12.3
		5	0.20	94–101	97	2.8	86–92	90	2.8
Bovine kidney	DFA	5	0.02	93–106	100	4.9	71–79	74	4.4
		5	0.20	89–94	92	2.2	76–78	77	1.4
Bovine fat	DFA	5	0.02	90–100	97	4.4	73–76	75	1.5
		5	0.20	86–96	91	4.1	85–94	89	3.6
Bovine milk	DFA	5	0.02	97–107	101	3.7	75–85	78	5.0
		5	0.20	94–98	96	1.6	83–86	84	1.6
Chicken egg	DFA	5	0.02	92–105	98	5.7	78–83	80	2.3
		5	0.20	90–102	94	5.0	81–96	85	7.2

Stability of pesticide residues in stored analytical samples

Plant matrices

To determine the freezer storage stability of the residues of BYI 02960 in plant materials, individual 5 g control samples of homogenised orange fruit (high acid content), spinach leaves and tomato fruit (high water content), wheat grain (high starch content), bean seed (high protein content), coffee bean and soya bean seed (high oil content) and sugar cane were separately fortified with 5.0 µg of either BYI 02960 parent compound, DFA or DFEAF (Netzband, Timberlake and Harbin 2012, RARVP046-1). This resulted in a fortification level of 1.0 µg/g (1 mg/kg) of each analyte. Except for the Day-0 analysis, samples were stored in glass containers in a freezer at an average temperature of –23 °C.

For Day-0 analysis, three treated samples of each material were chosen, as well as one control sample of each. Samples were then also analysed after nominal intervals of 1, 2½, 5–6, 12 and 18 months. (The intended final storage period to be covered in this study is 2 years.) At each of these intervals, two treated samples of each material were removed from storage and analysed, as well as a control sample and two samples for concurrent recovery. Analyses of parent compound, DFA and DFEAF were carried out using the data gathering method (01304).

The storage stability data for BYI 02960 and its metabolites is summarized below in Table 81.

Table 81 Summary of stability data for deep frozen samples fortified at 1 mg/kg with Parent, DFA or DFEAF

Matrix	Storage period (d)	Parent		DFA		DFEAF	
		Concurrent recovery (%)	Recovery in stored sample (%)	Concurrent recovery (%)	Recovery in stored sample (%)	Concurrent recovery (%)	Recovery in stored sample (%)
sugar cane	0	(100)	100	(100)	98	(100)	110
	29	101	92	97	96	89	81
	77	91	85	97	92	93	88
	149	98	94	86	100	117	112
	372	87	93	97	99	87	93
	559	89	92	93	137	83	94
coffee bean	0	(100)	94	(100)	90	(100)	101
(green)	33	97	93	90	89	100	94
	81	81	81	77	76	89	88
	152	85	104	80	94	118	100
	370	93	83	73	79	90	86
	560	91	94	85	108	88	89
orange fruit	0	(100)	96	(100)	95	(100)	104
	28	89	84	98	100	100	109
	77	86	95	89	99	98	94
	148	104	84	87	93	119	110
	365	102	80	94	98	97	92
	556	80	90	92	119	97	85
soya bean seed	0	(100)	93	(100)	79	(100)	107
	28	93	93	89	71	99	106
	75	93	96	93	82	103	94
	148	91	97	75	98	117	117
	371	96	98	73	79	84	85
	558	95	89	79	87	97	87
navy bean	0	(100)	111	(100)	96	(100)	102
	26	91	107	96	99	109	102
	75	94	110	98	108	107	93
	148	100	123	79	100	116	103
	364	113	127	80	78	93	88
	558	100	94	99	128	114	104
tomato fruit	0	(100)	95	(100)	101	(100)	110
	28	90	92	99	96	96	116
	76	86	89	94	94	103	100
	148	107	105	84	98	118	101
	370	89	95	98	105	97	84
	558	102	94	102	134	111	89
spinach	0	(100)	91	(100)	100	(100)	106
	26	96	97	98	97	107	109
	75	90	85	90	87	101	96
	147	94	104	85	100	114	109
	364	119	97	70	74	105	95
	557	98	100	107	147	102	95
wheat grain	0	(100)	94	(100)	92	(100)	103
	27	93	90	97	98	101	106
	76	89	82	88	94	96	91
	186	97	109	78	98	104	99
	362	92	77	72	71	92	90
	557	97	91	88	116	100	87

At Day 0, average residue recoveries of BYI 02960 ranged from 91–111% of nominal, of DFEAF from 101–110% of nominal and of DFA from 79–101% of nominal. In samples analysed after approximately 18 months of frozen storage, storage stability recoveries, ranged from 89–100% for BYI 02960, 85–104% for DFEAF and 87–147% for DFA. Storage stability after approximately 18 months of frozen storage adjusted for concurrent recoveries, were 92–113% for BYI 02960, 80–112% for DFEAF and 111–147% for DFA.

At all sampling dates and in all sample materials, the relevant components of the residue of BYI 02960 were above 70%. Even in the case of the lower values in the given ranges, there was no evidence of any continued degradation of any of the analytes in any of the sample materials. Thus, all analytes can be considered stable in all relevant plant matrix types for a period of at least 18 months (556 to 560 days).

Animal matrices

All samples in the laying hen feeding study were analysed within thirty days of collection. Therefore, was no necessity for freezer storage stability data. In the dairy cattle feeding study, the tissues and milk samples were analysed within 25 days of collection for residues of BYI 02960, BYI 02960-OH and BYI 02960-AMCP and therefore freezer storage stability data was not required for these analytes. Samples of fat, kidney, liver and muscle were stored under freezer conditions for 41 days prior to analysis for DFA residues therefore storage stability data was necessary.

To determine the freezer storage stability, individual control samples of bovine fat, kidney, liver, and muscle were separately fortified with DFA, at a nominal concentration of 0.20 mg/kg (Moore and Harbin 2012, RARV P050-1). Samples were stored in a freezer at a temperature of –15 °C. For Day-0 analysis, three treated samples of each material were chosen, as well as one control sample of each. Samples were then analysed after an interval of 43 days, in order to cover the longest period of storage for these matrices in the GLP feeding study. At this interval, two treated samples of each material were removed from storage and analysed, as well as a control sample and two samples for concurrent recovery. Results are summarized below in Table 82.

Table 82 Summary of stability data for deep frozen samples fortified at 0.2 mg/kg with DFA

Matrix	Storage period (d)	Concurrent recovery (%)	Recovery in stored samples (%)
bovine fat	0	–	85.8
	43	87.8	86.0
bovine kidney	0	–	68.7
	43	72.5	72.5
bovine muscle	0	–	65.3
	43	69.0	69.0
bovine liver	0	–	60.3
	43	68.0	65.8

At Day 0, average residue recoveries of DFA ranged from 60–86% of nominal. In samples analysed after 43 days of frozen storage, storage stability recoveries, ranged from 66–86%. There was no evidence of any continued degradation of DFA in any of the sample materials. Thus, it can be considered stable in all relevant animal matrix types over the tested period of 43 days.

USE PATTERN

Information on registered uses made available to this Meeting is shown in Table 83.

Table 83 Registered uses of flupyradifurone on bushberry fruits, low growing berry fruits, small fruit vine climbing, citrus fruit, pome fruits, leafy vegetables, fruiting vegetables cucurbits, fruiting vegetables other than cucurbits, legume vegetables, root vegetables, tuber vegetables, cereal grains, peanuts, cotton, tree nuts and forage and fodder crops

Crop	Country	Formulation		Application				PHI [days]
		g ai/L or [g ai/kg]	Type	Method	Timing [Interval – days]	Rate [g ai/ ha]	Season Max. [g ai/ ha/year] or (no. per crop)	
Citrus Fruits								
Crop Group 10-10	USA	200	SC	Foliar	10	205	409	1
Citrus Fruits	Guatemala, Belize, Honduras, Nicaragua, Costa Rica, Panama, Cuba, Dominican Republic	200	SL	Foliar	7–10	200	2–3 applications per crop cycle	1
Crop Group 10-10	USA	200	SC	Soil	–	409	409	30
Citrus Fruits	Guatemala, Belize, Honduras, Nicaragua, Costa Rica, Panama, Cuba, Dominican Republic	200	SL	Soil	–	300	300	1
Pome Fruits								
Crop Group 11-10	USA	200	SC	Foliar	10	205	409	14
Prickly Pear/ Cactus Pear								
Prickly Pear/ Cactus Pear	USA	200	SC	Foliar	7	205	409	21
Bushberries								
Crop Group 13-07B (except cranberry)	USA	200	SC	Foliar	7	205	409	3
Small Fruit Vine Climbing (except Fuzzy kiwifruit)								
Crop Subgroup 13-07F	USA	200	SC	Foliar	10	205	409	0
Crop Subgroup 13-07F	USA	200	SC	Soil	–	409	409	30
Low growing berry								
Crop Group 13-07G	USA	200	SC	Foliar	10	205	409	0
Hops								
Hops	USA	200	SC	Foliar	–	153	153	21
Tree nuts								
Crop Group 14 (except almond)	USA	200	SC	Foliar	14	205	409	7
Root Vegetables								
Crop Subgroup 1B	USA	200	SC	Foliar	10	205	409	7
Tuberous and Corm Vegetables								
Crop Subgroup 1C	USA	200	SC	Foliar	7	205	409	7
Potato	Guatemala, Belize, Honduras, Nicaragua, Costa Rica, Panama, Cuba, Dominican Republic	200	SL	Foliar	7–10	200	2–3 applications per crop cycle	7
Potato	Guatemala, Belize, Honduras, Nicaragua, Costa Rica, Panama, Cuba, Dominican Republic	200	SL	Soil	–	300	300	7
Leafy Vegetables								
Crop Group 4	USA	200	SC	Foliar	7	205	409	1
Brassica (Cole) Leafy Vegetables								
Crop Group 5	USA	200	SC	Foliar	7	205	409	1
Legume Vegetables (succulent or dried)								

Crop	Country	Formulation		Application				PHI [days]
		g ai/L or [g ai/kg]	Type	Method	Timing [Interval – days]	Rate [g ai/ ha]	Season Max. [g ai/ ha/year] or (no. per crop)	
Crop Group 6 forage, leaves, vines, pods, cutting for hay or seed (fresh or dry, except dry soya bean seed)	USA	200	SC	Foliar	10	205	409	7
Crop Group 6 dry soya bean seed	USA	200	SC	Foliar	10	205	409	21
Cucurbit Vegetables								
Crop Group 9	USA	200	SC	Foliar	7	205	409	1
Melon, Cucumber, Watermelon	Guatemala, Belize, Honduras, Nicaragua, Costa Rica, Panama, Cuba, Dominican Republic	200	SL	Foliar	7–10	200	2–3 applications per crop cycle	1
Crop Group 9	USA	200	SC	Soil	–	409	409	21
Melon, Cucumber, Watermelon	Guatemala, Belize, Honduras, Nicaragua, Costa Rica, Panama, Cuba, Dominican Republic	200	SL	Soil	–	300	300	1
Crop Group 9 (seedlings for transplants)	USA	200	SC	Planthouse	–	2/10,000 plants		21
Fruiting Vegetables								
Crop Group 8-10	USA	200	SC	Foliar	7	205	409	1
Tomato, Chilli	Guatemala, Belize, Honduras, Nicaragua, Costa Rica, Panama, Cuba, Dominican Republic	200	SL	Foliar	7–10	200	2–3 applications per crop cycle	1
Crop Group 8-10	USA	200	SC	Soil	–	409	409	45
Tomato, Chilli	Guatemala, Belize, Honduras, Nicaragua, Costa Rica, Panama, Cuba, Dominican Republic	200	SL	Soil	–	300	300	1
Crop Group 8-10 (seedlings for transplants)	USA	200	SC	Planthouse	–	2/10,000 plants		45
Cereal Grains								
Crop Group 15 (except rice) dried grain, stover or straw)	USA	200	SC	Foliar	7	205	409	21
Crop Group 15 (except rice) hay, forage, sweet corn	USA	200	SC	Foliar	7	205	409	7
Oilseeds								
Cotton	USA	200	SC	Foliar	10	205	409	14
Peanut	USA	200	SC	Foliar	10	205	409	7
Non-grass animal feeds (alfalfa and clover only)—foliar forage, straw and hay								
Forage, Silage, Alfalfa Hay	USA	200	SC	Foliar	10	205		7
Clover hay	USA	200	SC	Foliar	10	205		14

Note: When both foliar and soil treatments are allowed only one application method can be used, up to the seasonal maximum rate.

Crops of Crop Subgroup 1B Including:

Beet (garden), Burdock (edible), Carrot, Celeriac (celery root), Chervil (turnip-rooted), Chicory, Ginseng, Horseradish, Parsley (turnip-rooted), Parsnip, Radish, Oriental radish (daikon), Rutabaga, Salsify (black), Salsify (oyster plant), Salsify (Spanish), Skirret, Turnip

Crops of Crop Subgroup 1C Including:

Arracacha, Arrowroot, Artichoke (Chinese and Jerusalem), Canna (edible), Cassava (bitter and sweet), Chayote (root), Chufa, Dasheen (taro), Ginger, Leren, Potato, Sweet potato, Tanier (cocoyam), Turmeric, Yam bean (jicama, manioc pea), Yam (true)

Crops of Crop Group 4 Including:

Amaranth (leafy amaranth, Chinese spinach, tampala), Arugala (Roquette), Cardoon, Celery, Celtuce, Chervil, Chinese celery, Chrysanthemum (edible-leaved and garland), Corn salad, Cress (garden), Cress (upland, yellow rocket, winter cress), Dandelion, Dock (sorrel), Endive, Florence fennel (sweet anise, sweet fennel, Finocchio), Lettuce (head and leaf), Orach, Parsley, Purslane (garden and winter), Radicchio (red chicory), Rhubarb, Spinach [including New Zealand and vine (malabar spinach, Indian spinach)], Swiss chard, Taro leaves

Crops of Crop Group 5 Including:

Broccoli, Broccoli raab (*rapini*), Brussels sprouts, Cabbage, Cauliflower, Cavalo broccolo, Chinese broccoli (*gai lon*), Chinese cabbage (*bok choy*), Chinese cabbage (*napa*), Chinese mustard cabbage (*gai choy*), Collards, Kale, Kohlrabi, Mizuna, Mustard greens, Mustard spinach, Rape greens, Turnip greens

Crops of Crop Group 6 Including:

Edible Podded and Succulent Shelled Pea and Bean and Dried Shelled Pea and Bean

Bean (*Lupinus* spp. including grain lupin, sweet lupin, white lupin, and white sweet lupin)

Bean (*Phaseolus* spp., including field bean, kidney bean, lima bean, navy bean, pinto bean, runner bean, snap bean, tepary bean, wax bean)

Bean (*Vigna* spp. Including adzuki bean, asparagus bean, blackeyed pea, catjang, Chinese longbean, cowpea, Crowder pea, moth bean, mung bean, rice bean, Southern pea, urd bean, yardlong bean)

Pea (*Pisum* spp. Including dwarf pea, edible-pod pea, English pea, field pea, garden pea, green pea, snow pea, sugar snap pea)

Other Beans and Peas (Broad bean (fava bean), Chickpea (garbanzo bean), Guar, Jackbean, Lablab bean (hyacinth bean, Lentil, Pigeon pea, soya bean (immature seed), Sword bean)

Soya bean

Crops of Crop Group 8-10 Including:

Cocona, Eggplant (including: African, Pea and Scarlet eggplants), Garden huckleberry, Goji berry, Groundcherry, Martynia, Naranjilla, Okra, Pepino, Pepper (including all peppers i.e. bell, non-bell, hot, sweet, etc), Roselle, Sunberry, Tomatillo, Tomato (including: Bush, Currant, Tree) including cultivars, varieties and/or hybrids of theses commodities

Crops of Crop Group 9 Including:

Chayote (fruit), Chinese waxgourd (Chinese preserving melon), Citron melon, Cucumber, Gherkin, Gourd (edible, includes hyotan, cucuzza, hechima, Chinese okra), *Momordica* spp. (includes balsam apple, balsam pear, bitter melon, Chinese cucumber), Muskmelon (hybrids and/or cultivars of *Cucumis melo* including true cantaloupe, cantaloupe, casaba, Crenshaw melon, golden pershaw melon, honeydew melon, honey balls, mango melon, Persian melon, pineapple melon, Santa Claus melon, snake melon), Pumpkin, Squash (includes summer squash types such as: crookneck squash, scallop squash, straightneck squash, vegetable marrow, zucchini, and winter squash types such as acorn squash, butternut squash, calabaza, cushaw, Hubbard squash, spaghetti squash), Watermelon (includes hybrids and/or varieties of Citrullus lanatus)

Crops of Crop Group 10-10 Including:

Calamondin, Citrus citron, Citrus hybrids (*Citrus* spp., *Eremocitrus* spp., *Fortunella* spp., *Microcitrus* spp. and *Poncirus* spp., Grapefruit (including Japanese summer), Kumquat, Lemon, Lime, Lime (Sweet, Australian desert, Australian finger, Australian round, Brown River finger, Mount White, New Guinea wild, Russell River, Tahiti), Mandarin (Mediterranean, Satsuma), Orange (sour, sweet, Tachibana, Trifoliate), Pummelo, Tangelo, Tangerine [includes Tangerine (mandarin or mandarin orange). Clementine, Mediterranean mandarin, Satsuma mandarin, Tangelo, Tangor, cultivats and varieties], Tangor, Uniq fruit, and cultivars, varieties and/or hybrids of these commodities.

Crops of Crop Group 11-10 Including:

Apple, Azarole, Crabapples (Chinese apple, Chinese crab apple, Chinese flowering apple, Crab apple, Cutleaf crab apple, Florentine crab apple, Hall crab apple, Iowa crab apple, Japanese crab apple, Kai do crab apple, Manchurian crab apple, Paradise apple, Sargent's crab apple, Siberian crab apple, Soulard crab apple, Southern crab apple, Sweet crab apple, Tea crab apple, Toringa crab apple, Western Crabapple, Yunnan crab apple, and varieties and/or hybrids of these), Loquat, Mayhaw, Medlar, Pear, Asian pear, Quince, Chinese quince, Japanese quince, Tejocote, and cultivars, varieties and/or hybrids of these

Crops of Crop Subgroup 13-07B (except cranberry) Including:

Aronia berry, Blueberry (*Vaccinium* spp. – highbush, lowbush and cultivars and/or hybrids of these [=all bluberry species]), Chilean guava, Currant (black, buffalo, native and red), Elderberry, European barberry, Gooseberry (*Ribes* spp), Honeysuckle (edible), Huckleberry, Jostaberry, Juneberry, Lingonberry, Salal, Sea buckthorn, and cultivars, varieties and/or hybrids of these

Crops of Crop Subgroup 13-07F Including:

Amur river grape, Gooseberry (*Ribes* spp.), Grape, Kiwifruit (hardy, only), Maypop, Schisandra berry, and cultivars, varieties and/or hybrids of these

Crops of Crop Subgroup 13-07G (except cranberry) Including:

Bearberry, Bilberry, Blueberry (lowbush), Cloudberry, Lingonberry, Muntries, Partridgeberry, Strawberry, plus cultivars, varieties and/or hybrids of these

Crops of Crop Group 14 (except almond) Including:

Beech nut, Brazil nut, Butternut, Cashew, Chestnut, Chinquapin, Hazelnut (filbert), Hickory nut, Macadamia nut (bush nut), Pecan, Pistachio, Walnut [including black and English (Persian) walnuts]

Crops of Crop Group 15 (except rice) Including:

Barley, Buckwheat, Corn (including: field corn, seed corn, sweet corn and popcorn), Millet (pearl and proso), Oats, Rye, Sorghum, Teosinte, Triticale and Wheat

The label for Sivanto 200SL (U.S.A.) states that immediate plant-back applies to the following crops:

Cereal grains (except rice), cotton, non-grass animal feeds (alfalfa and clover only), peanut, root vegetables (except sugar beet), tuberous and corm vegetables, leafy vegetables, brassica (cole) leafy vegetables, legume vegetables (succulent or dried), fruiting vegetables, cucurbit vegetables, hop, citrus fruit, pome fruit, bushberry (except cranberry), low growing berry (except cranberry), small fruit vine climbing (except fuzzy kiwifruit), tree nut (except almond) prickly pear/cactus pear.

Bulb vegetable (*Allium* Spp.), Group 3-07 including: Chive (fresh leaves), Chinese chive (fresh leaves), Daylily (bulb), Elegans hosta, Fritillaria (bulb and leaves), Garlic (common group, great-headed group, serpent group), Kurrat group, Leek group (including common, lady's, wild), Lily (bulb), Onion (bulb and green leaves including: common group, Beltsville bunching, Chinese bulb, fresh, green, macrostem, Pearl group, potato onion group, tree onion-tops, Welsh-tops), Shallot (bulb, fresh leaves), and cultivars, varieties, and/or hybrids of these.

For crops not listed in the immediate plant-back section of this label, or for crops for which no tolerances for the active ingredient have been established, a 12-month plant-back interval must be observed, except for sugarcane (Florida only).

Group	Commodity	Country/ Countries	Table
FC Citrus Fruits	Mandarins	USA	85
	Grapefruit	USA	86
	Lemons	USA	87
	Oranges	USA	88
FP Pome Fruits	Apples	USA	89
	Pears	USA, Canada	90
FB Berries and other small fruits	Blueberries	USA, Canada, Australia, Chile, New Zealand, United Kingdom, Italy, Spain, Denmark	91
	Grapes	USA, Canada	92
	Strawberries	USA, Canada	93
FI Assorted Tropical and Sub-Tropical Fruits	Prickly Pear	USA	94
VA Bulb Vegetables	Bulb Onions	USA, Canada	95
	Green Onions	USA	96
VB Brassica Vegetables	Broccoli	USA, Canada	97
	Cabbage	USA, Canada	98
	Cauliflower	USA, Canada	99
VC Fruiting Vegetables, Cucurbits	Cucumber	USA, Canada	100
	Summer squash	USA, Canada	101
	Melons	USA	102
VO Fruiting Vegetables, other than Cucurbits	Tomatoes	USA, Canada	103
	Pepper (bell and non-bell)	USA, Canada	104
	Chilli peppers	USA	105
	Sweet Corn	USA, Canada	106

Group	Commodity	Country/ Countries	Table
VL Leafy Vegetables (including Brassica Leafy Vegetables)	Mustard greens	USA	107
	Spinach	USA, Canada	108
	Head lettuce	USA, Canada	109
	Leaf lettuce	USA, Canada	110
VP Legume Vegetables	Common bean	USA, Canada	111
	Snow peas	USA, Canada	112
	Lima Beans	USA, Canada	113
	Peas	USA	114
VD Pulses	Beans (dried seed)	USA, Canada	115
	Peas (dried seed)	USA, Canada	116
	Soya beans (dried seed)	USA, Canada	117
VR Root and Tuber Vegetables	Carrot	USA, Canada	118
	Radish	USA, Canada	119
	Potato	USA, Canada	120
VS Stalk and Stem Vegetables	Celery		121
GC Cereal Grains	Barley	USA, Canada	122
	Wheat	USA, Canada	123
	Sorghum	USA	124
	Maize/Corn	USA, Canada	125
TN Tree Nuts	Almonds	USA	126
	Pecans	USA	127
SO Oilseeds	Cotton	USA	128
	Peanuts	USA	129
SB Seeds for Beverages and Sweets	Coffee Beans	Guatemala, Mexico, Brazil	130
DH Dried Herbs	Hops	USA	131
Animal Feeds	Pea vines and hay	USA, Canada	132
	Bean forage and hay	USA, Canada	133
	Soya bean forage and hay	USA, Canada	134
	Barley hay and straw	USA, Canada	135
	Wheat forage, hay and straw	USA, Canada	136
	Field corn forage and stover	USA, Canada	137
	Sweet corn forage and stover	USA, Canada	138
	Sorghum forage and stover	USA	139
	Alfalfa forage and hay	USA	140
	Clover forage and hay	USA	141
	Almond hulls	USA	142
	Cotton gin by-products	USA	143
	Peanut hay	USA	144

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on supervised trials for the uses of flupyradifurone on citrus fruits (grapefruit, lemons, mandarins and oranges, pome fruits (apples and pears), berries and other small fruits (blueberries, grapes and strawberries), assorted tropical and sub-tropical fruit—inedible peel (prickly pear), bulb vegetables (bulb onion and green onions), Brassica vegetables (broccoli, cabbage, and cauliflower), cucurbits (cucumber, melons and summer squash), fruiting vegetables other than cucurbits (tomatoes, chilli and sweet peppers and sweet corn), leafy vegetables (mustard greens, head and leafy lettuce, spinach), legume vegetables (common bean, lima bean and peas), pulses (peas, beans and soya beans), root and tuber vegetables (carrots, potatoes and radishes), stalk and stem vegetables (celery), cereals (barley, maize, sorghum and wheat), tree nuts (almonds and pecans), oilseeds (cotton and peanuts), coffee, hops and animal feeds (alfalfa forage and hay, clover forage and hay, pea, bean and soya bean forage and hay, peanut hay, barley hay and straw, maize and sweet corn forage and stover, sorghum forage and stover, wheat forage, hay and straw, almond hull and cotton gin by-products).

Trials were well documented with laboratory and field reports. The former included method validation including recoveries with spiking at residue levels like those occurring in samples from the supervised trials. Dates of analyses or duration of sample storage were also provided. Samples were collected and stored frozen immediately or soon after sampling. Although trials included control plots, no control data are recorded in the Tables because, unless noted, residues in control samples did not exceed the LOQ. Residues are unadjusted for recoveries.

Residues from the trials conducted per maximum GAP have been used for the estimation of maximum residue levels and dietary risk assessment and are underlined. If a higher residue level was observed at a longer PHI than the GAP, the higher value has been used in MRL setting and dietary risk assessment.

For replicate samples (from the same plot), the mean value was used for maximum residue level estimation and dietary intake assessment. Where mean values have been considered, if an individual observation is higher than the highest mean, this is also listed (as the HR). Where parent or DFA residues were not detected or were less than the LOQ (*i.e.* < 0.01 mg/kg for parent or 0.05 mg/kg for DFA) the LOQ value was utilized for maximum residue estimation and dietary intake assessment. For 6-CNA, values less than the LOQ were not added for calculation of total residues of flupyradifurone.

Table 84 shows how residues in the trials were added to give total residues of flupyradifurone

Parent	DFA	6-CNA	Total
< 0.01	0.05	0.01	0.07
0.01	< 0.05	0.01	0.07
< 0.01	< 0.05	< 0.01	< 0.06
0.01	0.05	< 0.01	0.06
0.01	0.05	0.01	0.07

For multiple trials on a crop from the same location, the result from the trial yielding the highest residue was utilized for maximum residue level estimation and dietary intake assessment. In this case the trials are separated with a dotted line. Residues were added as unrounded values.

The results of these supervised trials are shown in the following tables:

Citrus Fruit

Supervised trials were carried out on mandarins (eight trials-Table 85) in the USA, during the 2011 growing season (Woodard 2012, RARVP064) to determine residues in mandarins following either two airblast spray applications, two ultra-low volume applications or one soil drench application using a flupyradifurone 200 g/L SL (soluble concentrate) formulation. Applications were made to plots using ground-based equipment. Adjuvant was used in all applications.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in mandarins were determined using LC-MS/MS method 01304. Acceptable recovery data were obtained for all analytes in mandarins.

Table 85 Residues from the foliar and soil application of flupyradifurone to mandarins in the USA (Woodard 2012, RARVP064)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Citrus Foliar	2 (10)		205			1					
GAP, USA, Citrus Soil	1		409			30					
RV221-11DA,	2	81	204	2110	Fruit	–0	0.12	< 0.05	< 0.01	< 0.01	0.17
Oviedo,	(10)	83	204	2110		0	0.14	< 0.05	< 0.01	< 0.01	0.19
Florida,						1	0.16	< 0.05	< 0.01	< 0.01	0.21

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
USA, 2011						3	0.053	< 0.05	< 0.01	< 0.01	0.10
(W. Murcotts)						10	0.070	< 0.05	< 0.01	< 0.01	0.12
						21	0.071	0.054	< 0.01	< 0.01	0.13
	2	81	204	26	Fruit	–0	0.011	< 0.05	< 0.01	< 0.01	0.061
	(10)	83	206	26		0	0.025	< 0.05	< 0.01	< 0.01	0.075
						1	0.025	< 0.05	< 0.01	< 0.01	0.075
						3	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						10	0.015	< 0.05	< 0.01	< 0.01	0.065
						21	0.017	< 0.05	< 0.01	< 0.01	0.067
	1	81	408		Fruit	30	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV222-11DA,	2	81	203	2100	Fruit	–0	0.13	< 0.05	< 0.01	< 0.01	0.18
Clermont,	(10)	83	203	2100		0	0.29	< 0.05	< 0.01	< 0.01	0.34
Florida,						1	0.35 *c 0.017*	< 0.05	< 0.01	< 0.01	0.40
USA, 2011						3	0.24	< 0.05	< 0.01	< 0.01	0.29
(Sunburst)						10	0.23	< 0.05	< 0.01	< 0.01	0.28
						20	0.20	0.069	< 0.01	< 0.01	0.27
	2	81	207	26	Fruit	–0	0.048	< 0.05	< 0.01	< 0.01	0.098
	(10)	83	194	24		0	0.056	< 0.05	< 0.01	< 0.01	0.11
						1	0.056 *c 0.017*	< 0.05	< 0.01	< 0.01	0.11
						3	0.044	< 0.05	< 0.01	< 0.01	0.094
						10	0.052	< 0.05	< 0.01	< 0.01	0.10
						20	0.053	< 0.05	< 0.01	< 0.01	0.10
	1	79	409		Fruit	30	0.012	< 0.05	< 0.01	< 0.01	0.062
						-	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV225-11DA,	2	89	206	2420	Fruit	–0	0.13	< 0.05	< 0.01	< 0.01	0.18
Haines City,	(10)	89	205	2420		0	0.21	< 0.05	< 0.01	< 0.01	0.26
Florida,						1	0.15 *c 0.023*	< 0.05	< 0.01	< 0.01	0.20
USA, 2011						3	0.13	< 0.05	< 0.01	< 0.01	0.18
(Sunburst)						10	0.14	< 0.05	< 0.01	< 0.01	0.19
						21	0.069	< 0.05	< 0.01	< 0.01	0.12
	2	89	204	26	Fruit	–0	0.47	< 0.05	< 0.01	< 0.01	0.52
	(10)	89	205	26		0	0.51	< 0.05	< 0.01	< 0.01	0.56
						1	0.37 *c 0.023*	0.067	< 0.01	< 0.01	0.44
						3	0.51	< 0.05	< 0.01	< 0.01	0.56
						10	0.49	< 0.05	< 0.01	< 0.01	0.54
						21	0.38	< 0.05	< 0.01	< 0.01	0.43
	1	81	410		Fruit	28	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV226-11DA,	2	81	213	2350	Fruit	–0	0.18	< 0.05	< 0.01	0.027	0.25
Edinburg,	(8)	83	213	2340		0	0.23	< 0.05	< 0.01	< 0.01	0.28
Texas,						1	0.24	< 0.05	0.047	< 0.01	0.29
USA, 2011						3	0.33	< 0.05	0.045	< 0.01	0.38
(Dancy)						10	0.18	0.053	< 0.01	< 0.01	0.23
						21	0.15	0.063	< 0.01	< 0.01	0.21
	2	81	210	24	Fruit	–0	0.48	< 0.05	< 0.01	< 0.01	0.53
	(8)	83	211	25		0	0.86	< 0.05	< 0.01	< 0.01	0.91
						1	0.49	< 0.05	< 0.01	< 0.01	0.54
						3	0.23	< 0.05	< 0.01	< 0.01	0.28
						10	0.90	0.094	< 0.01	< 0.01	0.99
						21	0.41	0.092	< 0.01	< 0.01	0.51
	1	81	410		Fruit	29	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV223-11DA,	2	79	205	2330	Fruit	–0	0.10	< 0.05	< 0.01	< 0.01	0.15
Orland,	(10)	83	206	2330		0	0.13	< 0.05	< 0.01	< 0.01	0.18
California,						1	0.11	< 0.05	< 0.01	< 0.01	0.16
USA, 2011						3	0.10	< 0.05	< 0.01	< 0.01	0.15
(Satsuma)						10	0.16	< 0.05	< 0.01	< 0.01	0.21
						21	0.13	< 0.05	< 0.01	< 0.01	0.18
	2	79	205	28	Fruit	–0	0.15	< 0.05	< 0.01	< 0.01	0.20
	(10)	83	205	28		0	0.065	< 0.05	< 0.01	< 0.01	0.12

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
						1	0.17	< 0.05	< 0.01	< 0.01	0.22
						3	0.16	< 0.05	< 0.01	< 0.01	0.21
						10	0.20	< 0.05	< 0.01	< 0.01	0.25
						21	0.21	< 0.05	< 0.01	< 0.01	0.26
	1	72	410		Fruit	30	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV227-11DA,	2	79	205	2330	Fruit	–0	0.18	< 0.05	< 0.01	< 0.01	0.23
Orland,	(10)	83	205	2330		0	0.25	< 0.05	< 0.01	< 0.01	0.30
California,						1	0.14	< 0.05	< 0.01	< 0.01	0.19
USA, 2011						3	0.28	< 0.05	< 0.01	< 0.01	0.33
(Satsuma)						10	0.18	< 0.05	< 0.01	< 0.01	0.23
						21	0.25	< 0.05	< 0.01	< 0.01	0.30
	2	79	205	28	Fruit	–0	0.40	< 0.05	< 0.01	< 0.01	0.45
	(10)	83	205	28		0	0.15	< 0.05	< 0.01	< 0.01	0.20
						1	0.15	< 0.05	< 0.01	< 0.01	0.20
						3	0.19	< 0.05	< 0.01	< 0.01	0.24
						10	0.27	< 0.05	< 0.01	< 0.01	0.32
						21	0.39	< 0.05	< 0.01	< 0.01	0.44
	1	72	410		Fruit	30	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV224-11DA,	2	83	196	1860	Fruit	–0	0.11	< 0.05	< 0.01	< 0.01	0.16
Shiraz Ranch,	(8)	85	205	1980		0	0.27	< 0.05	< 0.01	< 0.01	0.32
California,						1	0.33	< 0.05	< 0.01	< 0.01	0.38
USA, 2011						3	0.28	< 0.05	< 0.01	< 0.01	0.33
(Tango)						10	0.36	< 0.05	< 0.01	< 0.01	0.41
						21	0.18	< 0.05	< 0.01	< 0.01	0.23
	2	83	214	28	Fruit	–0	0.46	< 0.05	< 0.01	< 0.01	0.51
	(8)	85	206	26		0	0.29	< 0.05	< 0.01	< 0.01	0.34
						1	0.40	< 0.05	< 0.01	< 0.01	0.45
						3	0.55	< 0.05	< 0.01	< 0.01	0.60
						10	0.36	< 0.05	< 0.01	< 0.01	0.41
						21	0.25	< 0.05	< 0.01	< 0.01	0.30
	1	81	1013		Fruit	29	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV228-11DA,	2	83	205	1940	Fruit	–0	0.084	< 0.05	< 0.01	< 0.01	0.13
Shiraz Ranch,	(8)	85	206	1990		0	0.19	< 0.05	< 0.01	< 0.01	0.24
California,						1	0.19	< 0.05	< 0.01	< 0.01	0.24
USA, 2011						3	0.16	< 0.05	< 0.01	< 0.01	0.21
(Owari Satsuma)						10	0.14	< 0.05	< 0.01	< 0.01	0.19
						21	0.12	< 0.05	< 0.01	< 0.01	0.17
	2	83	212	28	Fruit	–0	0.064	< 0.05	< 0.01	< 0.01	0.11
	(8)	85	207	26		0	0.33	< 0.05	< 0.01	< 0.01	0.38
						1	0.091	< 0.05	< 0.01	< 0.01	0.14
						3	0.61	< 0.05	< 0.01	< 0.01	0.66
						10	0.080	< 0.05	< 0.01	< 0.01	0.13
						21	0.035	< 0.05	< 0.01	< 0.01	0.085
	1	81	1013		Fruit	29	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

–0 = Sampled immediately before the second application

Supervised trials were carried out on grapefruit (six trials-Table 86), lemons (eight trials-Table 87) and oranges (12 trials-Table 88) in the USA, during the 2010 and 2011 growing seasons (Beedle and Niczyporowicz 2012, RARVY012) to determine residues in citrus fruit following either two airblast applications or two ultra-low volume applications of a flupyradifurone 200 g/L SL formulation or one soil drench application of a 200 g/L SL formulation. Applications were made to plots using ground-based equipment. Adjuvant was added in all applications. Potential residue reduction was investigated in four trials (RV159-10HA orange, RV163-10HA orange, RV166-10DA lemon and RV174-10HA grapefruit).

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in grapefruit, lemons and oranges were determined using LC-MS/MS method 01304. Acceptable method and concurrent recovery data were obtained for all analytes in all fruit, peel and pulp.

Table 86 Residues from the foliar and soil application of flupyradifurone to grapefruit in the USA (Beedle and Niczyporowicz 2012, RARVY012)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Citrus Foliar	2 (10)		205			1					
GAP, USA, Citrus Soil	1		409			30					
RV172-10DA,	2	89	202	2270	Fruit	–0	0.070	< 0.020	< 0.01	< 0.01	0.090
Umatilla,	(9)	89	209	2334		0	0.11	< 0.020	< 0.01	< 0.01	0.13
Florida,						1	0.13 c 0.029	< 0.020	< 0.01	< 0.01	0.15
USA, 2011						3	0.15	< 0.020	< 0.01	< 0.01	0.17
(Flame)						8	0.13	< 0.020	< 0.01	< 0.01	0.15
						21	0.085	< 0.020	< 0.01	< 0.01	0.11
	2	89	206	23	Fruit	–0	0.069	< 0.020	< 0.01	< 0.01	0.089
	(9)	89	208	24		0	0.48	< 0.020	< 0.01	< 0.01	0.50
						1	0.19 c0.029	< 0.020	< 0.01	< 0.01	0.21
						3	0.15	< 0.020	< 0.01	< 0.01	0.17
						8	0.053	< 0.020	< 0.01	< 0.01	0.073
						21	0.046	< 0.020	< 0.01	< 0.01	0.066
	1	83	403		Fruit	30	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
							0.01	< 0.020	< 0.01	< 0.01	0.030
RV173-10HA,	2	85	204	2012	Fruit	–0	0.13	< 0.020	< 0.01	< 0.01	0.15
Clermont,	(9)	89	207	1982		1	0.19 c0.021	< 0.020	< 0.01	< 0.01	0.21
Florida,	2	85	202	24	Fruit	–0	0.085	< 0.020	< 0.01	< 0.01	0.11
USA, 2010	(9)	89	205	24		1	0.17 c0.021	< 0.020	< 0.01	< 0.01	0.19
(White)	1	81	414		Fruit	30	0.047	< 0.020	< 0.01	< 0.01	0.067
							0.029	< 0.020	< 0.01	< 0.01	0.049
RV174-10HA,	2	85	204	2569	Fruit	–0	0.11	< 0.020	< 0.01	< 0.01	0.13
Alturas,	(9)	89	205	2509		1	0.16 c0.014	< 0.020	< 0.01	< 0.01	0.18
Florida,	2	85	203	26	Fruit	–0	0.19	< 0.020	< 0.01	< 0.01	0.21
USA, 2010	(9)	89	224	29		1	0.29 c0.014	< 0.020	< 0.01	< 0.01	0.31
(White)					Fruit	1	0.347 (av.)	< 0.020 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.37 (av.)
					Peel	1	0.596 (av.)	< 0.020 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.62 (av.)
					Pulp	1	< 0.01 (av.)	< 0.020 (av.)	< 0.01 (av.)	< 0.01 (av.)	< 0.030 (av.)
	1	81	412		Fruit	30	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
							0.015	< 0.020	< 0.01	< 0.01	0.035
RV175-10HA,	2	83	209	2368	Fruit	–0	0.065	< 0.020	< 0.01	< 0.01	0.085
Raymondville,	(9)	83	206	2339		1	0.12	< 0.020	< 0.01	< 0.01	0.14

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Texas,	2	83	207	24	Fruit	–0	0.11	< 0.020	< 0.01	< 0.01	0.13
USA, 2010	(9)	83	213	24		1	0.16	< 0.020	< 0.01	< 0.01	0.18
(Rio Red)	1	81	414		Fruit	29	0.014	< 0.020	< 0.01	< 0.01	0.034
							0.014	< 0.020	< 0.01	< 0.01	0.034
RV176-10DA,	2	89	204	2321	Fruit	–0	0.12	< 0.020	< 0.01	< 0.01	0.14
Fresno,	(11)	89	205	2336		0	0.13	< 0.020	< 0.01	0.018	0.17
California,						1	0.20 c0.038 av.	< 0.020	< 0.01	0.030	0.25
USA, 2011						3	0.10	< 0.020	< 0.01	0.024	0.14
(Oro Blanco)						10	0.13	< 0.020	< 0.01	0.027	0.18
						21	0.084	< 0.020	< 0.01	0.027	0.13
	2	89	205	25	Fruit	–0	0.074	< 0.020	< 0.01	0.015	0.11
	(11)	89	204	25		0	0.21	< 0.020	< 0.01	0.015	0.24
						1	0.13 c0.038 av.	< 0.020	< 0.01	0.031	0.18
						3	0.27	< 0.020	< 0.01	0.024	0.32
						10	0.13	< 0.020	< 0.01	0.024	0.17
						21	0.15	< 0.020	< 0.01	0.021	0.19
	1	85	408		Fruit	30	0.056	< 0.020	< 0.01	0.024	0.10
							0.041	< 0.020	< 0.01	0.011	0.072
RV177-10HA,	2	81	208	2385	Fruit	–0	0.079	< 0.020	< 0.01	< 0.01	0.099
Sanger,	(11)	85	204	2068		1	0.19	< 0.020	< 0.01	< 0.01	0.21
California,	2	81	204	27	Fruit	–0	0.056	< 0.020	< 0.01	< 0.01	0.076
USA, 2010	(11)	85	203	24		1	0.062	< 0.020	< 0.01	< 0.01	0.082
(White)	1	81	410		Fruit	29	0.011	< 0.020	< 0.01	< 0.01	0.031
							< 0.01	< 0.020	< 0.01	< 0.01	< 0.030

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.02 mg/kg for DFA (parent equivs.)

–0 = Sampled immediately before the second application

Table 87 Residues from the foliar and soil application of flupyradifurone to lemons in the USA (Beedle and Niczyporowicz 2012, RARVY012)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Citrus Foliar	2 (10)		205			1					
GAP, USA, Citrus Soil	1		409			30					
RV164-10DA,	2	79	207	1968	Fruit	–0	0.27	0.063	< 0.01	0.014	0.35
Clermont,	(12)	83	206	1943		0	0.37	0.056	< 0.01	0.011	0.43
Florida,						1	0.35 c0.01	0.073	< 0.01	0.013	0.43
USA, 2010						3	0.44	0.098	< 0.01	0.013	0.55
(–)						10	0.19	0.10	< 0.01	0.011	0.31

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
						20	0.073	0.13	< 0.01	0.011	0.21
	2	79	207	25	Fruit	–0	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
	(12)	83	207	25		0	0.17	< 0.020	< 0.01	< 0.01	0.19
						1	0.21 c0.01	< 0.020	< 0.01	< 0.01	0.23
						3	0.10	< 0.020	< 0.01	< 0.01	0.12
						10	0.042	< 0.020	< 0.01	< 0.01	0.062
						20	0.018	< 0.020	< 0.01	< 0.01	0.038
	1	79	398		Fruit	31	< 0.01 c0.01	< 0.020	< 0.01	< 0.01	< 0.030
							< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
RV165-10HA,	2	83	200	2295	Fruit	–0	0.19	0.024	< 0.01	0.01 (av.)	0.21
Clermont,	(9)	83	202	2309		1	0.23	< 0.020	< 0.01	< 0.01	0.25
Florida,	2	83	206	23	Fruit	–0	0.056	< 0.020	< 0.01	< 0.01 (av.)	0.076
USA, 2010	(9)	83	207	23		1	0.052	< 0.020	< 0.01	< 0.01	0.072
(Myer)	1	83	408		Fruit	30	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
							< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
RV166-10DA,	2	81	205	2632	Fruit	–0	0.030	< 0.020	< 0.01	< 0.01	0.05
Arroyo Grande,	(10)	81	205	2701		0	0.12	< 0.020	< 0.01	< 0.01	0.14
California,						1	0.12	< 0.020	< 0.01	< 0.01	0.14
USA, 2011						3	0.12	< 0.020	< 0.01	< 0.01	0.14
(Lisbon)						9	0.12	< 0.020	< 0.01	< 0.01	0.14
						21	0.089	< 0.020	< 0.01	< 0.01	0.11
	2	81	214	28	Fruit	–0	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
	(10)	81	205	26		0	0.038	< 0.020	< 0.01	< 0.01	0.058
						1	0.054	< 0.020	< 0.01	< 0.01	0.074
						3	0.025	< 0.020	< 0.01	< 0.01	0.045
						9	0.011	< 0.020	< 0.01	< 0.01	0.031
						21	0.016	< 0.020	< 0.01	< 0.01	0.036
					Fruit	1	0.015 (av.)	< 0.020 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.035 (av.)
					Peel	1	0.042 (av.)	< 0.020 (av.)	0.011 (av.)	< 0.01 (av.)	0.062 (av.)
					Pulp	1	< 0.01 (av.)	< 0.020 (av.)	< 0.01 (av.)	< 0.01 (av.)	< 0.030 (av.)
	1	81	410		Fruit	30	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
							< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
RV167-10DA,	2	89	203	2316	Fruit	–0	0.093	< 0.020	< 0.01	< 0.01	0.11
Fresno,	(11)	89	205	2355		0	0.79	< 0.020	< 0.01	< 0.01	0.81
California,						1	0.26	< 0.020	< 0.01	< 0.01	0.28
USA, 2011						3	0.33	< 0.020	< 0.01	< 0.01	0.35
(Lisbon)						10	0.18	< 0.020	< 0.01	< 0.01	0.20
						21	0.19	0.021	< 0.01	< 0.01	0.22
	2	89	207	25	Fruit	–0	0.14	< 0.020	< 0.01	< 0.01	0.16
	(11)	89	205	25		0	0.29	< 0.020	< 0.01	< 0.01	0.31
						1	0.71	< 0.020	< 0.01	< 0.01	0.73
						3	0.44	< 0.020	< 0.01	< 0.01	0.46
						10	0.54	< 0.020	< 0.01	< 0.01	0.56
						21	0.32	0.033	< 0.01	< 0.01	0.35
	1	85	409		Fruit	30	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
							< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
RV170-10HA,	2	89	204	2323	Fruit	–0	0.17	< 0.020	< 0.01	< 0.01	0.19

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Fresno,	(11)	89	205	2355		1	0.23	< 0.020	< 0.01	< 0.01	0.25
California,	2	89	207	25	Fruit	–0	0.35	< 0.020	< 0.01	< 0.01	0.37
USA, 2011	(11)	89	205	25		1	0.67	< 0.020	< 0.01	< 0.01	0.69
(Lisbon)	1	85	409		Fruit	30	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
							< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
RV168-10DA,	2	85	205	2126	Fruit	–0	0.15	< 0.020	< 0.01	< 0.01	0.17
Sanger,	(10)	89	205	2337		0	0.26	< 0.020	< 0.01	< 0.01	0.28
California,						1	0.28	< 0.020	< 0.01	< 0.01	0.30
USA, 2011						3	0.28	< 0.020	< 0.01	< 0.01	0.30
(Lisbon)						10	0.30	< 0.020	< 0.01	< 0.01	0.32
						21	0.29	< 0.020	< 0.01	< 0.01	0.31
	2	85	207	24	Fruit	0	0.14	< 0.020	< 0.01	< 0.01	0.16
	(10)	89	204	24		1	0.088	< 0.020	< 0.01	< 0.01	0.11
						3	0.091	< 0.020	< 0.01	< 0.01	0.11
						10	0.099	< 0.020	< 0.01	< 0.01	0.12
						21	0.05	< 0.020	< 0.01	< 0.01	0.070
	1	83	410		Fruit	30	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
							< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
RV169-10HA,	2	85	212	2391	Fruit	–0	0.20	< 0.020	< 0.01	< 0.01	0.22
Somis,	(10)	89	214	2451		1	0.28	< 0.020	< 0.01	< 0.01	0.30
California,	2	85	206	27	Fruit	–0	0.19	< 0.020	< 0.01	< 0.01	0.21
USA, 2010	(10)	89	206	27		1	0.35	< 0.020	< 0.01	< 0.01	0.37
(Eureka)	1	79	410		Fruit	30	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
							< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
RV171-10HA,	2	81	204	2674	Fruit	–0	0.073	< 0.020	< 0.01	< 0.01	0.093
Arroyo Grande,	(10)	83	206	2763		1	0.18	< 0.020	< 0.01	< 0.01	0.20
California,	2	83	207	28	Fruit	–0	0.018	< 0.020	< 0.01	< 0.01	0.038
USA, 2010	(10)	83	204	28		1	0.037	< 0.020	< 0.01	< 0.01	0.057
(Eureka)	1	79	410		Fruit	30	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
							< 0.01	< 0.020	< 0.01	< 0.01	< 0.030

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.02 mg/kg for DFA (parent equivs.)

–0 = Sampled immediately before the second application

Table 88 Residues from the foliar and soil application of flupyradifurone to oranges in the USA (Beedle and Niczyporowicz 2012, RARVY012)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Citrus Foliar	2 (10)		205			1					
GAP, USA, Citrus Soil	1		409			30					
RV152-10DA,	2	83	209	1977	Fruit	–0	0.037	< 0.020	< 0.01	< 0.01	0.057
Clermont,	(8)	83	206	1929		0	0.18	< 0.020	< 0.01	< 0.01	0.20
Florida,						1	0.19	< 0.020	< 0.01	< 0.01	0.21
USA, 2010						3	0.16	< 0.020	< 0.01	< 0.01	0.18
(Hamilins)						10	0.11	< 0.020	< 0.01	< 0.01	0.13
						21	0.068	< 0.020	< 0.01	< 0.01	0.088
	2	83	209	26	Fruit	–0	0.29	< 0.020	< 0.01	< 0.01	0.31

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
	(8)	83	209	27		0	0.80	< 0.020	< 0.01	< 0.01	0.82
						1	0.78	< 0.020	< 0.01	< 0.01	0.80
						3	1.2	0.034	< 0.01	< 0.01	1.2
						10	0.53	0.032	< 0.01	< 0.01	0.56
						21	0.34	0.061	< 0.01	< 0.01	0.40
	1	79	418		Fruit	29	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
						29	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
RV153-10DA,	2	89	202	2269	Fruit	–0	0.11	< 0.020	< 0.01	< 0.01	0.13
Umatilla,	(9)	89	206	2307		0	0.20	< 0.020	< 0.01	< 0.01	0.22
Florida,						1	0.27 c0.015	< 0.020	< 0.01	< 0.01	0.29
USA, 2011						3	0.26	< 0.020	< 0.01	< 0.01	0.28
(Valencia)						8	0.24	< 0.020	< 0.01	< 0.01	0.26
						21	0.11	< 0.020	< 0.01	< 0.01	0.13
	2	89	206	23	Fruit	–0	0.11	< 0.020	< 0.01	< 0.01	0.13
	(9)	89	208	24		0	0.67	< 0.020	< 0.01	< 0.01	0.69
						1	0.31 c0.015	< 0.020	< 0.01	< 0.01	0.33
						3	0.32	< 0.020	< 0.01	< 0.01	0.34
						8	0.34	0.024	< 0.01	< 0.01	0.36
						21	0.25	< 0.020	< 0.01	< 0.01	0.27
	1	81	404		Fruit	30	0.021 c0.015	< 0.020	< 0.01	< 0.01	0.041
							0.041	< 0.020	< 0.01	< 0.01	0.061
RV154-10DA,	2	81	202	2035	Fruit	–0	0.16	< 0.020	< 0.01	< 0.01	0.18
Oviedo,	(10)	83	203	2051		0	0.26	< 0.020	< 0.01	< 0.01	0.28
Florida,						1	0.27	< 0.020	< 0.01	< 0.01	0.29
USA, 2010						3	0.26	< 0.020	< 0.01	< 0.01	0.28
(Navel)						10	0.20	< 0.020	< 0.01	< 0.01	0.22
						21	0.21	0.034	< 0.01	< 0.01	0.24
	2	81	210	26	Fruit	–0	0.26	0.23	< 0.01	< 0.01	0.28
	(10)	83	203	26		0	0.29	< 0.020	< 0.01	< 0.01	0.31
						1	0.60	0.026	< 0.01	< 0.01	0.63
						3	0.88	0.028	< 0.01	< 0.01	0.91
						10	0.17	0.041	< 0.01	< 0.01	0.21
						21	0.080	0.057	< 0.01	< 0.01	0.14
	1	81	410		Fruit	30	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
							< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
RV155-10HA,	2	89	209	2632	Fruit	–0	0.091	< 0.020	< 0.01	< 0.01	0.11
Groveland,	(10)	89	206	2487		1	0.098	< 0.020	< 0.01	< 0.01	0.12
Florida,	2	89	207	27	Fruit	–0	0.13	< 0.020	< 0.01	< 0.01	0.15
USA, 2010	(10)	89	205	26		1	0.21	< 0.020	< 0.01	< 0.01	0.23
(Navel)	1	81	409		Fruit	30	0.020	< 0.020	< 0.01	< 0.01	0.040
							0.026	< 0.020	< 0.01	< 0.01	0.046
RV156-10HA,	2	89	207	2612	Fruit	–0	0.10	< 0.020	< 0.01	< 0.01	0.12
Groveland,	(10)	89	206	2489		1	0.29	< 0.020	< 0.01	< 0.01	0.31
Florida,	2	89	206	27	Fruit	–0	0.076	< 0.020	< 0.01	< 0.01	0.096
USA, 2010	(10)	89	208	26		1	0.23	< 0.020	< 0.01	< 0.01	0.25
(Hamlin)	1	81	427		Fruit	30	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
							< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
RV157-10HA,	2	81	202	2055	Fruit	–0	0.10	< 0.020	< 0.01	< 0.01	0.12
Oak Hill,	(10)	83	203	2067		1	0.25	< 0.020	< 0.01	< 0.01	0.27
Florida,	2	81	202	25	Fruit	–0	0.24	< 0.020	< 0.01	< 0.01	0.26
USA, 2010	(10)	83	203	26		1	0.23	< 0.020	< 0.01	< 0.01	0.25
(Hamlin)	1	81	410		Fruit	30	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
							< 0.01	< 0.020	< 0.01	< 0.01	< 0.030

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RV158-10HA,	2	89	208	2321	Fruit	–0	0.071	< 0.020	< 0.01	< 0.01	0.091
Eustis,	(9)	89	209	2338		1	0.18	< 0.020	< 0.01	< 0.01	0.20
Florida,	2	89	206	23	Fruit	–0	0.25	< 0.020	< 0.01	< 0.01	0.27
USA, 2011	(9)	89	206	23		1	0.63	< 0.020	< 0.01	< 0.01	0.65
(Early Gold)	1	89	413		Fruit	30	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
							< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
RV159-10HA,	2	83	209	1905	Fruit	–0	0.12	< 0.020	< 0.01	< 0.01	0.14
Mt Dora,	(10)	83	215	1864		1	0.70 c0.016	< 0.020	< 0.01	< 0.01	0.72
Florida,	2	83	209	29	Fruit	–0	0.031	< 0.020	< 0.01	< 0.01	0.051
USA, 2010	(10)	83	207	29		1	0.23 c0.016	< 0.020	< 0.01	< 0.01	0.25
(Valencia)					Fruit	1	0.085 (av.)	< 0.020 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.11 (av.)
					Peel	1	0.346 (av.)	< 0.020 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.37 (av.)
					Pulp	1	< 0.01 (av.)	< 0.020 (av.)	< 0.01 (av.)	< 0.01 (av.)	< 0.030 (av.)
	1	79	416		Fruit	30	0.014	< 0.020	< 0.01	< 0.01	0.034
							< 0.01 c0.016	< 0.020	< 0.01	< 0.01	< 0.030
RV160-10HA,	2	83	208	2364	Fruit	–0	0.096	< 0.020	< 0.01	< 0.01	0.12
Raymondville,	(9)	83	207	2345		1	0.14	< 0.020	< 0.01	< 0.01	0.16
Texas,	2	83	206	24	Fruit	–0	0.083	< 0.020	< 0.01	< 0.01	0.10
USA, 2010	(9)	83	214	24		1	0.19	< 0.020	< 0.01	< 0.01	0.21
(N-33)	1	81	414		Fruit	30	0.029	< 0.020	< 0.01	< 0.01	0.049
							0.013	< 0.020	< 0.01	< 0.01	0.033
RV161-10DA,	2	81	209	2580	Fruit	–0	0.94	0.034	< 0.01	< 0.01	0.97
Orland,	(11)	83	209	2580		0	0.43	< 0.020	< 0.01	0.020	0.47
California,						1	0.75	< 0.020	< 0.01	0.019	0.79
USA, 2011						3	1.5	0.053	< 0.01	< 0.01	1.5
(Valencia)						10	0.41	0.041	< 0.01	0.018	0.47
						21	0.49	0.079	< 0.01	0.061	0.63
	2	81	204	27	Fruit	–0	0.30	< 0.020	< 0.01	0.014	0.34
	(11)	83	204	27		0	0.49	< 0.020	< 0.01	0.044	0.55
						1	0.58	< 0.020	< 0.01	< 0.01	0.60
						3	0.23	< 0.020	< 0.01	0.024	0.27
						10	2.1	0.097	< 0.01	0.048	2.2
						21	0.31	0.080	< 0.01	0.088	0.48
	1	81	409		Fruit	30	< 0.01	< 0.020	< 0.01	0.012	0.042
							0.015	< 0.020	< 0.01	0.073	0.11
RV162-10HA,	2	81	205	2324	Fruit	–0	0.041	< 0.020	< 0.01	< 0.01	0.061
Orland,	(7)	83	205	2325		1	0.12	< 0.020	< 0.01	< 0.01	0.14
California,	2	81	200	24	Fruit	–0	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
USA, 2010	(7)	83	205	24		1	0.069	< 0.020	< 0.01	< 0.01	0.089
(Mandarin-Satsuma)	1	79	410		Fruit	30	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
							0.012	0.027	< 0.01	< 0.01	0.039
RV163-10HA,	2	85	216	2817	Fruit	–0	0.089	< 0.020	< 0.01	< 0.01	0.11
Arroya Grande,	(10)	89	205	2694		1	0.067	< 0.020	< 0.01	< 0.01	0.087
California,	2	85	175	25	Fruit	–0	0.042	< 0.020	< 0.01	< 0.01	0.062
USA, 2010	(10)	89	211	21		1	0.020	< 0.020	< 0.01	< 0.01	0.040
(Olinda Valencia)					Fruit	1	0.048 (av.)	< 0.020 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.068 (av.)
					Peel	1	0.115 (av.)	< 0.020 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.14 (av.)
					Pulp	1	< 0.01 (av.)	< 0.020 (av.)	< 0.01 (av.)	< 0.01 (av.)	< 0.030 (av.)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
	1	83	410		Fruit	29	< 0.01	< 0.020	< 0.01	< 0.01	< 0.030
							< 0.01	< 0.020	< 0.01	< 0.01	< 0.030

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.02 mg/kg for DFA (parent equivs.)

–0 = Sampled immediately before the second application

Pome fruits

Supervised trials were carried out on apples (14 trials—Table 89) and pears (nine trials—Table 90) in the USA and, in one pear trial, Canada, during the 2011 growing season (Lam 2012, RARVY013). Two foliar applications of a 200 g/L SL formulation were made. Applications were made to plots using ground-based equipment with either concentrated or dilute sprays. Adjuvant [either non-ionic surfactant at 0.20% v/v, methylated seed oil at 0.25% v/v or crop oil concentrate at 1.0% v/v] was added in all applications.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in apples and pears were determined using LC-MS/MS method 01304. Acceptable method and concurrent recovery data were obtained for all analytes in apples and pears.

Table 89 Residues from the foliar application of flupyradifurone to apples in the USA (Lam 2012, RARVY013)

Trial No., Location, Year, (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Pome fruits	2 (10)		205			14					
RV050-11HA,	2	76	206	375	Fruit	–0	0.094	< 0.05	< 0.01	< 0.01	0.14
Alton, New York,	(10)	78	208	378		14	0.25	< 0.05	< 0.01	< 0.01	0.30
USA, 2011	2	76	205	1496	Fruit	–0	0.062	< 0.05	< 0.01	< 0.01	0.11
(Jonagold)	(10)	78	206	1496		14	0.11	< 0.05	< 0.01	< 0.01	0.16
RV051-11DA,	2	85	204	439	Fruit	–0	0.072	< 0.05	< 0.01	< 0.01	0.12
Hereford,	(11)	87	203	438		0	0.34	< 0.05	< 0.01	< 0.01	0.39
Pennsylvania,						5	0.21	< 0.05	< 0.01	< 0.01	0.26
USA, 2011						13	0.11	< 0.05	< 0.01	< 0.01	0.16
(Rome Law						21	0.13	< 0.05	< 0.01	< 0.01	0.18
Strain)						26	0.11	< 0.05	< 0.01	< 0.01	0.16
						33	0.10	< 0.05	< 0.01	< 0.01	0.15
	2	85	213	1543	Fruit	–0	0.081	< 0.05	< 0.01	< 0.01	0.13
	(11)	87	212	1533		0	0.29	< 0.05	< 0.01	< 0.01	0.34
						5	0.17	< 0.05	< 0.01	< 0.01	0.22
						13	0.11	< 0.05	< 0.01	< 0.01	0.16
						21	0.11	< 0.05	< 0.01	< 0.01	0.16
						26	0.081	< 0.05	< 0.01	< 0.01	0.13
						33	0.080	< 0.05	< 0.01	< 0.01	0.13
RV052-11HA,	2	81	205	367	Fruit	–0	0.041	< 0.05	< 0.01	< 0.01	0.091
Blairsville, Georgia,	(10)	87	205	427		14	0.05	< 0.05	< 0.01	< 0.01	0.10
USA, 2011	2	81	205	1627	Fruit	–0	0.05	< 0.05	< 0.01	< 0.01	0.10
(Rome Beauty)	(10)	87	205	1599		14	0.084	< 0.05	< 0.01	< 0.01	0.13
RV053-11HA,	2	81	205	440	Fruit	–0	0.018	< 0.05	< 0.01	< 0.01	0.068
Deerfield,	(10)	85	203	436		14	0.016	< 0.05	< 0.01	< 0.01	0.066

Trial No., Location,	Application				Sample	DALA	Residues as parent (mg/kg)				
Year, (Variety)	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Michigan,											
USA, 2011	2	81	204	1748	Fruit	–0	0.056	< 0.05	< 0.01	< 0.01	0.11
(Red Delicious)	(10)	85	203	1767		14	0.060	0.05	< 0.01	< 0.01	0.11
RV054-11DA,	2	85	206	237	Fruit	–0	0.38	< 0.05	< 0.01	< 0.01	0.43
Centralia, Illinois,	(10)	87	205	254		0	0.45	< 0.05	< 0.01	< 0.01	0.50
USA, 2011						7	0.32	< 0.05	< 0.01	< 0.01	0.37
(Golden						14	0.30	0.088	< 0.01	< 0.01	0.38
Delicious)						21	0.25	0.16	< 0.01	< 0.01	0.41
						28	0.19	0.16	< 0.01	< 0.01	0.35
						35	0.24	0.27	< 0.01	< 0.01	0.51
	2	85	205	1636	Fruit	–0	0.18	< 0.05	< 0.01	< 0.01	0.23
	(10)	87	205	1767		0	0.40	< 0.05	< 0.01	< 0.01	0.45
						7	0.37	0.090	< 0.01	< 0.01	0.46
						14	0.21	0.15	< 0.01	< 0.01	0.36
						21	0.18	0.19	< 0.01	< 0.01	0.37
						28	0.26	0.36	< 0.01	< 0.01	0.62
						35	0.20	0.30	< 0.01	< 0.01	0.49
RV055-11HA,	2	78	202	481	Fruit	–0	0.088	< 0.05	< 0.01	< 0.01	0.14
Perry, Utah,	(10)	81	204	434		14	0.077	< 0.05	< 0.01	< 0.01	0.13
USA, 2011	2	78	201	1926	Fruit	–0	0.079	< 0.05	< 0.01	< 0.01	0.13
(Gala)	(10)	81	202	1935		14	0.12	< 0.05	< 0.01	< 0.01	0.17
RV056-11HA,	2	77	203	468	Fruit	–0	0.20	0.055	< 0.01	< 0.01	0.26
Los Molinios, California,	(10)	79	205	462		14	0.068	< 0.05	< 0.01	< 0.01	0.12
USA, 2011	2	77	200	2094	Fruit	–0	0.19	0.05	< 0.01	< 0.01	0.24
(Summerfield)	(10)	79	210	2085		14	0.21	0.079	< 0.01	< 0.01	0.28
RV057-11HA,	2	79	211	428	Fruit	–0	0.075	< 0.05	< 0.01	< 0.01	0.13
Weiser, Idaho,	(10)	81	209	424		12	0.19	< 0.05	< 0.01	< 0.01	0.24
USA, 2011	2	79	207	1561	Fruit	–0	0.13	< 0.05	< 0.01	< 0.01	0.18
(Early Spur Rome)	(10)	81	207	1571		12	0.22	< 0.05	< 0.01	< 0.01	0.27
RV059-11DA,	2	79	203	388	Fruit	–0	0.072	< 0.05	< 0.01	< 0.01	0.12
Caldwell, Idaho,	(10)	81	203	388		0	0.20	< 0.05	< 0.01	< 0.01	0.25
USA, 2011						7	0.15	< 0.05	< 0.01	< 0.01	0.20
(Jonathan)						14	0.18	< 0.05	< 0.01	< 0.01	0.23
						21	0.082	< 0.05	< 0.01	< 0.01	0.13
						28	0.13	0.057	< 0.01	< 0.01	0.19
						35	0.070	0.069	< 0.01	< 0.01	0.14
	2	79	204	1879	Fruit	–0	0.085	< 0.05	< 0.01	< 0.01	0.14
	(10)	81	206	1842		0	0.092	< 0.05	< 0.01	< 0.01	0.14
						7	0.11	< 0.05	< 0.01	< 0.01	0.16
						14	0.12	< 0.05	< 0.01	< 0.01	0.17
						21	0.087	0.05	< 0.01	< 0.01	0.14
						28	0.069	0.052	< 0.01	< 0.01	0.12
						35	0.063	0.056	< 0.01	< 0.01	0.12
RV060-11HA,	2	81	207	470	Fruit	–0	0.066	< 0.05	< 0.01	< 0.01	0.12
North Rose, New York,	(10)	85	209	474		14	0.059	< 0.05	< 0.01	< 0.01	0.11
USA, 2011	2	81	207	2113	Fruit	–0	0.067	< 0.05	< 0.01	< 0.01	0.12
(Greening Perennial)	(10)	85	206	2104		14	0.097	< 0.05	< 0.01	< 0.01	0.15
RV061-11HA,	2	78	205	421	Fruit	–0	0.14	< 0.05	< 0.01	< 0.01	0.19
Conklin, Michigan,	(10)	81	206	427		14	0.22	0.064	< 0.01	< 0.01	0.28
USA, 2011	2	78	203	1673	Fruit	–0	0.11	< 0.05	< 0.01	< 0.01	0.16
(Golden	(10)	81	204	1711		14	0.15	0.060	< 0.01	< 0.01	0.21

Trial No., Location,	Application				Sample	DALA	Residues as parent (mg/kg)				
Year, (Variety)	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Delicious)											
RV062-11DA,	2	77	206	323	Fruit	–0	0.086	< 0.05	< 0.01	< 0.01	0.14
Hart, Michigan,	(10)	81	206	341		0	0.34	< 0.05	< 0.01	< 0.01	0.39
USA, 2011						7	0.15	< 0.05	< 0.01	< 0.01	0.20
(Yellow						14	0.15	< 0.05	< 0.01	< 0.01	0.20
Delicious)						21	0.11	< 0.05	< 0.01	< 0.01	0.16
						28	0.15	0.097	< 0.01	< 0.01	0.25
						35	0.13	0.10	< 0.01	< 0.01	0.23
	2	77	206	1851	Fruit	–0	0.11	< 0.05	0.01	< 0.01	0.16
	(10)	81	206	1888		0	0.25	< 0.05	< 0.01	< 0.01	0.30
						7	0.13	< 0.05	< 0.01	< 0.01	0.18
						14	0.11	< 0.05	< 0.01	< 0.01	0.16
						21	0.13	< 0.05	< 0.01	< 0.01	0.18
						28	0.090	0.056	< 0.01	< 0.01	0.15
						35	0.077	0.064	< 0.01	< 0.01	0.14
RV058-11HA,	2	78	202	290	Fruit	–0	0.042	< 0.05	< 0.01	< 0.01	0.092
Hood River, Oregon,	(10)	81	204	291		13	0.060	< 0.05	< 0.01	< 0.01	0.11
USA, 2011	2	78	204	1636	Fruit	–0	0.049	< 0.05	< 0.01	< 0.01	0.099
(Honey Crisp)	(10)	81	205	1580		13	0.094	< 0.05	< 0.01	< 0.01	0.14
RV063-11HA,	2	81	205	314	Fruit	–0	0.072	< 0.05	< 0.01	< 0.01	0.12
Hood River, Oregon,	(10)	85	207	369		14	0.10	< 0.05	< 0.01	< 0.01	0.15
USA, 2011	2	81	207	1580	Fruit	–0	0.05	< 0.05	< 0.01	< 0.01	0.10
(Jonagold)	(10)	85	204	1589		14	0.14	< 0.05	< 0.01	< 0.01	0.19

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

–0 = Sampled immediately before the second application

Table 90 Residues from the foliar application of flupyradifurone to pears in the USA and Canada (Lam 2012, RARVY013)

Trial No., Location,	Application				Sample	DALA	Residues as parent (mg/kg)				
Year (Variety)	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Pome fruits	2 (10)		205			14					
RV064-11DA,	2	75	206	374	Fruit	0	0.39	0.10	< 0.01	< 0.01	0.49
	(10)	76	205	373		7	0.34	0.16	< 0.01	< 0.01	0.50
Alton,						14	0.22	0.15	< 0.01	< 0.01	0.37
New York,						21	0.17	0.16	< 0.01	< 0.01	0.33
USA, 2011						28	0.17	0.20	< 0.01	< 0.01	0.37
(Bartlett)						35	0.10	0.18	< 0.01	< 0.01	0.28
	2	75	205	1496	Fruit	0	0.45	0.18	< 0.01	< 0.01	0.62
	(10)	76	212	1543		7	0.34	0.24	< 0.01	< 0.01	0.57
						14	0.20	0.29	< 0.01	< 0.01	0.49
						21	0.17	0.25	< 0.01	< 0.01	0.43
						28	0.16	0.23	< 0.01	< 0.01	0.39
						35	0.16	0.27	< 0.01	< 0.01	0.43
RV065-11HA,	2	77	205	435	Fruit	14	0.21	0.23	< 0.01	< 0.01	0.44
Conklin,	(10)	78	205	429							

Trial No., Location,	Application				Sam ple	DA LA	Residues as parent (mg/kg)				
Year (Variety)	No. (RTI, days)	Growt h Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Michigan,											
USA, 2011	2	77	205	1739	Frui t	14	0.14	0.15	< 0.01	< 0.01	0.29
(Bartlett)	(10)	78	206	1739							
RV066-11HA,	2	75	205	449	Frui t	13	0.059	< 0.05	< 0.01	< 0.01	0.11
Marysville, California,	(11)	76	206	452							
USA, 2011	2	75	204	2197	Frui t	13	0.20	0.097	< 0.01	< 0.01	0.29
(Barlett)	(11)	76	205	2216							
RV067-11DA,	2	85	204	389	Frui t	0	0.19	< 0.05	< 0.01	< 0.01	0.24
Parlier,	(10)	85	205	379		7	0.21	< 0.05	< 0.01	< 0.01	0.26
California,						14	0.17	< 0.05	< 0.01	< 0.01	0.22
USA, 2011						21	0.14	0.070	< 0.01	< 0.01	0.21
(Shinko)						28	0.069	0.081	< 0.01	< 0.01	0.15
						35	0.055	0.093	< 0.01	< 0.01	0.15
	2	85	212	1515	Frui t	0	0.28	< 0.05	< 0.01	< 0.01	0.33
	(10)	85	204	1505		7	0.24	< 0.05	< 0.01	< 0.01	0.29
						14	0.18	< 0.05	< 0.01	< 0.01	0.23
						21	0.17	< 0.05	< 0.01	< 0.01	0.22
						28	0.13	0.066	< 0.01	< 0.01	0.20
						35	0.12	0.087	< 0.01	< 0.01	0.21
RV068-11HA,	2	81	207	376	Frui t	14	0.26	0.16	< 0.01	0.013	0.43
Ephrata, Washington,	(10)	85	206	374							
USA, 2011	2	81	209	1879	Frui t	14	0.23	0.21	< 0.01	0.011	0.45
(Concorde)	(10)	85	208	1870							
RV069-11DA,	2	78	205	455	Frui t	0	0.25	< 0.05	< 0.01	< 0.01	0.30
Caldwell,	(10)	79	210	423		7	0.20	< 0.05	< 0.01	< 0.01	0.25
Idaho,						14	0.14	< 0.05	< 0.01	< 0.01	0.19
USA, 2011						21	0.19	0.088	< 0.01	0.01	0.29
(Bartlett)						28	0.18	0.11	< 0.01	< 0.01	0.28
						35	0.18	0.14	< 0.01	< 0.01	0.32
	2	78	199	1888	Frui t	0	0.30	< 0.05	< 0.01	< 0.01	0.35
	(10)	79	204	1804		7	0.17	< 0.05	< 0.01	< 0.01	0.22
						14	0.16	0.056	< 0.01	< 0.01	0.21
						21	0.13	0.072	< 0.01	< 0.01	0.20
						28	0.11	0.081	< 0.01	< 0.01	0.19
						35	0.13	0.083	< 0.01	< 0.01	0.21
RV070-11HA,	2	78	205	363	Frui t	14	0.32	0.26	< 0.01	0.013	0.59
Shelby, Michigan,	(10)	81	205	352							
USA, 2011	2	78	206	2019	Frui t	14	0.16	0.19	< 0.01	< 0.01	0.35
(Bartlett)	(10)	81	206	1776							
RV071-11DA,	2	75	205	436	Frui t	0	0.65	0.068	< 0.01	< 0.01	0.72
Grand Rapids,	(10)	77	205	443		7	0.51	0.11	< 0.01	< 0.01	0.61
Michigan,						14	0.47	0.17	< 0.01	< 0.01	0.63

Trial No., Location, Year (Variety)	Application No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)	Sample	DALA	Residues as parent (mg/kg) Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
USA, 2011						21	0.39	0.21	< 0.01	0.011	0.60
(Bartlett)						28	0.26	0.27	< 0.01	< 0.01	0.53
						35	0.28	0.33	< 0.01	0.012	0.61
	2	75	206	1739	Fruit	0	0.36	< 0.05	< 0.01	< 0.01	0.41
	(10)	77	204	1776		7	0.31	0.086	< 0.01	< 0.01	0.40
						14	0.21	0.10	< 0.01	< 0.01	0.31
						21	0.17	0.16	< 0.01	< 0.01	0.33
						28	0.14	0.15	< 0.01	< 0.01	0.29
						35	0.11	0.14	< 0.01	< 0.01	0.25
RV072-11HA,	2	79	214	415	Fruit	14	0.39	0.30	< 0.01	< 0.01	0.69
Okanagan Falls, British Columbia,	(10)	85	216	404							
Canada, 2011	2	79	209	1515	Fruit	14	0.17	0.14	< 0.01	< 0.01	0.31
(Anju)	(10)	85	213	1533							

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Berries and other small fruits

Supervised trials were carried out on blueberries (26 trials—Table 91) in the USA (nine trials), Canada (4), Australia (3), Chile (3), New Zealand (2), United Kingdom (2) and one each in Italy, Spain and Denmark during the 2011 growing season (Dorschner 2012b, IR-4 PR No. 10637). Low bush blueberries were grown in four trials (ME01, NS01, NS02 and NS03), rabbit eye blueberries in one trial (AU04) and high bush blueberries in the 21 other trials. Two high bush blueberry trials (UK01 and SP01) were grown under plastic covered tunnels. Two foliar applications of a 200 g/L SL formulation were made with ground equipment. Non-ionic surfactant, crop oil concentrate or another adjuvant was included in the tank mix for all applications except in NZ02 and SP01.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in blueberries were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data were obtained for all analytes.

Table 91 Residues from the foliar application of flupyradifurone to blueberries in North America, South America, Australasia and Europe (Dorschner 2012b, IR-4 PR No. 10637)

Trial No., Location, Year (Variety)	Application No. (RTI, days)	Rate (g ai/ha)	Volume (L/ha)	Sample	DALA	Residues as parent (mg/kg) Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Bushberries	2 (7)	205			3					
DK01,	2	138	202	Fruit	–4	0.54	< 0.05	< 0.01	< 0.01	0.59
Sonder Omme,	(7)	192	282			0.65	< 0.05	< 0.01	< 0.01	0.70
Denmark, 2011					3	1.0	< 0.05	< 0.01	< 0.01	1.05
(Herbert high bush)						0.96	< 0.05	< 0.01	< 0.01	1.0
UK01,	2	205	700	Fruit	–4	0.47	< 0.05	< 0.01	< 0.01	0.52
Peterborough,	(7)	205	702			0.50	< 0.05	< 0.01	< 0.01	0.55
United Kingdom,					0	0.99	< 0.05	< 0.01	< 0.01	1.0

Trial No.,	Application			Sample	DALA	Residues as parent (mg/kg)				
Location,	No.	Rate	Volume			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Year	(RTI,	(g ai/ha)	(L/ha)							
(Variety)	days)									
2011						1.1	< 0.05	< 0.01	< 0.01	1.1
(Duke high bush)					1	0.84	< 0.05	< 0.01	< 0.01	0.89
(Protective						0.69	< 0.05	< 0.01	< 0.01	0.74
tunnel)					3	0.58	< 0.05	< 0.01	< 0.01	0.63
						0.55	< 0.05	< 0.01	< 0.01	0.60
						0.56 (mean)				0.61 (mean)
					8	0.48	< 0.05	< 0.01	< 0.01	0.53
						0.49	< 0.05	< 0.01	< 0.01	0.54
					15	0.35	< 0.05	< 0.01	0.011	0.41
						0.40	< 0.05	< 0.01	0.01	0.46
UK02,	2	204	697	Fruit	–3	0.44	< 0.05	< 0.01	< 0.01	0.49
Peterborough,	(6)	205	703			0.39	< 0.05	< 0.01	< 0.01	0.44
United Kingdom,					0	0.66	< 0.05	< 0.01	< 0.01	0.71
2011						0.58	< 0.05	< 0.01	< 0.01	0.63
(Bluecrop high					1	1.4	< 0.05	< 0.01	< 0.01	1.5
bush)						1.4	< 0.05	< 0.01	< 0.01	1.4
					3	0.64	< 0.05	< 0.01	< 0.01	0.69
						0.70	< 0.05	< 0.01	< 0.01	0.75
						0.67 (mean)				0.72 (mean)
					8	0.42	< 0.05	< 0.01	< 0.01	0.47
						0.48	< 0.05	< 0.01	< 0.01	0.53
					15	0.32	< 0.05	< 0.01	< 0.01	0.37
						0.30	< 0.05	< 0.01	< 0.01	0.35
IT01,	2	211	514	Fruit	–4	0.80	< 0.05	< 0.01	< 0.01	0.85
Mondovi,	(7)	205	500			0.77	< 0.05	< 0.01	< 0.01	0.82
Italy, 2011					0	3.6	< 0.05	< 0.01	< 0.01	3.6
(Duke high bush)						3.9	< 0.05	< 0.01	< 0.01	4.0
					1	3.4	< 0.05	< 0.01	< 0.01	3.5
						3.5	< 0.05	< 0.01	< 0.01	3.6
					3	1.6	< 0.05	< 0.01	< 0.01	1.6
						1.7	< 0.05	< 0.01	< 0.01	1.7
					7	1.7	< 0.05	< 0.01	0.011	1.7
						1.5	< 0.05	0.011	0.012	1.6
					14	1.9	0.075	< 0.01	0.015	1.9
						1.6	0.074	< 0.01	0.013	1.7
						1.7 (mean)				1.8 (mean)
SP01,	2	206	806	Fruit	–5	0.31	< 0.05	< 0.01	< 0.01	0.36
Lucena del Puetro,	(8)	202	788			0.31	< 0.05	< 0.01	< 0.01	0.36
Spain, 2011					0	0.48	< 0.05	< 0.01	0.027	0.55
(Jewel high bush)						0.59	< 0.05	< 0.01	0.020	0.66
(Protective					1	0.24	< 0.05	< 0.01	0.014	0.30
tunnel)						0.26	< 0.05	< 0.01	0.018	0.33
					3	0.14	< 0.05	< 0.01	0.022	0.21
						0.13	< 0.05	< 0.01	0.020	0.20
					7	0.15	0.094	< 0.01	0.066	0.31
						0.24	< 0.05	< 0.01	0.051	0.35
						0.20 (mean)				0.33 (mean)
					14	0.15	0.075	< 0.01	0.067	0.30

Trial No.,	Application			Sample	DALA	Residues as parent (mg/kg)				
Location,	No.	Rate	Volume			Parent	DFA	DFEAF	6-CNA	Parent +
Year	(RTI,	(g ai/ha)	(L/ha)							DFA + 6-CNA
(Variety)	days)									
						0.17	0.095	< 0.01	0.076	0.34
NS01,	2	207	350	Fruit	–4	0.14	< 0.05	< 0.01	< 0.01	0.19
Sheffield Mills,	(7)	208	352			0.16	< 0.05	< 0.01	< 0.01	0.21
Nova Scotia					0	0.32	< 0.05	< 0.01	< 0.01	0.37
Canada, 2011						0.86	< 0.05	< 0.01	< 0.01	0.91
(Wild clones low					1	0.61	< 0.05	< 0.01	< 0.01	0.66
bush)						0.25	< 0.05	< 0.01	< 0.01	0.30
					3	0.31	< 0.05	< 0.01	< 0.01	0.36
						0.39	< 0.05	< 0.01	< 0.01	0.44
						0.35 (mean)				0.40 (mean)
					7	0.29	< 0.05	< 0.01	< 0.01	0.34
						0.30	< 0.05	< 0.01	< 0.01	0.35
					14	0.25	< 0.05	< 0.01	< 0.01	0.30
						0.32	< 0.05	< 0.01	< 0.01	0.37
NS02,	2	209	354	Fruit	–4	0.36	< 0.05	< 0.01	< 0.01	0.41
Rawdon Gold Mines,	(7)	210	355			0.46	< 0.05	< 0.01	< 0.01	0.51
Nova Scotia					3	0.89	< 0.05	< 0.01	< 0.01	0.94
Canada, 2011						0.77	< 0.05	< 0.01	< 0.01	0.82
(Wild clones low bush)						0.83 (mean)				0.88 (mean)
NS03,	2	205	355	Fruit	–4	0.52	< 0.05	< 0.01	< 0.01	0.57
Rawdon,	(7)	206	349			0.50	< 0.05	< 0.01	< 0.01	0.55
Nova Scotia					3	1.6	< 0.05	< 0.01	< 0.01	1.7
Canada, 2011						1.5	< 0.05	< 0.01	< 0.01	1.6
(Wild clones low bush)						1.6 (mean)				1.6 (mean)
QC16,	2	212	516	Fruit	–4	0.47	< 0.05	< 0.01	< 0.01	0.52
Freligshsburg,	(7)	210	512			0.43	< 0.05	< 0.01	< 0.01	0.48
Quebec					3	0.37	< 0.05	< 0.01	< 0.01	0.42
Canada, 2011						0.52	< 0.05	< 0.01	< 0.01	0.57
(Bluecrop low bush)						0.45 (mean)				0.50 (mean)
ME01,	2	202	231	Fruit	–3	0.76	< 0.05	< 0.01	< 0.01	0.81
Jonesboro,	(6)	199	227			1.14	< 0.05	< 0.01	< 0.01	1.19
Maine					2	2.5	0.11	< 0.01	< 0.01	2.6
USA, 2011						2.5	0.11	< 0.01	< 0.01	2.6
(Low bush)						2.5 (mean)				2.6 (mean)
MI01,	2	208	749	Fruit	–4	0.17	< 0.05	< 0.01	< 0.01	0.22
Fennville,	(7)	206	734			0.22	< 0.05	< 0.01	< 0.01	0.27
Michigan					0	0.94	< 0.05	< 0.01	< 0.01	0.99
USA, 2011						0.53	< 0.05	< 0.01	< 0.01	0.58
(Jersey high bush)					1	0.47	< 0.05	< 0.01	< 0.01	0.52
						0.56	< 0.05	< 0.01	< 0.01	0.61
					3	0.41	< 0.05	< 0.01	< 0.01	0.46
						0.37	< 0.05	< 0.01	< 0.01	0.42
						0.39 (mean)				0.44 (mean)
					7	0.33	< 0.05	< 0.01	< 0.01	0.38
						0.28	< 0.05	< 0.01	< 0.01	0.33
					14	0.23	0.052	< 0.01	< 0.01	0.28
						0.24	0.059	< 0.01	< 0.01	0.30
MI02,	2	205	310	Fruit	–4	0.10	< 0.05	< 0.01	< 0.01	0.15
Fennville,	(7)	206	293			0.060	< 0.05	< 0.01	< 0.01	0.11

Trial No.,	Application			Sample	DALA	Residues as parent (mg/kg)				
Location,	No.	Rate	Volume			Parent	DFA	DFEAF	6-CNA	Parent +
Year	(RTI,	(g ai/ha)	(L/ha)							DFA + 6-CNA
(Variety)	days)									
Michigan					3	0.47	< 0.05	< 0.01	< 0.01	0.52
USA, 2011						0.37	< 0.05	< 0.01	< 0.01	0.42
(Jersey high bush)						0.42 (mean)				0.47 (mean)
MI03,	2	207	524	Fruit	–4	0.17	< 0.05	< 0.01	< 0.01	0.22
Fennville,	(7)	207	523			0.16	< 0.05	< 0.01	< 0.01	0.21
USA, 2011					3	0.22	< 0.05	< 0.01	< 0.01	0.27
(Jersey high bush)						0.24	< 0.05	< 0.01	< 0.01	0.29
						0.23 (mean)				0.28 (mean)
NC01,	2	202	300	Fruit	–3	0.38	< 0.05	< 0.01	< 0.01	0.43
Castle Hayne,	(6)	201	298			0.36	< 0.05	< 0.01	< 0.01	0.41
North Carolina					3	0.75	< 0.05	< 0.01	< 0.01	0.80
USA, 2011						0.81	< 0.05	< 0.01	< 0.01	0.86
(Crouton high bush)						0.78 (mean)				0.83 (mean)
NC02,	2	201	449	Fruit	–3	0.58	< 0.05	< 0.01	< 0.01	0.63
Castle Hayne,	(6)	202	451			0.52	< 0.05	< 0.01	< 0.01	0.57
North Carolina					3	0.72	< 0.05	< 0.01	< 0.01	0.77
USA, 2011						0.83	< 0.05	< 0.01	< 0.01	0.88
(Duplin high bush)						0.77 (mean)				0.82 (mean)
NJ01,	2	210	320	Fruit	–4	1.1	< 0.05	< 0.01	< 0.01	1.2
Cream Ridge,	(7)	212	324			1.0	< 0.05	< 0.01	< 0.01	1.1
New Jersey					3	0.65	< 0.05	< 0.01	< 0.01	0.70
USA, 2011						1.4	< 0.05	< 0.01	< 0.01	1.4
(Duke high bush)						1.0 (mean)				1.1 (mean)
NJ02,	2	211	292	Fruit	–4	0.71	< 0.05	< 0.01	< 0.01	0.76
Cream Ridge,	(7)	213	314			0.74	< 0.05	< 0.01	< 0.01	0.79
New Jersey					3	1.0	< 0.05	< 0.01	< 0.01	1.1
USA, 2011						1.3	< 0.05	< 0.01	< 0.01	1.4
(Bluecrop high bush)						1.2 (mean)				1.2 (mean)
OR01,	2	205	467	Fruit	–4	0.26	< 0.05	< 0.01	< 0.01	0.31
Aurora,	(7)	215	490			0.31	< 0.05	< 0.01	< 0.01	0.36
Oregon					3	0.51	< 0.05	< 0.01	< 0.01	0.56
USA, 2011						0.62	< 0.05	< 0.01	< 0.01	0.67
(Bluecrop high bush)						0.57 (mean)				0.62 (mean)
CL01,	2	208	352	Fruit	–4	0.89	< 0.05	< 0.01	< 0.01	0.94
Perquenco,						1.1	< 0.05	< 0.01	< 0.01	1.2
Chile, 2011	(7)	208	351		0	2.4	< 0.05	< 0.01	< 0.01	2.4
(Elliot high bush)						1.6	< 0.05	< 0.01	< 0.01	1.6
					1	1.5	< 0.05	< 0.01	< 0.01	1.6
						2.1	< 0.05	< 0.01	< 0.01	2.1
					3	1.1 c 0.016	< 0.05	< 0.01	< 0.01	1.2
						1.2 c 0.016	< 0.05	< 0.01	0.012	1.3
					7	1.6	0.051	< 0.01	0.015	1.7
						1.5	0.063	< 0.01	0.020	1.5
						1.5 (mean)				1.6 (mean)
					14	1.1	0.14	< 0.01	0.025	1.3

Trial No., Location, Year (Variety)	Application			Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
						1.1	0.098	< 0.01	0.016	1.2
CL02,	2	205	494	Fruit	–4	0.69	< 0.05	< 0.01	< 0.01	0.74
Perquenco,	(7)	209	504			0.66	< 0.05	< 0.01	< 0.01	0.71
Chile, 2011					3	1.1	< 0.05	< 0.01	< 0.01	1.2
(Elliot high bush)						1.3	< 0.05	< 0.01	< 0.01	1.4
						1.2 (mean)				1.3 (mean)
CL03,	2	204	602	Fruit	–4	0.84	< 0.05	< 0.01	< 0.01	0.89
Perquenco,	(7)	202	595			0.75	< 0.05	< 0.01	< 0.01	0.80
Chile, 2011					3	1.3	< 0.05	< 0.01	< 0.01	1.4
(Elliot high bush)						2.1	< 0.05	< 0.01	< 0.01	2.2
						1.7 (mean)				1.8 (mean)
AU01,	2	209	306	Fruit	–5	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Hoddles Creek,	(8)	207	304			1.4	< 0.05	< 0.01	< 0.01	1.5
Australia, 2011					3	2.5	0.06	0.011	0.013	2.6
(Reka high bush)						2.1	0.058	< 0.01	0.012	2.2
						2.3 (mean)				2.4 (mean)
AU02,	2	207	457	Fruit	–4	0.45	< 0.05	< 0.01	< 0.01	0.50
Hoddles Creek,	(7)	198	436			0.39	< 0.05	< 0.01	< 0.01	0.44
Australia, 2011					3	1.02	< 0.05	< 0.01	< 0.01	1.1
(Deasy high bush)						0.87	< 0.05	< 0.01	< 0.01	0.92
						0.95 (mean)				1.0 (mean)
AU04,	2	201	885	Fruit	–5	0.089	< 0.05	< 0.01	< 0.01	0.14
Corindi,	(8)	203	892			0.088	< 0.05	< 0.01	< 0.01	0.14
Australia, 2011					3	0.25	< 0.05	< 0.01	< 0.01	0.30
(Rahi rabbit eye)						0.28	< 0.05	< 0.01	< 0.01	0.33
						0.27 (mean)				0.32 (mean)
NZ02,	2	241	733	Fruit	–4	0.58	< 0.05	< 0.01	< 0.01	0.63
Hornby,	(7)	214	731			0.60	< 0.05	< 0.01	< 0.01	0.65
New Zealand,					3	0.89	< 0.05	< 0.01	< 0.01	0.94
2011						0.91	< 0.05	< 0.01	< 0.01	0.96
(Darrow high bush)						0.90 (mean)				0.95 (mean)
NZ01,	2	202	694	Fruit	–4	0.12	< 0.05	< 0.01	< 0.01	0.17
Flaxmere,	(7)	204	700			0.10	< 0.05	< 0.01	< 0.01	0.15
New Zealand,					3	0.13	< 0.05	< 0.01	0.019	0.20
2011						0.13	< 0.05	< 0.01	< 0.01	0.18
(Maru high bush)						0.13 (mean)				0.19 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Supervised trials were carried out on grapes (16 trials—Table 92) in the USA and Canada during the 2010 growing season (Dallstream 2012a, RARVY007). Two foliar applications or one soil drench application were made using a 200 g/L SL formulation. Applications were made to plots using ground-based equipment. Adjuvant at 0.25% v/v was used in all applications.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in grapes were determined using LC-MS/MS method 01304. Acceptable recovery data were obtained for all analytes in grapes.

Table 92 Residues from the foliar and soil application of flupyradifurone to grapes in the USA and Canada (Dallstream 2012a, RARVY007)

Trial No., Location,	Application				Sample	DALA	Residues as parent (mg/kg)				
Year (Variety)	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Grapes Foliar	2 (10)		205			0					
GAP, USA, Grapes Soil	1		409			30					
RV092-10DA,	2	85	209	476	Fruit	–0	0.092	< 0.05	< 0.01		
Dundee,	(10)	89	207	472			0.093	< 0.05	< 0.01		
New York						0	0.55	< 0.05	< 0.01	< 0.01	0.60
USA, 2010							0.36	< 0.05	< 0.01	< 0.01	0.41
(Concord)							0.46 (mean)				0.51 (mean)
						3	0.39	< 0.05	< 0.01	< 0.01	0.44
							0.34	< 0.05	< 0.01	< 0.01	0.39
						7	0.18	< 0.05	< 0.01	< 0.01	0.23
							0.25	< 0.05	< 0.01	< 0.01	0.30
						14	0.22	< 0.05	0.011	0.011	0.28
							0.25	0.053	0.014	< 0.01	0.30
						20	0.21	0.068	0.013	0.011	0.29
							0.16	< 0.05	0.011	< 0.01	0.21
	1	83	410		Fruit	30	< 0.01	< 0.05	< 0.01	0.011	0.071
							< 0.01	< 0.05	< 0.01	0.012	0.072
RV093-10HA,	2	85	207	1967	Fruit	–0	0.11	< 0.05	< 0.01		
Orefield,	(10)	89	206	1953			0.14	< 0.05	< 0.01		
Pennsylvania						0	0.39	< 0.05	< 0.01	< 0.01	0.44
USA, 2010							0.38	< 0.05	< 0.01	< 0.01	0.43
(Concord)							0.39 (mean)				0.44 (mean)
						3	0.33	< 0.05	< 0.01	< 0.01	0.38
							0.38	< 0.05	< 0.01	0.011	0.44
						7	0.23	< 0.05	< 0.01	< 0.01	0.28
							0.22	< 0.05	< 0.01	< 0.01	0.27
	1	81	410		Fruit	28	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV094-10HA,	2	81	188	1882	Fruit	–0	0.14	< 0.05	< 0.01		
Branchton,	(9)	85	199	2314			0.18	< 0.05	< 0.01		
Ontario						0	0.37	< 0.05	< 0.01	< 0.01	0.42
Canada, 2010							0.49	< 0.05	< 0.01	< 0.01	0.54
(Sebrevois)							0.43 (mean)				0.48 (mean)
						3	0.16	< 0.05	< 0.01	< 0.01	0.21
							0.17	< 0.05	< 0.01	< 0.01	0.22
						5	0.19	< 0.05	< 0.01	< 0.01	0.24
							0.17	< 0.05	< 0.01	< 0.01	0.22
	1	79	408		Fruit	30	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV095-10DA,	2	83	197	392	Fruit	–0	0.077	< 0.05	< 0.01		
Branchton,	(9)	89	207	353			0.072	< 0.05	< 0.01		
Ontario						0	0.33	< 0.05	< 0.01	0.013	0.39
Canada, 2010							0.31	< 0.05	< 0.01	0.013	0.37
(Concord)							0.32 (mean)				0.38 (mean)
						3	0.29	< 0.05	< 0.01	0.014	0.35

Trial No., Location,	Application				Sample	DALA	Residues as parent (mg/kg)				
Year (Variety)	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							0.26	< 0.05	< 0.01	0.013	0.33
						7	0.19	< 0.05	< 0.01	0.013	0.26
							0.18	< 0.05	< 0.01	0.014	0.24
						14	0.17	< 0.05	< 0.01	0.015	0.23
							0.17	< 0.05	< 0.01	0.017	0.23
						21	0.12	< 0.05	< 0.01	0.016	0.19
							0.16	< 0.05	< 0.01	0.016	0.23
	1	75	408		Fruit	30	< 0.01	0.058	< 0.01	< 0.01	0.068
							< 0.01	0.053	< 0.01	< 0.01	0.063
RV096-10HA,	2	81	205	1391	Fruit	–0	0.16	< 0.05	< 0.01		
Branchton,	(9)	85	211	2359			0.20	< 0.05	< 0.01		
Ontario						0	0.58	< 0.05	< 0.01	< 0.01	0.63
Canada, 2010							0.47	< 0.05	< 0.01	< 0.01	0.52
(Frontenac)							0.52 (mean)				0.57 (mean)
						3	0.11	< 0.05	< 0.01	< 0.01	0.16
							0.15	< 0.05	< 0.01	< 0.01	0.20
						5	0.13	< 0.05	< 0.01	< 0.01	0.18
							0.10	< 0.05	< 0.01	< 0.01	0.15
	1	79	408		Fruit	30	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV097-10HA,	2	83	211	387	Fruit	–0	0.087	< 0.05	< 0.01		
Branchton,	(9)	89	206	334			0.12	< 0.05	< 0.01		
Ontario						0	0.31	< 0.05	< 0.01	< 0.01	0.36
Canada, 2010							0.32	< 0.05	< 0.01	< 0.01	0.37
(Marechal Foch)							0.31 (mean)				0.36 (mean)
						3	0.24	< 0.05	< 0.01	< 0.01	0.29
							0.19	< 0.05	< 0.01	< 0.01	0.24
						7	0.20	< 0.05	< 0.01	< 0.01	0.25
							0.21	< 0.05	< 0.01	< 0.01	0.26
	1	79	408		Fruit	30	< 0.01	0.072	< 0.01	< 0.01	0.082
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV098-10DA,	2	83	206	2296	Fruit	–0	0.44	< 0.05	< 0.01		
Artois,	(11)	85	207	2304			0.11	< 0.05	< 0.01		
California						0	2.3	< 0.05	0.013	< 0.01	2.3
USA, 2010							1.5	< 0.05	< 0.01	< 0.01	1.6
(Rubired)							1.9 (mean)				2.0 (mean)
						3	2.1	< 0.05	0.013	0.011	2.2
							0.78	< 0.05	< 0.01	< 0.01	0.83
						7	1.0	< 0.05	< 0.01	< 0.01	1.1
							1.4	< 0.05	0.011	< 0.01	1.4
						14	0.69	< 0.05	< 0.01	< 0.01	0.74
							0.41	< 0.05	< 0.01	< 0.01	0.46
						21	0.87	< 0.05	< 0.01	< 0.01	0.92
							0.70	< 0.05	< 0.01	< 0.01	0.75
	1	77	410		Fruit	30	0.049	0.067	< 0.01	< 0.01	0.12
							0.031	< 0.05	< 0.01	< 0.01	0.081
RV103-10HA,	2	85	206	2294	Fruit	–0	0.20	< 0.05	< 0.01		
Artois,	(10)	89	205	2300			0.034	< 0.05	< 0.01		
California						0	0.21	< 0.05	< 0.01	< 0.01	0.26
USA, 2010							0.48	< 0.05	< 0.01	< 0.01	0.53
(Syrah Noir)						3	0.37	< 0.05	< 0.01	< 0.01	0.42
							0.50	< 0.05	< 0.01	< 0.01	0.55
							0.43 (mean)				0.49 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
						7	0.22	< 0.05	< 0.01	< 0.01	0.27
							0.18	< 0.05	< 0.01	< 0.01	0.23
	1	83	410		Fruit	30	0.015	< 0.05	< 0.01	< 0.01	0.065
							0.032	< 0.05	< 0.01	< 0.01	0.082
RV099-10DA,	2	81	207	2062	Fruit	–0	0.26	< 0.05	< 0.01		
Madera,	(10)	85	206	2076			0.27	< 0.05	< 0.01		
California						0	0.62	< 0.05	< 0.01	< 0.01	0.67
USA, 2010							0.51	< 0.05	< 0.01	< 0.01	0.56
(Thompson Seedless)							0.57 (mean)				0.62 (mean)
						3	0.50	< 0.05	< 0.01	< 0.01	0.55
							0.63	< 0.05	< 0.01	< 0.01	0.68
						7	0.48	0.091	< 0.01	< 0.01	0.57
							0.43	0.084	< 0.01	< 0.01	0.52
						14	0.51	0.12	< 0.01	< 0.01	0.63
							0.41	0.15	< 0.01	< 0.01	0.56
						21	0.38	0.20	< 0.01	< 0.01	0.58
							0.51	0.18	< 0.01	< 0.01	0.68
											0.63 (mean)
	1	75	410		Fruit	30	< 0.01	0.078	< 0.01	< 0.01	0.088
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV100-10HA,	2	81	209	2083	Fruit	–0	0.12	< 0.05	< 0.01		
Madera,	(10)	85	202	2085			0.11	< 0.05	< 0.01		
California						0	0.59	< 0.05	< 0.01	< 0.01	0.64
USA, 2010							0.39	< 0.05	< 0.01	< 0.01	0.44
(Thompson Seedless)							0.49 (mean)				0.54 (mean)
						3	0.32	< 0.05	< 0.01	< 0.01	0.37
							0.40	< 0.05	< 0.01	< 0.01	0.45
						7	0.27	< 0.05	< 0.01	< 0.01	0.32
							0.27	< 0.05	< 0.01	< 0.01	0.32
	1	75	410		Fruit	30	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	0.094	< 0.01	< 0.01	0.10
RV101-10HA,	2	85	211	2244	Fruit	–0	0.45	< 0.05	< 0.01		
Hughson,	(10)	89	206	2196			0.89	< 0.05	< 0.01		
California						0	0.90	< 0.05	< 0.01	< 0.01	0.95
USA, 2010							0.71	< 0.05	< 0.01	< 0.01	0.76
(Thompson Seedless)							0.80 (mean)				0.85 (mean)
						3	0.57	0.063	< 0.01	0.01	0.64
							0.64	0.068	< 0.01	0.01	0.71
						7	0.61	0.093	0.01	0.013	0.71
							0.54	0.091	< 0.01	0.013	0.65
	1	77	411		Fruit	29	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV102-10HA,	2	85	206	418	Fruit	–0	0.12	< 0.05	< 0.01		
Templeton,	(10)	89	208	430			0.043	< 0.05	< 0.01		
California						0	0.24	< 0.05	< 0.01	< 0.01	0.29
USA, 2010							0.38	< 0.05	< 0.01	< 0.01	0.43
(Syrah Noir)							0.31 (mean)				0.36 (mean)
						3	0.11	< 0.05	< 0.01	< 0.01	0.16
							0.30	< 0.05	< 0.01	< 0.01	0.35
						6	0.088	< 0.05	< 0.01	< 0.01	0.14
							0.052	< 0.05	< 0.01	< 0.01	0.10
	1	85	410		Fruit	30	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060

Trial No., Location, Year (Variety)	Application No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)	Sample	DALA	Residues as parent (mg/kg) Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV104-10HA,	2 (11)	85	205	2337	Fruit	–0	0.35	< 0.05	< 0.01		
Kerman,		85	207	2361			0.28	< 0.05	< 0.01		
California						0	0.65	< 0.05	< 0.01	< 0.01	0.70
USA, 2010							0.54	< 0.05	< 0.01	< 0.01	0.59
(Thompson Seedless)						3	0.83	< 0.05	0.01	< 0.01	0.88
							0.55	< 0.05	0.01	< 0.01	0.60
							0.69 (mean)				0.74 (mean)
						7	0.39	< 0.05	< 0.01	< 0.01	0.44
							0.33	< 0.05	< 0.01	< 0.01	0.38
	1	85	410		Fruit	30	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV105-10HA,	2	85	194	1881	Fruit	–0	0.60	< 0.05	< 0.01		
Sanger,	(10)	89	209	1980			0.56	0.053	< 0.01		
California						0	1.1	< 0.05	< 0.01	< 0.01	1.1
USA, 2010							0.93	< 0.05	< 0.01	< 0.01	0.98
(Thompson Seedless						3	0.88	0.071	< 0.01	< 0.01	0.95
							0.88	0.075	< 0.01	< 0.01	0.95
						7	0.79	0.10	< 0.01	< 0.01	0.89
							1.2	0.16	0.013	0.012	1.4
							1.0 (mean)				1.2 (mean)
	1	81	410		Fruit	30	< 0.01	0.083	< 0.01	< 0.01	0.093
							< 0.01	0.079	< 0.01	< 0.01	0.089
RV106-10HA,	2	89	204	465	Fruit	–0	0.33	< 0.05	< 0.01		
Ephrata,	(10)	89	206	468			0.30	< 0.05	< 0.01		
Washington						0	1.3	< 0.05	< 0.01	< 0.01	1.4
USA, 2010							0.95	< 0.05	< 0.01	< 0.01	1.0
(White Riesling)							1.1 (mean)				1.2 (mean)
						3	1.1	< 0.05	< 0.01	< 0.01	1.1
							1.0	< 0.05	< 0.01	0.011	1.1
						7	0.83	0.088	< 0.01	0.014	0.93
							0.96	0.090	< 0.01	0.016	1.1
	1	85	426		Fruit	30	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV107-10HA,	2	83	198	628	Fruit	–0	0.11	< 0.05	< 0.01		
Hood River,	(10)	85	201	587			0.15	< 0.05	< 0.01		
Oregon						0	0.46	< 0.05	< 0.01	< 0.01	0.51
USA, 2010							0.56	< 0.05	< 0.01	< 0.01	0.61
(Chardonnay)						3	0.68	< 0.05	< 0.01	< 0.01	0.73
							0.48	< 0.05	< 0.01	< 0.01	0.53
							0.58 (mean)				0.63 (mean)
						7	0.46	< 0.05	< 0.01	< 0.01	0.51
							0.18	< 0.05	< 0.01	< 0.01	0.23
	1	83	410		Fruit	28	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

–0 = Sampled immediately before the second application

Supervised trials were carried out on strawberries (10 trials—Table 93) in the USA and Canada during the 2011 growing season (Dallstream 2012b, RARVY006). Two foliar applications of a 200 g/L SL formulation were made with ground equipment. Adjuvant [non-ionic surfactant at 0.20% v/v, methylated seed oil at 0.25% v/v or crop oil concentrate at 1.0% v/v] was added in all applications. From three trials, additional strawberry samples were collected at a 0-day PHI and were washed and cooked to evaluate potential residue reduction resulting from the common food preparation practices.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in strawberries were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data (mean recoveries, RSDs) were obtained for all analytes in strawberry fruit, cooked fruit and washed fruit.

Table 93 Residues from the foliar application of flupyradifurone to strawberries in the USA and Canada (Dallstream 2012b, RARVY006)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth	Rate (g ai/ha)	Volume			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Strawberries	2 (10)		205			0					
RV037-11DB,	2	81	208	381	Fruit	3DAA1	0.26	< 0.05	0.022	< 0.01	0.31
Penn Yan,	(8)	85	204	374			0.30	< 0.05	0.018	< 0.01	0.35
New York,						0	0.43	< 0.05	0.011	< 0.01	0.48
USA, 2011							0.34	< 0.05	0.011	< 0.01	0.39
(Honeoye)							0.38 (mean)				0.43 (mean)
						3	0.30	0.051	< 0.01	< 0.01	0.35
							0.23	0.053	< 0.01	< 0.01	0.28
						7	0.17	0.072	< 0.01	< 0.01	0.24
							0.16	0.069	< 0.01	< 0.01	0.23
						14	0.15	0.11	< 0.01	< 0.01	0.26
							0.16	0.13	< 0.01	< 0.01	0.29
						20	0.062	0.15	< 0.01	< 0.01	0.21
							0.056	0.15	< 0.01	< 0.01	0.20
RV038-11HA,	2	85	205	268	Fruit	3DAA1	0.28	< 0.05	< 0.01	< 0.01	0.33
Colbert,	(10)	87	205	256			0.23	< 0.05	< 0.01	< 0.01	0.28
Georgia,						0	0.52	< 0.05	< 0.01	< 0.01	0.57
USA, 2011							0.56	< 0.05	< 0.01	< 0.01	0.61
(Camarosa)							0.54 (mean)				0.59 (mean)
						3	0.46	< 0.05	0.013	< 0.01	0.51
							0.34	< 0.05	0.015	< 0.01	0.39
					Fruit	0	0.45 (av.)	< 0.05(av.)	< 0.01 (av.)	< 0.01 (av.)	0.50
					Whole fruit, washed	0	0.38 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.43
					Fruit, cooked	0	0.21 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.26
RV039-11HA,	2	85	206	210	Fruit	3DAA1	0.31	< 0.05	< 0.01	< 0.01	0.36
Alachua,	(10)	89	201	204			0.41	< 0.05	< 0.01	< 0.01	0.46
Florida						0	0.59	< 0.05	< 0.01	< 0.01	0.64
USA, 2011							0.57	0.054	< 0.01	< 0.01	0.62
(Amirouche)							0.58 (mean)				0.63 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth	Rate (g ai/ha)	Volume			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
						3	0.20	0.064	0.022	< 0.01	0.26
							0.20	0.065	0.017	< 0.01	0.27
RV040-11HA,	2	85	196	239	Fruit	3DAA1	0.16	< 0.05	< 0.01	< 0.01	0.21
Portage la Prairie,	(10)	87	203	247			0.18	< 0.05	< 0.01	< 0.01	0.23
Manitoba,						0	0.34	< 0.05	< 0.01	< 0.01	0.39
Canada, 2011							0.32	< 0.05	< 0.01	< 0.01	0.37
(Glooscap)							0.33 (mean)				0.38 (mean)
						3	0.22	< 0.05	< 0.01	< 0.01	0.27
							0.21	< 0.05	< 0.01	< 0.01	0.26
					Fruit	0	0.12 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.17
					Whole fruit, washed	0	0.11 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.16
					Fruit, cooked	0	0.079 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.13
RV041-11HA,	2	85	206	227	Fruit	3DAA1	0.15	< 0.05	< 0.01	< 0.01	0.20
Gregory,	(9)	87	204	231			0.22	< 0.05	< 0.01	< 0.01	0.27
Michigan,						0	0.47	< 0.05	< 0.01	< 0.01	0.52
USA, 2011							0.56	0.062	< 0.01	< 0.01	0.62
(Jewel)							0.51 (mean)				0.57 (mean)
						3	0.28	0.081	< 0.01	< 0.01	0.36
							0.37	0.087	< 0.01	< 0.01	0.46
RV042-11DA,	2	81	205	167	Fruit	3DAA1	0.030	< 0.05	< 0.01	< 0.01	0.080
Hart,	(10)	85	205	170			0.027	< 0.05	< 0.01	< 0.01	0.077
Michigan,						0	0.63	< 0.05	< 0.01	< 0.01	0.68
USA, 2011							0.45	< 0.05	< 0.01	< 0.01	0.50
(Seascape Everbearing)							0.54 (mean)				0.59 (mean)
						3	0.16	< 0.05	< 0.01	< 0.01	0.21
							0.28	< 0.05	< 0.01	< 0.01	0.33
						7	0.17	< 0.05	< 0.01	< 0.01	0.22
							0.14	< 0.05	< 0.01	< 0.01	0.19
						14	0.078	0.12	< 0.01	< 0.01	0.19
							0.086	0.093	< 0.01	< 0.01	0.18
						21	0.052	0.12	< 0.01	< 0.01	0.17
							0.034	0.10	< 0.01	< 0.01	0.13
RV043-11HA,	2	85	205	281	Fruit	3DAA1	0.27	< 0.05	0.012	< 0.01	0.32
Madera,	(9)	87	206	287			0.25	< 0.05	0.016	< 0.01	0.30
California						0	0.37	< 0.05	< 0.01	< 0.01	0.42
USA, 2011							0.38	< 0.05	< 0.01	< 0.01	0.43
(Diamante 54)							0.38 (mean)				0.43 (mean)
						3	0.25	0.051	0.016	< 0.01	0.30
							0.33	0.055	0.018	< 0.01	0.39
					Fruit	0	0.32 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.37
					Whole fruit, washed	0	0.32 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.37
					Fruit, cooked	0	0.16 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.21

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth	Rate (g ai/ha)	Volume			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RV044-11DA,	2	81	204	301	Fruit	3DAA1	0.46	< 0.05	0.018	< 0.01	0.51
Sanger, California	(10)	85	201	321			0.49	< 0.05	0.025	< 0.01	0.54
USA, 2011						0	0.64	0.081	< 0.01	< 0.01	0.72
(Seascape)							0.60	0.085	< 0.01	< 0.01	0.69
							0.62 (mean)				
						3	0.39	0.14	0.015	< 0.01	0.53
							0.33	0.12	0.012	< 0.01	0.44
						7	0.18	0.22	< 0.01	< 0.01	0.39
							0.14	0.20	< 0.01	< 0.01	0.34
						14	0.090	0.52	< 0.01	< 0.01	0.61
							0.076	0.47	< 0.01	< 0.01	0.54
						21	0.029	0.91	< 0.01	< 0.01	0.94
							0.026	0.83	< 0.01	< 0.01	0.86
											0.90 (mean)
RV045-11DA,	2	85	207	284	Fruit	3DAA1	0.23	< 0.05	< 0.01	< 0.01	0.28
Fresno,	(10)	85	207	283			0.17	< 0.05	< 0.01	< 0.01	0.22
California						0	0.24	< 0.05	< 0.01	< 0.01	0.29
USA, 2011							0.21	< 0.05	< 0.01	< 0.01	0.26
(Albion)							0.23 (mean)				
						3	0.18	< 0.05	0.011	< 0.01	0.23
							0.17	< 0.05	0.013	< 0.01	0.22
						7	0.15	0.059	< 0.01	< 0.01	0.21
							0.20	0.063	0.012	< 0.01	0.27
						14	0.20	0.090	< 0.01	< 0.01	0.29
							0.27	0.10	< 0.01	< 0.01	0.37
											0.33 (mean)
						21	0.067	0.17	< 0.01	< 0.01	0.23
							0.076	0.14	< 0.01	< 0.01	0.21
RV046-11HA,	2	85	212	210	Fruit	3DAA1	0.22	< 0.05	< 0.01	< 0.01	0.27
Langley,	(8)	89	203	198			0.21	< 0.05	< 0.01	< 0.01	0.26
British						0	0.36	< 0.05	< 0.01	< 0.01	0.41
Columbia							0.40	< 0.05	< 0.01	< 0.01	0.45
Canada, 2011						3	0.44	< 0.05	0.014	< 0.01	0.49
(Puget Reliance)							0.41	< 0.05	0.014	< 0.01	0.46
							0.43 (mean)				0.48 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

DAA1 = Days after first application

Assorted tropical and sub-tropical fruits-inedible peel

Prickly pear

Supervised trials were carried out on prickly pear cactus (eight trials—Table 94) in the USA during the 2011 growing season (Dorschner 2012c, IR-4 PR No. 10722). Four trials generated fruit samples and four generated pad samples. Two foliar applications of a 200 g/L SL formulation were made with ground equipment. A non-ionic surfactant was included in each tank-mix.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in prickly pear cactus fruit or pads were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data (mean recoveries, RSDs) were obtained for all analytes in both fruit and pads.

Table 94 Residues from the foliar application of flupyradifurone to prickly pear cactus in the USA (Dorschner 2012c, IR-4 PR No 10722)

Trial No., Location, Year (Variety)	Application			Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Prickly Pear	2 (7)	205			21					
CA*01,	2	207	935	Fruit	21	0.15	< 0.05	< 0.01	< 0.01	0.20
Chualar, California	(7)	206	930			0.083	< 0.05	< 0.01	< 0.01	0.13
USA, 2011						0.12				0.17
(Andy Boy "Red")						(mean)				(mean)
CA*02,	2	208	678	Fruit	20	0.12	< 0.05	< 0.01	< 0.01	0.17
Chualar, California	(8)	211	682			0.13	< 0.05	< 0.01	< 0.01	0.18
USA, 2011						0.12				0.17
(Andy Boy "Red")						(mean)				(mean)
CA*143,	2	205	429	Fruit	20	0.11	< 0.05	< 0.01	< 0.01	0.16
Gonzales, California	(7)	205	428			0.095	< 0.05	< 0.01	< 0.01	0.15
USA, 2011						0.10				0.15
(Andy Boy "Red")						(mean)				(mean)
CA*144,	2	202	186	Fruit	21	0.047	< 0.05	< 0.01	< 0.01	0.097
Gonzales, California	(7)	202	187			0.089	< 0.05	< 0.01	< 0.01	0.14
USA, 2011						0.068				0.12
(Andy Boy "Red")						(mean)				(mean)
CA*160,	2	205	915	Pad	21	0.21	< 0.05	0.012	< 0.01	0.26
Chualar, California	(7)	207	924			0.20	< 0.05	< 0.01	< 0.01	0.25
USA, 2011						0.20				0.25
(Andy Boy "Red")						(mean)				(mean)
CA*161,	2	205	678	Pad	20	0.25	< 0.05	0.013	< 0.01	0.30
Chualar, California	(8)	210	690			0.15	< 0.05	< 0.01	< 0.01	0.20
USA, 2011						0.20				0.25
(Andy Boy "Red")						(mean)				(mean)
CA*162,	2	210	441	Pad	20	0.22	< 0.05	0.019	< 0.01	0.27
Gonzales, California	(7)	210	438			0.23	< 0.05	0.016	< 0.01	0.28
USA, 2011						0.23				0.28
(Andy Boy "Red")						(mean)				(mean)
CA*163,	2	206	200	Pad	21	0.24	< 0.05	0.023	< 0.01	0.29
Gonzales, California	(7)	215	198			0.27	< 0.05	0.025	< 0.01	0.32
USA, 2011						0.25				0.30
(Andy Boy "Red")						(mean)				(mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Bulb vegetables

Supervised trials were carried out on bulb onions (12 trials—Table 95) and green onions (five trials—Table 96) in the USA during the 2011 growing season (Fischer 2012a, RARVY025). Two foliar applications of a 200 g/L SL formulation were made with ground equipment. Adjuvant [non-ionic surfactant at 0.20% v/v, methylated seed oil at 0.25% v/v or crop oil concentrate at 1.0% v/v] was added in all applications.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in bulb onion bulbs and green onion whole plant without roots were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data were obtained for all analytes in both crops.

Table 95 Residues from the foliar application of flupyradifurone to bulb onions in the USA and Canada (Fischer 2012a, RARVY025)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth	Rate (g ai/ha)	Volume			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RV131-11DA,	2	41	212	280	Bulb	0	0.14	< 0.05	< 0.01	0.030	0.21
Germansville,	(8)	43	206	270			0.15	< 0.05	< 0.01	0.030	0.23
Pennsylvania,						7	0.049	< 0.05	< 0.01	0.048	0.15
USA, 2011							0.027	< 0.05	< 0.01	0.027	0.10
(Stuttgarter)						13	0.014	< 0.05	< 0.01	0.066	0.13
							0.013	0.053	< 0.01	0.069	0.13
						21	0.015	0.051	< 0.01	0.042	0.11
							0.013	< 0.05	< 0.01	0.05	0.11
						35	< 0.01	0.071	< 0.01	0.038	0.12
							< 0.01	0.067	< 0.01	0.030	0.11
						42	< 0.01	0.085	< 0.01	0.035	0.13
							< 0.01	0.067	< 0.01	0.027	0.10
RV132-11HA,	2	45	205	140	Bulb	13	0.042	< 0.05	< 0.01	0.031	0.12
Rockwood,	(8)	48	209	140			0.042	0.057	< 0.01	0.038	0.13
Ontario,											
Canada, 2011											
(Yellow Sweet Spanish)											
RV133-11HA,	2	45	201	130	Bulb	12	0.019	< 0.05	< 0.01	0.038	0.10
Springfield,	(9)	47	207	140			0.014	< 0.05	< 0.01	0.027	0.091
Nebraska,											
USA, 2011											
(Stuttgarter)											
RV134-11HA,	2	19	204	140	Bulb	14	< 0.01	< 0.05	< 0.01	0.016	0.076
Lenexa,	(9)	47	208	150			< 0.01	< 0.05	< 0.01	0.020	0.080
Kansas,											
USA, 2011											
(Stuttgarter)											
RV135-11DA,	2	43	208	160	Bulb	0	0.082	< 0.05	< 0.01	< 0.01	0.13
Carlyle,	(10)	45	205	150			0.064	< 0.05	< 0.01	< 0.01	0.11
Illinois,						7	< 0.01	< 0.05	< 0.01	0.014	0.074
USA, 2011							< 0.01	< 0.05	< 0.01	0.015	0.075
(White onion						14	< 0.01	0.082	< 0.01	0.013	0.10
sets)							< 0.01	0.080	< 0.01	< 0.01	0.090
						21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						35	< 0.01	0.15	< 0.01	< 0.01	0.16
							< 0.01	0.094	< 0.01	< 0.01	0.10
						42	< 0.01	0.11	< 0.01	0.012	0.13
							< 0.01	0.058	< 0.01	< 0.01	0.068
RV136-11DA,	2	47	205	220	Bulb	0	0.054	< 0.05	< 0.01	< 0.01	0.10
Stewardson,	(9)	48	208	250			0.10	< 0.05	< 0.01	< 0.01	0.15
Illinois,						7	0.068	< 0.05	< 0.01	0.014	0.13
USA, 2011							0.077	< 0.05	< 0.01	0.052	0.18
(Red Candy						14	0.049	< 0.05	< 0.01	0.026	0.12
Apple)							0.055	< 0.05	< 0.01	0.014	0.12
						21	0.021	< 0.05	< 0.01	0.013	0.083
							0.027	< 0.05	< 0.01	0.015	0.092
						33	0.020	< 0.05	< 0.01	< 0.01	0.070
							0.012	< 0.05	< 0.01	< 0.01	0.062

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth	Rate (g ai/ha)	Volume			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
						40	0.034	< 0.05	< 0.01	< 0.01	0.084
							0.041	< 0.05	< 0.01	0.033	0.12
RV137-11HA,	2	45	206	260	Bulb	14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
East Bernard,	(9)	47	204	270			< 0.01	< 0.05	< 0.01	0.012	0.072
Texas,											
USA, 2011											
(Texas Sweet 1015Y)											
RV138-11HA,	2	47	202	170	Bulb	12	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Wall, Texas,	(11)	48	207	180			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2011											
(White Bermuda)											
RV139-11HA,	2	47	206	240	Bulb	14	0.030	< 0.05	< 0.01	0.013	0.093
King City,	(10)	47	208	240			0.034	< 0.05	< 0.01	0.014	0.098
California, USA, 2011											
(Centerstone)											
RV140-11DA,	2	45	201	190	Bulb	0	0.088	< 0.05	< 0.01	0.013	0.15
Sanger,	(10)	48	204	190			0.086	< 0.05	< 0.01	0.016	0.15
California,						7	0.033	< 0.05	< 0.01	0.027	0.11
USA, 2011							0.028	< 0.05	< 0.01	0.023	0.10
(White						14	0.026	< 0.05	< 0.01	0.019	0.095
Ringmaster)							0.023	< 0.05	< 0.01	0.018	0.091
						21	0.014	< 0.05	< 0.01	0.017	0.080
							0.012	< 0.05	< 0.01	0.025	0.087
						35	0.032	< 0.05	< 0.01	0.026	0.11
							< 0.01	< 0.05	< 0.01	0.014	0.074
						42	< 0.01	< 0.05	< 0.01	0.024	0.084
							0.01	< 0.05	< 0.01	0.023	0.085
RV141-11HA,	2	43	203	190	Bulb	14	0.024	< 0.05	< 0.01	< 0.01	0.074
Ephrata,	(10)	45	201	190			0.023	< 0.05	< 0.01	0.01	0.083
Washington,											
USA, 2011											
(Colorado #6)											
RV142-11HA,	2	47	200	190	Bulb	14	0.045	< 0.05	< 0.01	< 0.01	0.095
Abbotsford,	(11)	48	211	210			0.053	< 0.05	< 0.01	< 0.01	0.10
British Columbia,											
Canada, 2011											
(Yellow)											

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 96 Residues from the foliar application of flupyradifurone to green onions in the USA (Fischer 2012, RARVY025)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RV143-11HA,	2	14	192	140	Whole	14	0.13	< 0.05	< 0.01	0.020	0.20
Carlyle,	(8)	17	206	160	plant		0.16	0.063	< 0.01	0.036	0.25
Illinois,					without						

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
USA, 2011											
(Gramex)					root						
RV144-11DA,	2	16	211	240	Whole	0	4.3	< 0.05	0.019	< 0.01	4.4
Stewardson,	(9)	17	211	220	plant		5.1	< 0.05	0.025	< 0.01	5.2
Illinois,					without	6	1.1	< 0.05	0.028	0.018	1.2
USA, 2011					root		1.0	< 0.05	0.032	0.020	1.1
(Spanish						12	0.40	< 0.05	0.016	0.019	0.47
Ringmaster)							0.47	< 0.05	0.014	0.01	0.53
						20	0.14	< 0.05	< 0.01	< 0.01	0.19
							0.11	< 0.05	< 0.01	< 0.01	0.16
						33	0.042	< 0.05	< 0.01	< 0.01	0.092
							0.036	< 0.05	< 0.01	< 0.01	0.086
						42	0.021	< 0.05	< 0.01	< 0.01	0.071
							0.022	< 0.05	< 0.01	< 0.01	0.072
RV145-11HA,	2	41	207	140	Whole	14	1.0	0.23	0.058	0.030	1.3
Wall,	(8)	41	203	140	plant		1.3	0.26	0.088	0.031	1.6
Texas,					without						
USA, 2011					root						
(Evergreen White Bunching)											
RV146-11HA,	2	13	204	190	Whole	14	0.41	< 0.05	0.12	0.014	0.47
Salinas,	(11)	15	211	190	plant		0.37	< 0.05	0.10	0.014	0.44
California,					without						
USA, 2011					root						
(Stuttgarter)											
RV147-11DA,	2	41	200	180	Whole	0	6.4	< 0.05	0.20	0.062	6.5
Sanger,	(10)	43	204	180	plant		6.9	< 0.05	0.21	0.067	7.0
California,					without	7	1.6	0.089	0.21	0.072	1.7
USA, 2011					Root		1.8	0.099	0.24	0.094	2.0
(Nebuka						14	0.89	0.20	0.16	0.090	1.2
Evergreen							1.0	0.20	0.16	0.087	1.3
White)						21	0.80	0.27	0.15	0.069	1.1
							0.64	0.16	0.13	0.051	0.86
						35	0.47	0.29	0.098	0.038	0.79
							0.41	0.24	0.079	0.037	0.69
						42	0.36	0.28	0.089	0.025	0.67
							0.32	0.26	0.079	0.034	0.62

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Brassica vegetables

Supervised trials were carried out on broccoli (four trials—Table 97), cabbage (10 trials—Table 98) and cauliflower (six trials—Table 99) in the USA during the 2010–11 growing seasons (Beedle 2012, RARVY023). Two foliar applications of a 200 g/L SL formulation were made with ground equipment. Adjuvant was added in all applications at 0.25% v/v. Cabbage heads with wrapper leaves removed were collected from each cabbage trial at 1 and 3-day PHIs. Additional broccoli samples were collected from one trial to evaluate potential residue reduction from washing and cooking.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in broccoli, cabbage and cauliflower were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data were obtained for each analyte in each vegetable.

Table 97 Residues from the foliar application of flupyradifurone to broccoli in the USA and Canada (Beedle 2012, RARVY023)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 5	2 (7)		205			1					
RV254-10DA,	2	46	200	138	Heads	–4	1.3	0.39	< 0.01	0.12	1.8
Stewardson,	(7)	49	209	150	and		1.6	0.40	< 0.01	0.10	2.1
Illinois,					stalks	0	3.2	0.67	< 0.01	0.18	4.1
USA, 2010							3.0	0.51	< 0.01	0.15	3.6
(Packman)						1	1.5	0.85	< 0.01	0.23	2.6
							2.3	0.85	< 0.01	0.24	3.4
							1.9 (mean)				
						3	0.24	1.2	< 0.01	0.45	1.9
							0.23	1.2	< 0.01	0.43	1.9
						5	0.12	1.4	< 0.01	0.45	2.0
							0.075	1.4	< 0.01	0.43	1.9
						12	0.048	3.0	< 0.01	0.46	3.5
							0.045	2.7	< 0.01	0.41	3.1
											3.3
											(mean)
					Heads stalks	1	0.59 (av.)	0.78 (av.)	0.59 (av.)	0.29	1.7
					Washed	1	0.60 (av.)	1.2 (av.)	< 0.01 (av.)	0.65 (av.)	2.4
					Washed / Cooked	1	0.31 (av.)	0.42 (av.)	< 0.01 (av.)	0.27 (av.)	1.0
RV255-10HA,	2	45	204	261	Heads	–3	0.22	0.30	< 0.01	0.17	0.69
Branchton,	(6)	48	204	231	and		0.22	0.26	< 0.01	0.14	0.62
Ontario,					stalks	1	0.36	0.82	< 0.01	0.47	1.6
Canada, 2010							0.38	0.88	< 0.01	0.50	1.8
							0.37 (mean)				
(Windsor)						3	0.18	1.7	< 0.01	0.73	2.6
							0.14	1.5	< 0.01	0.70	2.3
											2.5
											(mean)
RV256-10DA,	2	49	204	244	Heads	–4	0.20	< 0.05	< 0.01	< 0.01	0.25
Sanger,	(7)	49	205	306	and		0.18	< 0.05	< 0.01	< 0.01	0.23
California,					stalks	0	0.53	< 0.05	< 0.01	< 0.01	0.58
USA, 2010							0.48	< 0.05	< 0.01	0.011	0.54
(Green Magic)						1	0.42	< 0.05	< 0.01	0.013	0.48
							0.38	< 0.05	< 0.01	0.011	0.44
							0.40 (mean)				0.46 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
						3	0.29	< 0.05	< 0.01	0.019	0.36
							0.25	< 0.05	< 0.01	0.015	0.31
						7	0.22	< 0.05	< 0.01	0.017	0.28
							0.25	0.073	< 0.01	0.023	0.35
						14	0.16	0.081	< 0.01	0.028	0.27
							0.15	0.092	< 0.01	0.026	0.27
RV257-10HA,	2	48	202	368	Heads	–4	0.55	< 0.05	< 0.01	0.013	0.61
Aromas,	(7)	49	209	317	and		0.81	< 0.05	< 0.01	0.012	0.88
California,					stalks	1	0.88	0.19	< 0.01	0.092	1.2
USA, 2010							1.0	0.21	< 0.01	0.090	1.3
(Green Magic)							0.95 (mean)				1.2 (mean)
						3	0.79	0.27	< 0.01	0.097	1.2
							0.79	0.36	< 0.01	0.13	1.3

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 98 Residues from the foliar application of flupyradifurone to cabbage in the USA and Canada (Beedle 2012, RARVY023)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 5	2 (7)		205			1					
RV258-10DA,	2	46	207	284	Head	–4	0.26	0.11	< 0.01	0.023	0.39
Alton, New	(7)	49	205	281	with		0.20	0.10	< 0.01	0.024	0.32
York,					wrapper	0	0.75	0.13	< 0.01	0.026	0.91
USA, 2010 (Winner)					leaves		0.60	0.13	< 0.01	0.033	0.76
						1	0.44	0.22	< 0.01	0.042	0.70
							0.33	0.21	< 0.01	0.051	0.59
							0.38 (mean)				
						3	0.44	0.25	< 0.01	0.047	0.74
							0.30	0.24	< 0.01	0.052	0.60
											0.67 (mean)
						7	0.079	0.32	< 0.01	0.041	0.44
							0.11	0.34	< 0.01	0.047	0.49
						14	0.030	0.48	< 0.01	0.058	0.57
							0.024	0.30	< 0.01	0.036	0.36
					Head,	–4	0.021	< 0.05	< 0.01	0.014	0.085
					trimm		0.020	< 0.05	< 0.01	0.017	0.087
					ed	1	0.046	0.13	< 0.01	0.034	0.21
							0.073	0.14	< 0.01	0.042	0.25
						3	0.051	0.19	< 0.01	0.039	0.28
							0.047	0.19	< 0.01	0.045	0.28
											0.28 (mean)
RV259-10DA,	2	48	206	227	Head	–2	0.85	< 0.05	< 0.01	0.012	0.91
Elko, South	(5)	49	205	226	with		1.1	< 0.05	< 0.01	0.012	1.1

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Carolina,					wrapper	0	0.92	0.072	< 0.01	0.026	1.0
USA, 2010					leaves		1.6	0.11	< 0.01	0.028	1.7
(Bravo)						1	0.45	< 0.05	< 0.01	0.017	0.52
							1.2	0.092	< 0.01	0.027	1.3
							0.82 (mean)				
						3	0.50	0.093	< 0.01	0.039	0.63
							0.84	0.13	< 0.01	0.043	1.0
						6	0.81	0.34	< 0.01	0.083	1.2
							0.65	0.31	< 0.01	0.080	1.0
											1.1 (mean)
						14	0.12	0.56	< 0.01	0.14	0.81
							0.11	0.49	< 0.01	0.15	0.75
					Head,	–2	0.040	< 0.05	< 0.01	< 0.01	0.090
					trimm		0.026	< 0.05	< 0.01	< 0.01	0.076
					ed	1	0.05	< 0.05	< 0.01	0.020	0.12
							0.015	< 0.05	< 0.01	0.012	0.077
						3	0.064	0.056	< 0.01	0.032	0.15
							0.031	0.057	< 0.01	0.030	0.12
											0.14 (mean)
RV260-10HA,	2	48	203	278	Head	–4	0.12	< 0.05	< 0.01	0.052	0.23
Oviedo, Florida,	(7)	49	207	282			0.12	< 0.05	< 0.01	0.048	0.22
USA, 2010						1	0.30	0.20	< 0.01	0.090	0.58
(Big Flathead)							0.37	0.18	< 0.01	0.075	0.62
							0.33 (mean)				0.60 (mean)
						3	0.19	0.26	< 0.01	0.097	0.55
							0.17	0.23	< 0.01	0.095	0.49
					Head,	–4	< 0.01	< 0.05	< 0.01	0.053	0.11
					trimm		0.013	< 0.05	< 0.01	0.042	0.11
					ed	1	0.017	0.080	< 0.01	0.068	0.17
							0.063	0.10	< 0.01	0.074	0.24
						3	0.044	0.12	< 0.01	0.071	0.24
							0.040	0.15	< 0.01	0.076	0.27
											0.25
											(mean)
RV261-10HA,	2	48	204	230	Head	3	0.14	< 0.05	< 0.01	0.014	0.20
Branchton,	(6)	49	205	235	with		0.068	< 0.05	< 0.01	0.017	0.14
Ontario,					wrapper	1	0.074	< 0.05	< 0.01	0.023	0.15
Canada, 2010					leaves		0.17	< 0.05	< 0.01	0.028	0.25
(Constellation)							0.12				0.20
							(mean)				(mean)
						3	0.077	< 0.05	< 0.01	0.025	0.15
							0.081	0.051	< 0.01	0.026	0.16
					Head,	–3	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
					trimm		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
					ed	1	< 0.01	< 0.05	< 0.01	0.013	0.073
							< 0.01	< 0.05	< 0.01	0.011	0.071
						3	< 0.01	< 0.05	< 0.01	0.013	0.073
							0.011	< 0.05	< 0.01	0.017	0.078
											0.076 (mean)
RV262-10DA,	2	48	203	236	Head	–3	0.039	< 0.05	< 0.01	0.014	0.10
Branchton,	(6)	49	204	242	with		0.049	< 0.05	< 0.01	0.012	0.11

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Ontario,					wrapper	0	0.19	< 0.05	< 0.01	0.015	0.26
Canada, 2010					leaves		0.18	< 0.05	< 0.01	0.014	0.25
(Adaptor)						1	0.072	< 0.05	< 0.01	0.019	0.14
							0.077	< 0.05	< 0.01	0.022	0.15
						3	0.088	< 0.05	< 0.01	0.019	0.16
							0.066	< 0.05	< 0.01	0.019	0.14
							0.077 (mean)				
						6	0.032	0.065	< 0.01	0.024	0.12
							0.043	0.054	< 0.01	0.019	0.12
						14	0.011	0.12	< 0.01	0.036	0.17
							< 0.01	0.10	< 0.01	0.037	0.15
											0.16 (mean)
					Head,	–3	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
					trimm		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
					ed	1	< 0.01	< 0.05	< 0.01	0.01	0.070
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						3	< 0.01	< 0.05	< 0.01	0.013	0.073
							< 0.01	< 0.05	< 0.01	0.012	0.072
											0.073 (mean)
RV263-10DA, Stewardson, Illinois, USA, 2010 (Stonehead)	2	45	198	138	Head	–4	0.062	< 0.05	< 0.01	0.044	0.16
	(7)	49	202	145	with		0.11	0.05	< 0.01	0.068	0.23
					wrapper	0	0.55	0.10	< 0.01	0.092	0.74
					leaves		0.45	0.13	< 0.01	0.11	0.69
						1	0.67	0.21	< 0.01	0.10	0.98
							0.71	0.21	< 0.01	0.11	1.0
							0.69 (mean)				1.0 (mean)
						3	0.20	0.27	< 0.01	0.14	0.60
							0.20	0.23	< 0.01	0.13	0.55
						5	0.058	0.30	< 0.01	0.14	0.49
							0.082	0.35	< 0.01	0.15	0.58
						12	0.018	0.39	< 0.01	0.10	0.51
							0.020	0.34	< 0.01	0.10	0.47
					Head,	–4	0.014	< 0.05	< 0.01	0.061	0.13
					Trimm		0.014	< 0.05	< 0.01	0.065	0.13
					ed	1	0.027	0.14	< 0.01	0.11	0.27
							0.034	0.12	< 0.01	0.11	0.26
						3	0.030	0.20	< 0.01	0.14	0.37
							0.023	0.20	< 0.01	0.15	0.37
											0.37 (mean)
RV264-10HA, Conklin, Michigan, USA, 2010 (Megaton)	2	48	205	252	Head	–4	0.10	0.085	< 0.01	0.020	0.21
	(7)	49	206	257	with		0.15	0.088	< 0.01	0.020	0.26
					wrapper	1	0.21	0.14	< 0.01	0.034	0.38
					leaves		0.45	0.18	< 0.01	0.033	0.66
						3	0.47	0.30	< 0.01	0.045	0.81
							0.44	0.28	< 0.01	0.039	0.76
							0.45 (mean)				0.79 (mean)
					Head,	–4	0.067	< 0.05	< 0.01	0.01	0.13
					trimm		0.042	< 0.05	< 0.01	< 0.01	0.092
					ed	1	0.14	0.17	< 0.01	0.034	0.34

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							0.19	0.14	< 0.01	0.031	0.36
						3	0.20	0.19	< 0.01	0.032	0.42
							0.071	0.20	< 0.01	0.039	0.31
											0.37 (mean)
RV265-10HA,	2	48	212	191	Head	–4	0.30	< 0.05	< 0.01	< 0.01	0.35
Raymondville,	(7)	49	214	194	with		0.20	< 0.05	< 0.01	< 0.01	0.25
Texas, USA, 2011					wrapper	1	0.79	0.067	< 0.01	0.023	0.88
(Copenhagen Market)					leaves		0.88	0.070	< 0.01	0.029	0.97
							0.83 (mean)				0.93 (mean)
						3	0.43	0.12	< 0.01	0.036	0.58
							0.35	0.10	< 0.01	0.039	0.49
					Head,	–4	0.018	< 0.05	< 0.01	< 0.01	0.068
					trimm		0.034	< 0.05	< 0.01	< 0.01	0.084
					ed	1	0.17	< 0.05	< 0.01	0.018	0.24
							0.17	< 0.05	< 0.01	0.013	0.24
											0.24
											(mean)
						3	0.091	0.089	< 0.01	0.029	0.21
							0.067	0.076	< 0.01	0.024	0.17
RV266-10HA,	2	48	205	284	Head	–4	0.13	< 0.05	< 0.01	0.014	0.19
Sanger,	(7)	49	203	281	with		0.15	< 0.05	< 0.01	0.016	0.21
California, USA, 2010					wrapper	1	0.28	0.077	< 0.01	0.037	0.39
(Green Charmant)					leaves		0.37	0.099	< 0.01	0.032	0.50
							0.32 (mean)				0.44 (mean)
						3	0.14	0.13	< 0.01	0.053	0.33
							0.16	0.13	< 0.01	0.051	0.34
					Head,	–4	0.017	< 0.05	< 0.01	< 0.01	0.067
					trimm		0.013	< 0.05	< 0.01	0.013	0.076
					ed	1	0.040	< 0.05	< 0.01	0.031	0.12
							0.10	< 0.05	< 0.01	0.027	0.18
											0.15
											(mean)
						3	0.05	< 0.05	< 0.01	0.040	0.14
							0.025	< 0.05	< 0.01	0.044	0.12
RV267-10HA,	2	48	205	184	Head	–3	0.11	< 0.05	< 0.01	0.015	0.18
Portland,	(6)	49	208	194	with		0.095	< 0.05	< 0.01	0.013	0.16
Oregon, USA, 2010					wrapper	1	0.088	0.069	< 0.01	0.049	0.21
(Fast vantage F1)					leaves		0.12	0.081	< 0.01	0.056	0.26
							0.11 (mean)				
						3	0.052	0.12	< 0.01	0.079	0.25
							0.051	0.10	< 0.01	0.071	0.22
											0.24 (mean)
					Head,	–3	0.012	< 0.05	< 0.01	0.014	0.076
					trimm		0.012	< 0.05	< 0.01	0.016	0.078
					ed	1	0.028	0.076	< 0.01	0.074	0.18
							0.027	0.091	< 0.01	0.061	0.18
						3	0.022	0.088	< 0.01	0.064	0.17
							0.034	0.11	< 0.01	0.085	0.23

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
											0.20 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 99 Residues from the foliar application of flupyradifurone to cauliflower in the USA and Canada (Beedle 2012, RARVY023)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 5	2 (7)		205			1					
RV248-10HA, Alton, New York, USA, 2010 (Fremont)	2	46	207	284	Heads	–4	1.1	< 0.05	< 0.01	0.013	1.2
	(7)	49	206	282	and		0.86	< 0.05	< 0.01	0.014	0.92
					stalks	1	1.9	0.060	< 0.01	0.026	2.0
							2.2	0.066	< 0.01	0.029	2.3
							2.1 (mean)				2.2 (mean)
						3	0.88	0.088	< 0.01	0.028	1.0
							0.99	0.10	< 0.01	0.029	1.1
RV249-10DA, Branchton, Ontario, Canada, 2010 (Cupid)	2	48	204	240	Heads	–4	0.021	< 0.05	< 0.01	0.041	0.11
	(7)	49	205	257	and		0.054	0.052	< 0.01	0.048	0.15
					stalks	0	0.11	0.084	< 0.01	0.064	0.26
							0.090	0.081	< 0.01	0.059	0.23
						1	0.12	0.098	< 0.01	0.077	0.29
							0.058	0.11	< 0.01	0.074	0.24
							0.087 (mean)				
						3	0.054	0.15	< 0.01	0.095	0.30
							0.051	0.14	< 0.01	0.087	0.28
						7	0.025	0.26	< 0.01	0.13	0.41
							0.022	0.29	< 0.01	0.13	0.44
						14	0.016	0.33	< 0.01	0.11	0.45
							0.013	0.32	< 0.01	0.13	0.46
											0.46 (mean)
RV250-10HA, Stewardson, Illinois, USA, 2010 (Snow Crown)	2	46	198	137	Heads	–4	1.9	< 0.05	< 0.01	0.017	2.0
	(7)	49	212	153	and		1.8	< 0.05	< 0.01	0.016	1.8
					stalks	1	2.3	0.082	0.01	0.033	2.4
							2.5	0.078	< 0.01	0.035	2.6
							2.4 (mean)				2.5 (mean)
						3	0.22	0.071	< 0.01	0.067	0.35
							0.21	0.069	< 0.01	0.063	0.34
RV251-10HA,	2	49	205	172	Heads	–3	< 0.01	< 0.05	< 0.01	0.014	0.074
Guadalupe,	(6)	49	206	173	and		< 0.01	< 0.05	< 0.01	0.018	0.078
California,					stalks	1	0.016	< 0.05	< 0.01	0.038	0.10
USA, 2010							< 0.01	< 0.05	< 0.01	0.016	0.076
						3	0.014	< 0.05	< 0.01	0.042	0.11
							0.029	< 0.05	< 0.01	0.034	0.11
							0.022				0.11
							(mean)				(mean)
RV252-10DA,	2	48	202	313	Heads	–4	0.027	< 0.05	< 0.01	< 0.01	0.077
Sanger,	(7)	49	205	315	and		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
California,					stalks	0	0.059	< 0.05	< 0.01	< 0.01	0.11
USA, 2010							0.074	< 0.05	< 0.01	< 0.01	0.12
(Arctic)						1	0.015	< 0.05	< 0.01	0.015	0.080
							0.046	< 0.05	< 0.01	0.014	0.11
						3	0.032	0.054	< 0.01	0.018	0.10
							0.032	< 0.05	< 0.01	0.018	0.10
							0.032				
							(mean)				
						7	0.024	0.094	< 0.01	0.023	0.14
							0.030	0.079	< 0.01	0.020	0.13
						12	0.025	0.18	< 0.01	0.051	0.26
							0.028	0.19	< 0.01	0.044	0.26
											0.26
											(mean)
RV253-10HA,	2	49	211	199	Heads	–2	0.015	< 0.05	< 0.01	0.020	0.085
Portland,	(5)	49	204	208	and		0.012	< 0.05	< 0.01	0.019	0.081
Oregon,					stalks	1	0.10	< 0.05	< 0.01	0.042	0.19
USA, 2010							0.11	< 0.05	< 0.01	0.039	0.20
(Minute Man F1)							0.11 (mean)				0.20 (mean)
						3	0.11	0.069	< 0.01	0.044	0.23
							0.057	0.073	< 0.01	0.053	0.18

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Fruiting vegetables, Cucurbits

Supervised trials were carried out on cucumbers (nine trials—Table 100), summer squash (eight trials—Table 101) and muskmelons (five trials—Table 103) in the USA and Canada during the 2011 growing season (Beedle and Harbin 2012, RARVY021). Either two foliar applications of a 200 g/L SL formulation were made or one soil drench application of BYI 02960 200SL was made. Foliar applications were made with ground-based equipment. Adjuvant was added in all but one of the applications (NIS at 0.2% v/v, MSO at 0.25% v/v or a COC at 1.0% v/v). RV077-11HA soil drench was the exception.

Additional squash samples were collected at a 1-day PHI from the plots receiving foliar applications in three of the summer squash trials and were washed and cooked to evaluate potential residue reduction from common food preparation practices. Additionally, melon peeled fruit (pulp) samples were generated in all the muskmelon trials from both treated plots.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in cucumbers, muskmelons and summer squash were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data were obtained for each analyte in each vegetable.

Table 100 Residues from foliar and soil drench applications of flupyradifurone to cucumbers in the USA and Canada (Beedle and Harbin 2012, RARVY021)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 9 Foliar	2 (7)		205			1					
GAP, USA, Crop Group 9 Soil	1		409			21					
RV073-11DA, Seven Springs, North Carolina, USA, 2011 (Lancer 152)	2	51	202	158	Fruit	0	0.26	0.12	< 0.01	< 0.01	0.38
	(7)	71	207	160			0.21	0.14	< 0.01	< 0.01	0.35
						1	0.22	0.13	< 0.01	< 0.01	0.35
							0.16	0.14	< 0.01	< 0.01	0.30
							0.19 (mean)				
						7	0.10	0.36	< 0.01	< 0.01	0.46
							0.14	0.26	< 0.01	< 0.01	0.40
						14	0.072	0.44	< 0.01	< 0.01	0.51
							0.075	0.54	< 0.01	0.014	0.62
						21	0.036	0.64	< 0.01	0.018	0.70
							0.056	0.92	< 0.01	0.017	1.0
											0.85 (mean)
						28	< 0.01	0.55	< 0.01	< 0.01	0.56
							0.016	0.66	< 0.01	0.012	0.67
	1	23	410		Fruit	12	0.013	< 0.05	< 0.01	< 0.01	0.063
	(soil						0.023	< 0.05	< 0.01	< 0.01	0.073
	drench)					19	0.011	< 0.05	< 0.01	< 0.01	0.061
							0.014	< 0.05	< 0.01	< 0.01	0.064
							0.012 (mean)				
						26	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						33	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						40	< 0.01	0.054	< 0.01	< 0.01	0.064
							< 0.01	0.058	< 0.01	< 0.01	0.068
											0.066 (mean)
RV074-11DA, Sunsweet, Georgia, USA, 2011 (Thunder)	2	72	202	229	Fruit	0	0.093	0.23	< 0.01	< 0.01	0.32
	(7)	79	201	232			0.10	0.22	< 0.01	< 0.01	0.33
						1	0.10	0.28	< 0.01	0.012	0.40
							0.057	0.27	< 0.01	< 0.01	0.33
							0.081 (mean)				
						7	0.041	0.38	< 0.01	0.015	0.44
							0.032	0.41	< 0.01	0.013	0.45
											0.45 (mean)
						14	0.015	0.41	< 0.01	0.014	0.44
							< 0.01	0.35	< 0.01	0.014	0.37
						21	< 0.01	0.29	< 0.01	< 0.01	0.30
							< 0.01	0.36	< 0.01	< 0.01	0.37
						27	< 0.01	0.28	< 0.01	0.012	0.30
							< 0.01	0.32	< 0.01	< 0.01	0.33
	1	63	410		Fruit	14	0.012	0.064	< 0.01	0.014	0.091
	(soil						< 0.01	0.061	< 0.01	0.011	0.082
	drench)					21	< 0.01	0.075	< 0.01	< 0.01	0.085
							0.011	0.070	< 0.01	< 0.01	0.081
							0.011				
							(mean)				
						28	< 0.01	0.087	< 0.01	< 0.01	0.097

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							< 0.01	0.088	< 0.01	< 0.01	0.098
						35	< 0.01	0.094	< 0.01	< 0.01	0.10
							< 0.01	0.12	< 0.01	< 0.01	0.13
						42	< 0.01	0.13	< 0.01	< 0.01	0.14
							< 0.01	0.10	< 0.01	< 0.01	0.11
											0.13 (mean)
RV075-11DA, High Springs, Florida, USA, 2011 (Thunder)	2	69	210	232	Fruit	0	0.069	0.13	< 0.01	0.025	0.22
	(8)	76	206	229			0.055	0.16	< 0.01	0.017	0.24
						1	0.098	0.19	< 0.01	0.020	0.31
							0.13	0.13	< 0.01	0.015	0.27
							0.11 (mean)				
						7	0.043	0.34	< 0.01	0.014	0.39
							0.067	0.31	< 0.01	0.015	0.39
						14	0.028	0.58	< 0.01	0.025	0.63
							0.028	0.56	< 0.01	< 0.01	0.59
						21	0.027	0.61	< 0.01	0.019	0.65
							0.017	0.81	< 0.01	< 0.01	0.83
						28	0.018	1.11	< 0.01	0.015	1.1
							0.037	0.81	< 0.01	0.026	0.87
											1.0 (mean)
	1	29	410		Fruit	14	0.012	0.31	< 0.01	0.012	0.33
	(soil						0.013	0.41	< 0.01	0.017	0.44
	drench)					21	0.011	0.69	< 0.01	0.013	0.72
							0.018	0.85	< 0.01	0.014	0.88
							0.015 (mean)				
						28	0.012	0.53	< 0.01	< 0.01	0.54
							0.01	0.89	< 0.01	< 0.01	0.90
						35	0.012	1.2	< 0.01	< 0.01	1.2
							< 0.01	0.74	< 0.01	< 0.01	0.75
						42	< 0.01	0.55	< 0.01	< 0.01	0.56
							< 0.01	1.4	< 0.01	< 0.01	1.5
											1.0 (mean)
RV076-11DA, Bagley, Iowa, USA, 2011 (Marketmore 76)	2	72	202	980	Fruit	0	0.19	< 0.05	< 0.01	< 0.01	0.24
	(7)	83	203	930			0.26	< 0.05	< 0.01	< 0.01	0.31
						1	0.25	< 0.05	< 0.01	< 0.01	0.30
							0.20	0.05	< 0.01	< 0.01	0.25
							0.23 (mean)				0.28 (mean)
						7	0.15	< 0.05	< 0.01	< 0.01	0.20
							0.14	< 0.05	< 0.01	< 0.01	0.19
						14	0.028	< 0.05	< 0.01	< 0.01	0.078
							0.018	< 0.05	< 0.01	< 0.01	0.068
						21	0.041	0.056	< 0.01	< 0.01	0.097
							0.046	0.084	< 0.01	< 0.01	0.13
						28	0.043	0.10	< 0.01	< 0.01	0.14
							0.11	0.18	< 0.01	0.01	0.30
	1	65	413		Fruit	13	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(soil						< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	drench)					21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01 (mean)				

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
						28	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						34	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						41	< 0.01	0.12	< 0.01	< 0.01	0.13
							< 0.01	0.33	< 0.01	< 0.01	0.34
											0.24 (mean)
RV077-11HA, Branchton, Ontario, Canada, 2011 (Taladaga)	2	85	210	206	Fruit	1	0.096	0.095	< 0.01	< 0.01	0.19
	(7)	85	209	204			0.088	0.089	< 0.01	< 0.01	0.18
							0.092 (mean)				0.18 (mean)
	1	66	410			21	0.018	0.39	< 0.01	0.019	0.43
	(soil drench)						0.025	0.47	< 0.01	0.025	0.51
							0.022 (mean)				0.47 (mean)
RV078-11DA, York, Nebraska, USA, 2011 (Sweet Burpless Hybrid)	2	72	207	195	Fruit	0	0.052	0.128	< 0.01	0.01	0.19
	(7)	73	206	193			0.054	0.076	< 0.01	0.013	0.14
						1	0.034	0.076	< 0.01	< 0.01	0.11
							0.043	0.10	< 0.01	0.011	0.16
							0.039 (mean)				
						7	0.028	0.24	< 0.01	0.016	0.28
							0.026	0.21	< 0.01	0.013	0.25
						14	0.028	0.53	< 0.01	0.020	0.58
							0.040	0.41	< 0.01	0.021	0.47
						21	0.034	0.53	< 0.01	0.013	0.58
							0.026	0.42	< 0.01	0.012	0.46
						28	0.018	0.54	< 0.01	< 0.01	0.55
							0.015	0.65	< 0.01	0.019	0.68
											0.62 (mean)
	1	61	408		Fruit	14	0.015	0.32	< 0.01	0.015	0.35
	(soil						0.015	0.45	< 0.01	0.019	0.48
	drench)					20	< 0.01	0.12	< 0.01	< 0.01	0.13
							0.024	0.23	< 0.01	< 0.01	0.25
						28	0.032	0.44	< 0.01	< 0.01	0.47
							0.021	0.28	< 0.01	< 0.01	0.30
							0.027 (mean)				
						34	0.021	0.39	< 0.01	< 0.01	0.41
							0.012	0.32	< 0.01	< 0.01	0.33
						41	0.019	0.48	< 0.01	< 0.01	0.50
							0.014	0.32	< 0.01	< 0.01	0.34
											0.42 (mean)
RV079-11DA, Campbell, Minnesota, USA, 2011 (Speedway)	2	63	205	187	Fruit	0	0.064	< 0.05	< 0.01	< 0.01	0.11
	(7)	71	205	187			0.068	< 0.05	< 0.01	< 0.01	0.12
						1	0.099	0.068	< 0.01	< 0.01	0.17
							0.067	0.073	< 0.01	< 0.01	0.14
							0.083 (mean)				
						5	0.025	0.13	< 0.01	< 0.01	0.15
							0.022	0.13	< 0.01	0.011	0.16
											0.16

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
											(mean)
						14	< 0.01	0.078	< 0.01	< 0.01	0.088
							< 0.01	0.074	< 0.01	< 0.01	0.084
						21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	0.061	< 0.01	< 0.01	0.071
						28	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	1	54	410		Fruit	14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(soil						< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	drench)					19	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01 (mean)				
						28	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	0.051	< 0.01	< 0.01	0.061
											0.061 (mean)
						35	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						42	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV080-11DA, Uvalde, Texas, USA, 2011 (Stonewall)	2	79	208	228	Fruit	0	0.068	< 0.05	< 0.01	< 0.01	0.12
	(7)	84	208	221			0.14	< 0.05	< 0.01	< 0.01	0.19
						1	0.059	< 0.05	< 0.01	< 0.01	0.11
							0.14	0.055	< 0.01	< 0.01	0.20
							0.10 (mean)				
						7	0.037	0.28	< 0.01	< 0.01	0.32
							0.044	0.17	< 0.01	< 0.01	0.22
						14	0.040	0.25	< 0.01	< 0.01	0.29
							0.026	0.25	< 0.01	< 0.01	0.27
											0.28
											(mean)
						21	0.078	0.16	< 0.01	< 0.01	0.24
							0.056	0.15	< 0.01	< 0.01	0.21
						28	0.051	0.20	< 0.01	< 0.01	0.25
							0.026	0.22	< 0.01	< 0.01	0.25
	1	73	410		Fruit	14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(soil						< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	drench)					21	< 0.01	0.078	< 0.01	< 0.01	0.088
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01 (mean)				
						28	< 0.01	0.13	< 0.01	< 0.01	0.14
							< 0.01	0.16	< 0.01	< 0.01	0.17
						35	< 0.01	0.14	< 0.01	< 0.01	0.15
							< 0.01	0.14	< 0.01	< 0.01	0.15
						42	< 0.01	0.14	< 0.01	< 0.01	0.15
							< 0.01	0.19	< 0.01	< 0.01	0.20
											0.18 (mean)
RV081-11DA, Abbotsford, British Columbia, USA, 2011 (Marketmore)	2	71	212	207	Fruit	0	0.24	0.063	< 0.01	< 0.01	0.30
	(6)	87	199	194			0.19	0.056	< 0.01	< 0.01	0.24
						1	0.16	0.063	< 0.01	< 0.01	0.23
							0.10	< 0.05	< 0.01	< 0.01	0.15
							0.13 (mean)				
						7	0.13	0.18	< 0.01	< 0.01	0.31

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							0.13	0.17	< 0.01	< 0.01	0.30
						14	0.12	0.24	< 0.01	< 0.01	0.36
							0.13	0.19	< 0.01	< 0.01	0.32
											0.34 (mean)
						21	0.081	0.32	< 0.01	< 0.01	0.40
							0.072	0.17	< 0.01	< 0.01	0.25
						28	0.091	0.26	< 0.01	< 0.01	0.35
							0.042	0.14	< 0.01	< 0.01	0.18
	1	85	409		Fruit	14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(soil						< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	drench)					21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01 (mean)				< 0.060 (mean)
						28	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						35	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						42	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 101 Residues from foliar and soil drench applications of flupyradifurone to summer squash in the USA and Canada (Beedle and Harbin 2012, RARVY021)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 9 Foliar	2 (7)		205			1					
GAP, USA, Crop Group 9 Soil	1		409			21					
RV090-11DA,	2	68	205	214	Fruit	0	0.11	< 0.05	< 0.01	< 0.01	0.16
North Rose, New	(7)	71	208	216			0.17	< 0.05	< 0.01	< 0.01	0.22
York,						1	0.081	0.052	< 0.01	< 0.01	0.13
USA, 2011							0.080	< 0.05	< 0.01	< 0.01	0.13
(Spineless Beauty)							0.081 (mean)				
						7	0.068	0.078	< 0.01	< 0.01	0.15
							0.052	0.14	< 0.01	< 0.01	0.20
						14	0.027	0.31	< 0.01	< 0.01	0.34
							0.053	0.28	< 0.01	< 0.01	0.34
						21	0.036	0.44	< 0.01	< 0.01	0.47
							0.014	0.35	< 0.01	< 0.01	0.36
						28	0.045	0.51	< 0.01	< 0.01	0.55
							0.032	0.63	< 0.01	< 0.01	0.66
											0.61 (mean)
	1	61	414		Fruit	14	0.18	0.26	0.01	< 0.01	0.44
	(soil						0.13	0.31	< 0.01	< 0.01	0.43
	drench)					21	0.095	0.44	< 0.01	< 0.01	0.53

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							0.019	0.32	< 0.01	< 0.01	0.34
							0.057 (mean)				
						28	0.045	0.58	< 0.01	< 0.01	0.62
							0.055	0.66	< 0.01	< 0.01	0.72
						35	0.029	0.65	< 0.01	< 0.01	0.68
							0.023	0.63	< 0.01	< 0.01	0.65
						42	0.036	1.3	< 0.01	< 0.01	1.3
							0.056	1.5	< 0.01	< 0.01	1.5
											1.4 (mean)
RV091-11HA, Seven Springs, North Carolina, USA, 2011 (Early Prolific)	2	51	215	165	Fruit	1	0.042	0.11	< 0.01	< 0.01	0.15
	(7)	71	208	168			0.053	0.12	< 0.01	< 0.01	0.17
							0.048 (mean)				0.16 (mean)
	1	27	410		Fruit	19	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(soil						< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	drench)						< 0.01 (mean)				< 0.060 (mean)
RV092-11HA, High Springs, Florida, USA, 2011 (Dixie)	2	74	200	228	Fruit	1	0.082	0.17	< 0.01	< 0.01	0.25
	(8)	78	201	221			0.068	0.13	< 0.01	< 0.01	0.20
							0.075 (mean)				0.22 (mean)
					Fruit	1	0.053 (av.)	0.12 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.175
					Fruit, cooked	1	0.039 (av.)	0.099 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.138
					Fruit, washed	1	0.048 (av.)	0.103 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.151
	1	56	410		Fruit	22	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(soil						< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	drenc						< 0.01				< 0.060
	h)						(mean)				(mean)
RV093-11DA, Stewardson, Illinois, USA, 2011 (Gold Dawn III)	2	71	212	239	Fruit	0	0.22	0.41	< 0.01	< 0.01	0.62
	(7)	72	209	227			0.16	0.27	< 0.01	< 0.01	0.43
						1	0.11	0.36	< 0.01	< 0.01	0.47
							0.086	0.38	< 0.01	< 0.01	0.46
							0.10 (mean)				
						7	0.020	0.80	< 0.01	< 0.01	0.82
							0.011	0.69	< 0.01	< 0.01	0.70
						14	< 0.01	0.76	< 0.01	< 0.01	0.77
							< 0.01	0.56	< 0.01	< 0.01	0.57
						21	< 0.01	0.67	< 0.01	< 0.01	0.68
							< 0.01	0.62	< 0.01	< 0.01	0.63
						28	0.012	1.3	< 0.01	< 0.01	1.3
							0.01	0.96	< 0.01	< 0.01	0.97
											1.1 (mean)
	1	71	411		Fruit	14	0.031	0.29	< 0.01	0.0145	0.33
	(soil						0.040	0.21	< 0.01	0.0121	0.26
	drenc					20	0.021	0.60	< 0.01	0.0176	0.63
	h)						0.018	0.46	< 0.01	0.0130	0.49
							0.020 (mean)				
						28	< 0.01	0.60	< 0.01	< 0.01	0.61

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							0.01	0.95	< 0.01	0.019	0.97
						34	< 0.01	1.1	< 0.01	0.0126	1.1
							< 0.01	1.1	< 0.01	0.019	1.1
											1.1 (mean)
						41	< 0.01	0.73	< 0.01	< 0.01	0.74
							< 0.01	0.51	< 0.01	< 0.01	0.52
RV094-11DA, York, Nebraska, USA, 2011 (Burpee Hybrid Zucchini)	2	72	208	196	Fruit	0	0.014	0.22	< 0.01	0.016	0.25
	(10)	74	207	193			0.019	0.29	< 0.01	0.013	0.33
						1	0.043	0.093 c0.05	< 0.01	< 0.01	0.14 c0.060
							0.021	0.12	< 0.01	< 0.01	0.14
							0.032 (mean)				0.14 (mean)
						7	0.016	0.27	< 0.01	< 0.01	0.29
							0.011	0.40	< 0.01	< 0.01	0.41
						14	< 0.01	0.50	< 0.01	< 0.01	0.51
							< 0.01	1.4	< 0.01	< 0.01	1.4
											0.95 (mean)
						21	< 0.01	1.1	< 0.01	< 0.01	1.1
							< 0.01	0.70	< 0.01	< 0.01	0.71
						28	< 0.01	0.72	< 0.01	< 0.01	0.73
							< 0.01	0.53	< 0.01	< 0.01	0.53
	1	61	408			14	0.012	0.34	< 0.01	< 0.01	0.35
	(soil						< 0.01	0.63	< 0.01	< 0.01	0.64
	drenc					20	0.016	0.26	< 0.01	< 0.01	0.28
	h)						< 0.01	0.34	< 0.01	< 0.01	0.35
						28	< 0.01	0.90	< 0.01	< 0.01	0.91
							< 0.01	1.3	< 0.01	< 0.01	1.3
											1.1 (mean)
						34	0.026	0.86	< 0.01	< 0.01	0.89
							0.021	0.74	< 0.01	< 0.01	0.76
							0.024 (mean)				
						41	< 0.01	0.40	< 0.01	< 0.01	0.41
							< 0.01	0.49	< 0.01	< 0.01	0.50
RV095-11HA, Campbell, Minnesota, USA, 2011 (Black Beauty)	2	64	205	187	Fruit	1	0.066	0.088	< 0.01	< 0.01	0.15
	(9)	71	205	187			0.043	0.082	< 0.01	< 0.01	0.12
							0.055 (mean)				0.14 (mean)
					Fruit	1	0.053 (av.)	0.074 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.127
					Fruit, cooked	1	0.01 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.060
					Fruit, washed	1	0.023 (av.)	0.051 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.074
	1	56	412		Fruit	19	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(soil						< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	drench)						< 0.01 (mean)				< 0.060 (mean)
RV096-11DA,	2	59	209	187	Fruit	0	0.031	< 0.05	< 0.01	0.012	0.093
Orland,	(7)	71	205	186			0.080	< 0.05	< 0.01	< 0.01	0.13
California,						1	0.016	< 0.05	< 0.01	< 0.01	0.066

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
USA, 2011							0.033	0.10	< 0.01	< 0.01	0.14
(Golden Crook Neck)											
						5	0.039	< 0.05	< 0.01	< 0.01	0.089
							0.027	0.089	< 0.01	< 0.01	0.12
							0.033 (mean)				
						14	0.023	0.11	< 0.01	< 0.01	0.13
							0.020	0.091	< 0.01	< 0.01	0.11
						21	< 0.01	0.17	< 0.01	< 0.01	0.18
							< 0.01	0.23	< 0.01	< 0.01	0.24
											0.21
											(mean)
						28	< 0.01	0.18	< 0.01	< 0.01	0.19
							< 0.01	0.19	< 0.01	< 0.01	0.20
	1	56	410		Fruit	14	0.013	< 0.05	< 0.01	< 0.01	0.063
	(soil						0.034	< 0.05	< 0.01	< 0.01	0.084
	drenc					21	0.022	< 0.05	< 0.01	< 0.01	0.072
	h)						0.013	< 0.05	< 0.01	< 0.01	0.063
						28	0.029	0.11	< 0.01	< 0.01	0.14
							0.032	0.14	< 0.01	< 0.01	0.17
							0.031 (mean)				0.16 (mean)
						35	0.015	0.12	< 0.01	< 0.01	0.14
							0.018	0.12	< 0.01	0.011	0.15
						42	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV097-11HA, Abbotsford, British Columbia, Canada, 2011 (Black Beauty)	2	87	209	204	Fruit	1	0.061	< 0.05	< 0.01	< 0.01	0.11
	(6)	89	207	202			0.047	< 0.05	< 0.01	< 0.01	0.097
							0.054 (mean)				0.10 (mean)
					Fruit	1	0.051 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.101
					Fruit, cooked	1	0.028 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.078
					Fruit, washed	1	0.028 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.078
	1	63	408		Fruit	20	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(soil						< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	drenc						< 0.01				< 0.060
	h)						(mean)				(mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 102 Residues from foliar and soil drench applications of flupyradifurone to melons in the USA and Canada (Beedle and Harbin 2012, RARVY021)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 9 Foliar	2 (7)		205			1					

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 9 Soil	1		409			21					
RV084-11DA,	2	72	196	179	Fruit	0	0.22	< 0.05	< 0.01	< 0.01	0.27
Lime Springs, Iowa,	(6)	73	199	182			0.11	< 0.05	< 0.01	< 0.01	0.16
USA, 2011						1	0.15	< 0.05	< 0.01	< 0.01	0.20
(Yuma Grande F1)							0.14	< 0.05	< 0.01	< 0.01	0.19
							0.15 (mean)				0.20 (mean)
						7	0.061	< 0.05	< 0.01	< 0.01	0.11
							0.052	< 0.05	< 0.01	< 0.01	0.10
						14	0.068	< 0.05	< 0.01	< 0.01	0.12
							0.024	< 0.05	< 0.01	< 0.01	0.074
						21	0.041	0.059	< 0.01	< 0.01	0.10
							0.056	0.064	< 0.01	< 0.01	0.12
					Fruit	1	0.069 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.079 (av.)
					Pulp	1	0.012 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.062 (av.)
	1	69	409		Fruit	14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(soil						< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	drench)					21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01 (mean)				
						28	< 0.01	0.052	< 0.01	< 0.01	0.062
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						35	< 0.01	0.077	< 0.01	< 0.01	0.087
							< 0.01	0.060	< 0.01	< 0.01	0.070
						42	< 0.01	0.075	< 0.01	< 0.01	0.085
							< 0.01	0.068	< 0.01	< 0.01	0.078
											0.082 (mean)
					Fruit	21	< 0.01 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	< 0.060 (av.)
					Pulp	21	< 0.01 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	< 0.060 (av.)
RV086-11DA,	2	71	207	226	Fruit	0	0.15	< 0.05	< 0.01	< 0.01	0.20
Uvalde, Texas,	(7)	81	209	222			0.066	< 0.05	< 0.01	< 0.01	0.12
USA, 2011						1	0.062	< 0.05	< 0.01	< 0.01	0.11
(Rocket)							0.060	< 0.05	< 0.01	< 0.01	0.11
							0.061 (mean)				
						7	0.044	0.088	< 0.01	< 0.01	0.13
							0.032	0.070	< 0.01	< 0.01	0.10
						14	0.019	0.11	< 0.01	< 0.01	0.13
							0.019	0.14	< 0.01	< 0.01	0.16
						21	0.023	0.18	< 0.01	< 0.01	0.20
							0.034	0.24	< 0.01	< 0.01	0.27
						28	0.017	0.37	< 0.01	< 0.01	0.38
							0.016	0.35	< 0.01	< 0.01	0.37
											0.38 (mean)
					Fruit	1	0.051 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.101 (av.)
					Pulp	1	< 0.01 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	< 0.060 (av.)
	1	69	410		Fruit	13	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(soil						< 0.01	< 0.05	< 0.01	< 0.01	< 0.060

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
	drench)					20	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	0.054	< 0.01	< 0.01	0.064
						27	< 0.01	0.055	< 0.01	< 0.01	0.065
							0.014	0.064	< 0.01	< 0.01	0.078
							0.012 (mean)				
						34	< 0.01	0.084	< 0.01	< 0.01	0.094
							< 0.01	0.072	< 0.01	< 0.01	0.082
											0.088 (mean)
						41	< 0.01	0.086	< 0.01	< 0.01	0.096
							< 0.01	0.055	< 0.01	< 0.01	0.065
					Fruit	21	< 0.01 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	< 0.060 (av.)
					Pulp	21	< 0.01 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	< 0.060 (av.)
RV087-11DA, Madera, California, USA, 2011	2	89	207	235	Fruit	0	0.27	< 0.05	< 0.01	< 0.01	0.32
	(7)	89	212	246			0.098	< 0.05	< 0.01	< 0.01	0.15
						1	0.10	< 0.05	< 0.01	< 0.01	0.15
							0.11	< 0.05	< 0.01	< 0.01	0.16
							0.11 (mean)				
						7	0.097	0.10	< 0.01	0.013	0.21
							0.13	0.11	< 0.01	< 0.01	0.24
							0.11 (mean)				
						14	0.072	0.30	< 0.01	0.012	0.39
							0.12	0.34	< 0.01	0.018	0.47
						21	0.074	0.61	< 0.01	0.023	0.71
							0.056	0.57	< 0.01	0.025	0.65
											0.68 (mean)
						28	0.030	0.71	< 0.01	0.015	0.76
							0.022	0.26	< 0.01	0.018	0.30
					Fruit	1	0.25 (av.)	< 0.05 (av.)	< 0.01 (av.)	0.012 (av.)	0.31 (av.)
					Pulp	1	< 0.01 (av.)	< 0.05 (av.)	< 0.01 (av.)	0.015 (av.)	0.075 (av.)
	1 (soil drench)	71	410		Fruit	14	0.016	< 0.05	< 0.01	< 0.01	0.066
							0.017	0.11	< 0.01	< 0.01	0.13
						21	0.012	0.28	< 0.01	0.012	0.30
							0.015	0.37	< 0.01	0.020	0.40
						28	0.013	0.18	< 0.01	< 0.01	0.20
							0.020	0.17	< 0.01	< 0.01	0.19
							0.017 (mean)				
						35	< 0.01	0.21	< 0.01	< 0.01	0.22
							< 0.01	0.20	< 0.01	< 0.01	0.21
						42	< 0.01	0.41	< 0.01	< 0.01	0.42
							< 0.01	0.43	< 0.01	< 0.01	0.44
											0.43 (mean)
					Fruit	21	0.011 (av.)	0.32 (av.)	< 0.01 (av.)	0.025 (av.)	0.35 (av.)
					Pulp	21	< 0.01 (av.)	0.35 (av.)	< 0.01 (av.)	0.023 (av.)	0.38 (av.)
RV088-11DA,	2	75	206	234	Fruit	0	0.13	< 0.05	< 0.01	< 0.01	0.18

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Paso Robles,	(6)	75	204	234			0.068	< 0.05	< 0.01	< 0.01	0.12
California,						1	0.077	< 0.05	< 0.01	< 0.01	0.13
USA, 2010							0.099	< 0.05	< 0.01	< 0.01	0.15
(Top Mark)							0.088 (mean)				
						7	0.084	< 0.05	< 0.01	< 0.01	0.13
							0.044	0.051	< 0.01	< 0.01	0.095
						14	0.038	0.097	< 0.01	0.012	0.15
							0.034	0.085	< 0.01	< 0.01	0.12
						21	0.030	0.19	< 0.01	< 0.01	0.22
							0.042	0.19	< 0.01	< 0.01	0.23
											0.22 (mean)
						28	0.030	0.18	< 0.01	< 0.01	0.21
							0.026	0.19	< 0.01	< 0.01	0.22
					Fruit	1	0.13 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.18 (av.)
					Pulp	1	< 0.01 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	< 0.060 (av.)
	1	68	410		Fruit	14	< 0.01	0.12	< 0.01	< 0.01	0.13
	(soil						< 0.01	0.12	< 0.01	< 0.01	0.13
	drench)					21	< 0.01	0.56	< 0.01	< 0.01	0.57
							< 0.01	0.20	< 0.01	< 0.01	0.21
						27	< 0.01	0.39	< 0.01	< 0.01	0.40
							< 0.01	0.39	< 0.01	< 0.01	0.40
						34	< 0.01	1.2	< 0.01	< 0.01	1.2
							< 0.01	0.51	< 0.01	< 0.01	0.52
											0.87 (mean)
						41	0.013	0.59	< 0.01	< 0.01	0.60
							< 0.01	0.75	< 0.01	< 0.01	0.76
							0.012 (mean)				
					Fruit	21	< 0.01 (av.)	0.253 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.263 (av.)
					Pulp	21	< 0.01 (av.)	0.212 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.222 (av.)
RV089-11DA,	2	72	207	229	Fruit	0	0.27	< 0.05	< 0.01	< 0.01	0.32
Sanger,	(7)	75	204	190			0.18	< 0.05	< 0.01	< 0.01	0.23
California,						1	0.17	< 0.05	< 0.01	< 0.01	0.22
USA, 2011							0.20	< 0.05	< 0.01	< 0.01	0.25
(Oro Rico)							0.19 (mean)				
						7	0.11	0.11	< 0.01	0.011	0.23
							0.15	0.14	< 0.01	0.014	0.30
						14	0.078	0.28	< 0.01	0.024	0.38
							0.075	0.31	< 0.01	0.021	0.40
						21	0.048	0.32	< 0.01	0.025	0.40
							0.046	0.35	< 0.01	0.023	0.42
						28	0.036	0.48	< 0.01	0.019	0.53
							0.029	0.46	< 0.01	0.013	0.50
											0.52 (mean)
					Fruit	1	0.251 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.30 (av.)
					Pulp	1	< 0.01 (av.)	0.08 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.095 (av.)
	1	76	410			14	0.020	0.054	< 0.01	< 0.01	0.074

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
	(soil						0.018	0.058	< 0.01	< 0.01	0.076
	drench)					21	0.020	0.099	< 0.01	< 0.01	0.12
							0.023	0.087	< 0.01	< 0.01	0.11
						27	0.028	0.15	< 0.01	< 0.01	0.17
							0.024	0.14	< 0.01	< 0.01	0.16
						34	0.027	0.18	< 0.01	0.011	0.22
							0.028	0.17	< 0.01	< 0.01	0.20
							0.028 (mean)				
						41	0.014	0.18	< 0.01	< 0.01	0.19
							0.024	0.23	< 0.01	0.012	0.27
											0.23 (mean)
					Fruit	21	0.014 (av.)	0.215 (av.)	< 0.01 (av.)	0.013 (av.)	0.23 (av.)
					Pulp	21	0.026 (av.)	0.104 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.13 (av.)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Fruiting vegetables, other than Cucurbits

Supervised trials were carried out in the USA and Canada during the 2011 growing season on tomatoes (19 trials—Table 103), sweet peppers (10 trials—Table 104) and chilli peppers (four trials in the USA only—Table 105) (Miller and Helfrich 2012, RARVY022), with either two foliar applications or one soil drench application of a 200 g/L SL formulation. Applications were made with ground-based equipment. Adjuvant was added in all applications (NIS at 0.2% v/v, MSO at 0.25% v/v or a COC at 1.0% v/v).

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in tomatoes, sweet peppers and chilli peppers were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data were obtained for each analyte in each vegetable.

Table 103 Residues from foliar and soil drench applications of flupyradifurone to tomatoes in the USA and Canada (Miller and Helfrich 2012, RARVY022)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 8-10 Foliar	2 (7)		205			1					
GAP, USA, Crop Group 8-10 Soil	1		409			45					
RV098-11HA, North Rose, New York, USA, 2011 (Early Girl)	2	82	205	234	Fruit	–6	0.092	< 0.05	< 0.01	< 0.01	0.14
	(7)	84	205	234			0.091	< 0.05	< 0.01	< 0.01	0.14
						1	0.13	< 0.05	< 0.01	< 0.01	0.18
							0.14	< 0.05	< 0.01	< 0.01	0.19
							0.13 (mean)				0.18 (mean)
	1	61	416		Fruit	45	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(soil						< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	drench)						< 0.01				< 0.060

Trial No., Location, Year (Variety)	Application No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)	Sample	DALA	Residues as parent (mg/kg) Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							(mean)				(mean)
RV099-11HA,	2	79	206	188	Fruit	–6	0.069	< 0.05	< 0.01	< 0.01	0.12
Athens,	(7)	81	206	200			0.065	< 0.05	< 0.01	< 0.01	0.12
Georgia,						1	0.090	< 0.05	< 0.01	0.017	0.16
USA, 2011							0.085	< 0.05	< 0.01	0.015	0.15
(Celebrity)							0.088 (mean)				0.15 (mean)
	1	61	408		Fruit	45	< 0.01	0.29	< 0.01	0.022	0.33
	(soil						< 0.01	0.33	< 0.01	0.012	0.36
	drench)						< 0.01 (mean)				0.34 (mean)
RV100-11HA,	2	75	207	259	Fruit	–6	0.15	< 0.05	< 0.01	< 0.01	0.20
Greenville,	(7)	79	203	272			0.15	< 0.05	< 0.01	< 0.01	0.20
Florida,						1	0.20	< 0.05	< 0.01	0.021	0.27
USA, 2011							0.25	< 0.05	< 0.01	0.019	0.32
(6 02)							0.23 (mean)				0.30 (mean)
	1	56	410		Fruit	44	0.030	0.16	< 0.01	0.022	0.21
	(soil						0.028	0.15	< 0.01	0.012	0.19
	drench)						0.029 (mean)				0.20 (mean)
RV101-11HA,	2	77	203	280	Fruit	–6	0.043	< 0.05	< 0.01	< 0.01	0.09
Oviedo,	(7)	81	201	277			0.029	< 0.05	< 0.01	< 0.01	0.08
Florida						1	0.05	< 0.05	< 0.01	< 0.01	0.10
USA, 2011							0.064	< 0.05	< 0.01	0.013	0.13
(Beefmaster)							0.057 (mean)				0.11 (mean)
	1	63	403		Fruit	43	0.012	0.13	< 0.01	< 0.01	0.14
	(soil						0.011	0.15	< 0.01	< 0.01	0.16
	drench)						0.012 (mean)				0.15 (mean)
RV102-11HA,	2	73	206	141	Fruit	–5	0.057	< 0.05	< 0.01	< 0.01	0.11
Richland,	(6)	84	207	188			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Iowa,						1	0.098	< 0.05	< 0.01	< 0.01	0.15
USA, 2011							0.17	< 0.05	< 0.01	< 0.01	0.22
(Keepsake)							0.14 (mean)				0.19 (mean)
	1	55	408		Fruit	45	< 0.01	0.065	< 0.01	< 0.01	0.075
	(soil						< 0.01	0.072	< 0.01	< 0.01	0.082
	drench)						< 0.01				0.079
							(mean)				(mean)
RV103-11HA,	2	84	200	196	Fruit	–4	0.16	< 0.05	< 0.01	< 0.01	0.21
St George,	(5)	85	209	205			0.17	< 0.05	< 0.01	< 0.01	0.22
Ontario,						1	0.32	< 0.05	< 0.01	< 0.01	0.37
Canada, 2011							0.22	< 0.05	< 0.01	< 0.01	0.27
(TSH 28)							0.27 (mean)				0.32 (mean)
	1	69	410		Fruit	43	0.017	0.061	< 0.01	< 0.01	0.078
	(soil						0.012	< 0.05	< 0.01	< 0.01	0.062
	drench)						0.015 (mean)				0.070 (mean)
RV104-11HA,	2	81	202	980	Fruit	–5	0.082	< 0.05	< 0.01	< 0.01	0.13
Elm Creek,	(6)	82	205	100			0.071	< 0.05	< 0.01	< 0.01	0.12
Manitoba,						1	0.062	< 0.05	< 0.01	< 0.01	0.11
Canada, 2011							0.056	< 0.05	< 0.01	< 0.01	0.11
(Bush Beefsteak)							0.059 (mean)				0.11 (mean)
	1	51–60	410		Fruit	45	0.039	0.85	< 0.01	0.080	0.96

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
	(soil drench)						0.022	0.74	< 0.01	0.077	0.84
							0.031 (mean)				0.90 (mean)
RV105-11DA,	2	79	209	255	Fruit	–6	0.090	< 0.05	< 0.01	< 0.01	0.14
Stewardson,	(7)	81	209	240			0.054	< 0.05	< 0.01	< 0.01	0.1
Illinois,						0	0.083	< 0.05	< 0.01	< 0.01	0.13
USA, 2011							0.082	< 0.05	< 0.01	< 0.01	0.13
(Early Girl)						1	0.057	< 0.05	< 0.01	< 0.01	0.11
							0.11	< 0.05	< 0.01	< 0.01	0.16
							0.086 (mean)				
						7	0.046	< 0.05	< 0.01	0.01	0.11
							0.052	< 0.05	< 0.01	< 0.01	0.10
						14	0.026	0.063	< 0.01	< 0.01	0.089
							0.021	0.091	< 0.01	0.01	0.12
						21	0.020	0.14	< 0.01	< 0.01	0.16
							0.024	0.11	< 0.01	< 0.01	0.13
											0.15 (mean)
						27	0.022	0.11	< 0.01	< 0.01	0.13
							0.027	0.14	< 0.01	< 0.01	0.16
	1	53	410		Fruit	41	0.017	1.1	< 0.01	0.061	1.2
	(soil						0.013	1.0	< 0.01	0.028	1.0
	drench)					45	0.013	0.71	< 0.01	0.017	0.74
							0.011	0.75	< 0.01	0.018	0.78
						50	0.013	0.65	< 0.01	0.014	0.68
							0.013	0.79	< 0.01	0.012	0.82
							0.013 (mean)				
						59	< 0.01	0.73	< 0.01	< 0.01	0.74
							< 0.01	0.66	< 0.01	< 0.01	0.67
						70	< 0.01	0.81	< 0.01	< 0.01	0.82
							< 0.01	0.78	< 0.01	< 0.01	0.79
											0.81 (mean)
RV106-11HA, Lenexa, Kansas, USA, 2011 (Celebrity)	2	72	202	143	Fruit	–5	0.048	< 0.05	< 0.01	< 0.01	0.10
	(6)	89	209	147			0.065	< 0.05	< 0.01	< 0.01	0.12
						1	0.17	< 0.05	< 0.01	< 0.01	0.22
							0.11	< 0.05	< 0.01	< 0.01	0.16
							0.14 (mean)				0.19 (mean)
	1	62	409		Fruit	44	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(soil						< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	drench)						< 0.01				< 0.060
							(mean)				(mean)
RV107-11HA, Delevan, Wisconsin, USA, 2011 (Red Defender)	2	89	205	191	Fruit	–6	0.021	< 0.05	< 0.01	< 0.01	0.07
	(7)	89	205	196			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						1	0.05	< 0.05	< 0.01	< 0.01	0.10
							0.060	< 0.05	< 0.01	< 0.01	0.11
							0.055 (mean)				0.11 (mean)
	1	61	411		Fruit	45	0.011	< 0.05	< 0.01	< 0.01	0.061
	(soil						0.016	< 0.05	< 0.01	< 0.01	0.066
	drench)						0.014 (mean)				0.064 (mean)
RV108-11DA,	2	82	204	244	Fruit	–6	0.064	< 0.05	< 0.01	< 0.01	0.11
Carlyle,	(7)	83	207	249			0.098	< 0.05	< 0.01	< 0.01	0.15

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Illinois,						0	0.12	< 0.05	< 0.01	0.018	0.19
USA, 2011							0.085	0.060	< 0.01	< 0.01	0.15
(Jet Star)						1	0.055	< 0.05	< 0.01	0.021	0.12
							0.072	< 0.05	< 0.01	0.029	0.15
						7	0.054	0.070	< 0.01	0.053	0.18
							0.082	0.088	< 0.01	0.065	0.24
							0.068 (mean)				
						14	0.042	0.10	< 0.01	0.055	0.20
							0.049	0.11	< 0.01	0.071	0.23
											0.22 (mean)
						21	0.039	0.088	< 0.01	0.043	0.17
							0.034	0.090	< 0.01	0.039	0.16
						28	0.023	0.080	< 0.01	0.034	0.14
							0.029	0.085	< 0.01	0.029	0.14
	1	65	410		Fruit	38	0.013	0.75	< 0.01	0.015	0.78
	(soil						< 0.01	1.1	< 0.01	0.026	1.1
	drench)					45	0.01	0.93	< 0.01	0.019	0.96
							< 0.01	0.81	< 0.01	0.022	0.84
							0.01				
							(mean)				
						50	< 0.01	1.3	< 0.01	0.030	1.3
							< 0.01	0.79	< 0.01	0.027	0.83
											1.1 (mean)
						59	< 0.01	0.90	< 0.01	0.023	0.94
							< 0.01	0.64	< 0.01	0.017	0.67
						70	< 0.01	0.64	< 0.01	0.018	0.65
							< 0.01	0.35	< 0.01	< 0.01	0.36
RV109-11DA,	2	79	212	146	Fruit	–5	0.094	< 0.05	< 0.01	0.020	0.16
Rockwood,	(6)	79	197	144			0.10	< 0.05	< 0.01	< 0.01	0.15
Ontario,						0	0.21	0.064	< 0.01	0.047	0.32
Canada, 2011							0.16	0.051	< 0.01	0.059	0.27
(H2401)						1	0.097	< 0.05	< 0.01	0.046	0.19
							0.12	0.067	< 0.01	0.061	0.24
							0.11 (mean)				
						7	0.068	0.089	< 0.01	0.062	0.22
							0.099	0.14	< 0.01	0.088	0.33
											0.27 (mean)
						14	0.070	0.12	< 0.01	0.077	0.27
							0.05	0.094	< 0.01	0.048	0.19
						20	0.067	0.11	< 0.01	0.038	0.21
							0.046	0.071	< 0.01	0.029	0.15
						28	0.054	0.12	< 0.01	0.018	0.19
							0.036	0.076	< 0.01	0.011	0.12
	1	29	410		Fruit	40	0.011	0.15	< 0.01	< 0.01	0.16
	(soil						0.011	0.13	< 0.01	< 0.01	0.15
	drench)					45	0.013	0.20	< 0.01	0.020	0.23
							0.015	0.22	< 0.01	0.018	0.25
											0.24 (mean)
						49	< 0.01	0.16	< 0.01	0.017	0.18
							< 0.01	0.16	< 0.01	0.021	0.19
						60	0.014	0.18	< 0.01	0.011	0.21
							0.016	0.21	< 0.01	0.012	0.24

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							0.015 (mean)				
						68	0.013	0.21	< 0.01	< 0.01	0.22
							0.01	0.16	< 0.01	< 0.01	0.17
RV110-11DA,	2	88	205	187	Fruit	–6	0.17	< 0.05	< 0.01	< 0.01	0.22
Corning,	(7)	89	208	187			0.27	< 0.05	< 0.01	< 0.01	0.32
California,						0	0.41	< 0.05	< 0.01	0.01	0.47
USA, 2011							0.32	< 0.05	< 0.01	0.01	0.38
(Sun 6366)						1	0.41	< 0.05	< 0.01	< 0.01	0.46
							0.49	< 0.05	< 0.01	0.01	0.55
							0.45 (mean)				
						7	0.35	0.15	0.01	0.023	0.52
							0.28	0.084	< 0.01	0.013	0.38
						14	0.23	0.21	0.012	0.019	0.46
							0.40	0.18	0.020	0.025	0.60
											0.53 (mean)
						21	0.24	0.18	0.012	0.020	0.44
							0.25	0.21	0.013	0.021	0.48
						28	0.15	0.20	< 0.01	0.020	0.37
							0.29	0.35	0.017	0.026	0.67
	1	73	410		Fruit	40	0.047	0.088	< 0.01	< 0.01	0.14
	(soil						0.19	0.066	< 0.01	< 0.01	0.25
	drench)					45	0.05	0.21	< 0.01	0.016	0.27
							0.087	0.16	< 0.01	0.01	0.25
							0.069 (mean)				
						50	0.021	0.20	< 0.01	< 0.01	0.22
							0.043	0.19	< 0.01	0.012	0.24
						60	0.01	0.36	< 0.01	< 0.01	0.37
							0.020	0.44	< 0.01	0.011	0.47
						70	0.016	0.55	< 0.01	< 0.01	0.57
							0.021	1.0	< 0.01	0.023	1.1
											0.81 (mean)
RV111-11DA,	2	87	205	187	Fruit	–6	0.16	< 0.05	< 0.01	< 0.01	0.21
Kerman,	(7)	85	204	186			0.15	< 0.05	< 0.01	< 0.01	0.20
California,						0	0.42	< 0.05	< 0.01	< 0.01	0.47
USA, 2011							0.33	< 0.05	< 0.01	< 0.01	0.38
(AB3)						1	0.33	< 0.05	< 0.01	< 0.01	0.38
							0.26	< 0.05	< 0.01	< 0.01	0.31
						7	0.40	< 0.05	< 0.01	< 0.01	0.45
							0.22	< 0.05	< 0.01	< 0.01	0.27
							0.31 (mean)				
						14	0.29	0.056	< 0.01	< 0.01	0.35
							0.22	< 0.05	< 0.01	< 0.01	0.27
						21	0.33	0.15	0.016	0.017	0.50
							0.22	0.085	0.011	< 0.01	0.30
											0.40 (mean)
						28	0.19	0.19	0.012	0.020	0.39
							0.13	0.17	0.01	0.018	0.32
	1	71	400		Fruit	40	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(soil						< 0.01	0.058	< 0.01	< 0.01	0.068
	drench)					45	< 0.01	0.059	< 0.01	< 0.01	0.069

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01 (mean)				
						50	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	0.077	< 0.01	< 0.01	0.087
						60	< 0.01	0.085	< 0.01	< 0.01	0.095
							< 0.01	0.085	< 0.01	< 0.01	0.095
						70	< 0.01	0.12	< 0.01	< 0.01	0.13
							< 0.01	0.11	< 0.01	< 0.01	0.12
											0.13 (mean)
RV112-11HA, Paso Robles, California, USA, 2011 (Washington Cherry)	2 (6)	89	207	235	Fruit	–5	0.37	< 0.05	< 0.01	< 0.01	0.42
		89	205	233			0.30	< 0.05	< 0.01	< 0.01	0.35
						1	0.60	< 0.05	< 0.01	< 0.01	0.65
							0.54	< 0.05	< 0.01	< 0.01	0.59
							0.57 (mean)				0.62 (mean)
	1 (soil drench)	81	410		Fruit	44	0.012	0.18	< 0.01	< 0.01	0.19
							< 0.01	0.17	< 0.01	< 0.01	0.18
							0.011 (mean)				0.19 (mean)
RV113-11HA, Porterville, California, USA, 2011 (Roma AB2)	2 (6)	88	210	257	Fruit	–5	0.13	< 0.05	< 0.01	< 0.01	0.18
		89	202	250			0.12	< 0.05	< 0.01	< 0.01	0.17
						1	0.24	< 0.05	< 0.01	< 0.01	0.29
							0.33	< 0.05	< 0.01	< 0.01	0.38
							0.28 (mean)				0.33 (mean)
	1 (soil drench)	86	411		Fruit	44	< 0.01	0.15	< 0.01	< 0.01	0.16
							< 0.01	0.13	< 0.01	< 0.01	0.14
							< 0.01 (mean)				0.15 (mean)
RV114-11DA, Sanger, California, US, 2011 (Quali T-27)	2 (7)	81	202	192	Fruit	–6	0.12	< 0.05	< 0.01	< 0.01	0.17
		82	207	193			0.078	< 0.05	< 0.01	< 0.01	0.13
						0	0.23	< 0.05	< 0.01	0.021	0.30
							0.22	< 0.05	< 0.01	0.018	0.28
						1	0.17	< 0.05	< 0.01	0.019	0.23
							0.14	< 0.05	< 0.01	0.018	0.21
							0.15 (mean)				
						7	0.099	0.072	< 0.01	0.028	0.20
							0.070	0.064	< 0.01	0.020	0.15
						14	0.074	0.12	< 0.01	0.025	0.22
							0.085	0.16	< 0.01	0.038	0.28
						21	0.11	0.32	< 0.01	0.056	0.48
							0.062	0.26	< 0.01	0.039	0.36
						28	0.079	0.30	< 0.01	0.046	0.43
							0.096	0.35	< 0.01	0.043	0.49
											0.46 (mean)
	1 (soil drench)	54	411		Fruit	40	0.029	0.60	< 0.01	0.094	0.72
							0.017	0.35	< 0.01	0.065	0.44
						45	0.029	0.55	< 0.01	0.076	0.65
							0.033	0.56	< 0.01	0.085	0.68
						49	0.038	0.56	< 0.01	0.078	0.68
							0.029	0.58	< 0.01	0.076	0.69
							0.034 (mean)				0.68 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
						60	0.024	0.54	< 0.01	0.040	0.61
							0.028	0.48	< 0.01	0.040	0.54
						69	0.027	0.59	< 0.01	0.031	0.65
							0.025	0.59	< 0.01	0.032	0.65
RV115-11DA,	2	82	210	239	Fruit	–6	0.052	< 0.05	< 0.01	< 0.01	0.10
Madera,	(7)	89	206	239			0.072	< 0.05	< 0.01	< 0.01	0.12
California,						0	0.12	< 0.05	< 0.01	< 0.01	0.17
USA, 2011							0.080	< 0.05	< 0.01	< 0.01	0.13
(Quality 27)						1	0.12	< 0.05	< 0.01	< 0.01	0.17
							0.10	< 0.05	< 0.01	< 0.01	0.15
						7	0.19	< 0.05	< 0.01	0.01	0.25
							0.088	0.067	< 0.01	< 0.01	0.16
							0.14 (mean)				
						14	0.10	0.077	< 0.01	< 0.01	0.18
							0.10	0.077	< 0.01	0.017	0.19
						21	0.15	0.15	< 0.01	0.019	0.32
							0.13	0.13	< 0.01	0.01	0.26
											0.29 (mean)
						28	0.097	0.15	< 0.01	0.012	0.26
							0.064	0.16	< 0.01	0.011	0.23
	1	69	394		Fruit	40	< 0.01	0.077	< 0.01	< 0.01	0.09
	(soil						< 0.01	0.087	< 0.01	< 0.01	0.10
	drench)					45	< 0.01	0.059	< 0.01	< 0.01	0.07
							< 0.01	0.073	< 0.01	< 0.01	0.08
							< 0.01 (mean)				
						50	< 0.01	0.085	< 0.01	< 0.01	0.10
							< 0.01	0.082	< 0.01	< 0.01	0.09
						60	< 0.01	0.078	< 0.01	< 0.01	0.09
							< 0.01	0.095	< 0.01	0.013	0.12
											0.10 (mean)
						70	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV116-11DA,	2	83	205	187	Fruit	–6	0.18	< 0.05	< 0.01	0.013	0.24
Chico,	(7)	89	206	187			0.21	< 0.05	< 0.01	0.013	0.27
California,						0	0.44	< 0.05	< 0.01	0.011	0.50
USA, 2011							0.68	0.055	< 0.01	0.020	0.76
(Sun 6366)						1	0.28	< 0.05	< 0.01	< 0.01	0.33
							0.44	< 0.05	0.013	0.017	0.50
						7	0.88	0.16	0.020	0.043	1.1
							0.58	0.14	0.020	0.028	0.74
							0.73 (mean)				0.91 (mean)
						14	0.59	0.57	0.018	0.033	1.2
							0.33	0.21	0.013	0.030	0.56
						21	0.46	0.30	0.018	0.034	0.79
							0.30	0.23	0.013	0.041	0.57
						28	0.33	0.51	0.019	0.019	0.86
							0.21	0.60	0.017	0.023	0.82
	1	73	410		Fruit	40	0.18	0.50	0.011	0.031	0.71
	(soil						0.23	0.85	< 0.01	0.033	1.1
	drench)					45	0.12	0.68	< 0.01	0.01	0.81
							0.36	0.72	0.01	0.028	1.1
							0.24				

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							(mean)				
						50	0.086	0.92	< 0.01	< 0.01	1.0
							0.28	1.5	0.016	0.038	1.8
						60	0.28	1.7	0.017	0.070	2.1
							0.18	1.5	0.021	0.038	1.7
											1.9 (mean)
						70	0.11	1.1	0.012	0.031	1.3
							0.12	1.5	0.011	0.016	1.6

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 104 Residues from foliar and soil drench applications of flupyradifurone to sweet peppers in the USA and Canada (Miller and Helfrich 2012, RARVY022)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 8-10 Foliar	2 (7)		205			1					
GAP, USA, Crop Group 8-10 Soil	1		409			45					
RV117-11DA, Chula, Georgia, USA, 2011 (Ariststotle)	2	84	205	186	Fruit	0	0.091	< 0.05	< 0.01	< 0.01	0.14
	(6)	89	204	197			0.12	< 0.05	< 0.01	< 0.01	0.17
						1	0.083	< 0.05	< 0.01	< 0.01	0.13
							0.082	< 0.05	< 0.01	< 0.01	0.13
							0.083 (mean)				
						7	0.082	< 0.05	< 0.01	< 0.01	0.13
							0.066	< 0.05	< 0.01	< 0.01	0.12
						14	0.038	0.070	< 0.01	< 0.01	0.11
							0.052	0.079	< 0.01	< 0.01	0.13
						21	0.042	0.13	< 0.01	< 0.01	0.17
							0.033	0.13	< 0.01	< 0.01	0.16
											0.17 (mean)
						28	0.030	0.10	< 0.01	< 0.01	0.13
							0.024	0.12	< 0.01	< 0.01	0.14
	1	52	410		Fruit	40	< 0.01	0.063	< 0.01	< 0.01	0.073
							< 0.01	0.10	< 0.01	< 0.01	0.11
						45	< 0.01	0.096	< 0.01	< 0.01	0.11
							< 0.01	0.080	< 0.01	< 0.01	0.090
							< 0.01 (mean)				0.098 (mean)
						49	< 0.01	< 0.05	0.012	< 0.01	< 0.060
							< 0.01	0.063	< 0.01	< 0.01	0.073
						59	< 0.01	0.067	< 0.01	< 0.01	0.077
							< 0.01	0.085	< 0.01	< 0.01	0.095
						70	< 0.01	0.079	< 0.01	< 0.01	0.089
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV118-11HA, Greenville, Florida, USA, 2011	2	74	203	185	Fruit	1	0.11	< 0.05	< 0.01	< 0.01	0.16
	(7)	89	202	183			0.12	< 0.05	< 0.01	< 0.01	0.17
							0.12				0.17

Trial No., Location, Year (Variety)	Application No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)	Sample	DALA	Residues as parent (mg/kg) Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
(Aristotle)							(mean)				(mean)
	1	51	410		Fruit	44	0.033	0.15	< 0.01	< 0.01	0.18
							0.020	0.067	< 0.01	< 0.01	0.087
							0.027 (mean)				0.13 (mean)
RV119-11DA, Springfield, Nebraska, USA, 2011 (California Wonder)	2	87	205	134	Fruit	0	0.044	< 0.05	< 0.01	< 0.01	0.094
	(5)	89	204	137			0.061	< 0.05	< 0.01	< 0.01	0.11
						1	0.056	< 0.05	< 0.01	< 0.01	0.11
							0.045	< 0.05	< 0.01	< 0.01	0.10
							0.051 (mean)				
						6	0.024	< 0.05	< 0.01	< 0.01	0.074
							0.023	< 0.05	< 0.01	< 0.01	0.073
						13	0.030	0.064	< 0.01	< 0.01	0.094
							0.015	0.071	< 0.01	< 0.01	0.086
						20	0.017	0.098	< 0.01	< 0.01	0.12
							0.012	0.084	< 0.01	< 0.01	0.096
						27	0.048	0.17	< 0.01	< 0.01	0.22
							0.033	0.14	< 0.01	< 0.01	0.18
											0.20 (mean)
	1	52	410		Fruit	40	< 0.01	0.11	< 0.01	< 0.01	0.12
							0.012	0.076	< 0.01	< 0.01	0.088
						44	< 0.01	0.11	< 0.01	< 0.01	0.12
							< 0.01	0.097	< 0.01	< 0.01	0.11
						48	< 0.01	0.11	< 0.01	< 0.01	0.12
							0.012	0.13	< 0.01	< 0.01	0.14
							0.011 (mean)				
						59	< 0.01	0.13	< 0.01	< 0.01	0.14
							0.01	0.13	< 0.01	< 0.01	0.14
						68	< 0.01	0.15	< 0.01	< 0.01	0.16
							< 0.01	0.15	< 0.01	< 0.01	0.16
											0.16 (mean)
RV120-11DA, Stewardson, Illinois, USA, 2011 (Better Bell)	2	72	211	239	Fruit	0	0.11	< 0.05	< 0.01	< 0.01	0.16
	(5)	74	205	250			0.089	< 0.05	< 0.01	< 0.01	0.14
						1	0.12	< 0.05	< 0.01	< 0.01	0.17
							0.13	< 0.05	< 0.01	< 0.01	0.18
							0.12 (mean)				
						7	0.094	0.083	< 0.01	< 0.01	0.18
							0.085	0.098	< 0.01	< 0.01	0.18
						14	0.035	0.17	< 0.01	< 0.01	0.20
							0.046	0.22	< 0.01	< 0.01	0.27
						21	0.032	0.26	< 0.01	< 0.01	0.29
							0.017	0.23	< 0.01	< 0.01	0.25
						28	< 0.01	0.20	< 0.01	< 0.01	0.21
							0.018	0.30	< 0.01	< 0.01	0.31
											0.26 (mean)
	1	55	410		Fruit	41	0.033	0.25	< 0.01	< 0.01	0.29
							0.037	0.19	< 0.01	< 0.01	0.23
						45	0.041	0.25	< 0.01	< 0.01	0.29
							0.028	0.34	< 0.01	< 0.01	0.37
							0.035 (mean)				
						50	0.029	0.55	< 0.01	< 0.01	0.58

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							0.025	0.44	< 0.01	< 0.01	0.47
											0.52 (mean)
						59	0.012	0.33	< 0.01	< 0.01	0.34
							0.017	0.29	< 0.01	< 0.01	0.31
						70	0.016	0.21	< 0.01	< 0.01	0.22
							< 0.01	0.29	< 0.01	< 0.01	0.30
RV121-11HA, Rockwood, Ontario, Canada, 2011 (California Wonder)	2	87	209	144	Fruit	1	0.088	< 0.05	< 0.01	< 0.01	0.14
	(6)	87	202	148			0.085	< 0.05	< 0.01	< 0.01	0.14
							0.087 (mean)				0.14 (mean)
	1	51	410		Fruit	45	0.011	0.075	< 0.01	< 0.01	0.086
							0.015	0.10	< 0.01	< 0.01	0.12
							0.013 (mean)				0.10 (mean)
RV122-11DA, Delavan, Wisconsin, USA, 2011 (California Wonder)	2	89	206	192	Fruit	0	0.028	< 0.05	< 0.01	< 0.01	0.078
	(7)	89	205	207			0.021	< 0.05	< 0.01	< 0.01	0.071
						1	0.043	< 0.05	< 0.01	< 0.01	0.093
							0.016	< 0.05	< 0.01	< 0.01	0.066
							0.030 (mean)				
						7	0.025	0.070	< 0.01	< 0.01	0.095
							0.019	0.069	< 0.01	< 0.01	0.088
						14	0.023	0.087	< 0.01	< 0.01	0.11
							0.018	0.085	< 0.01	< 0.01	0.10
											0.11 (mean)
						21	0.012	0.10	< 0.01	< 0.01	0.12
							0.020	0.073	< 0.01	< 0.01	0.093
						28	0.016	0.085	< 0.01	< 0.01	0.10
							0.013	0.065	< 0.01	< 0.01	0.078
	1	65	411		Fruit	40	< 0.01	0.084	< 0.01	< 0.01	0.094
							0.013	0.099	< 0.01	< 0.01	0.11
						45	0.012	0.086	< 0.01	< 0.01	0.098
							< 0.01	0.067	< 0.01	< 0.01	0.077
							0.011 (mean)				
						50	< 0.01	0.096	< 0.01	< 0.01	0.11
							< 0.01	0.12	< 0.01	< 0.01	0.13
						60	< 0.01	0.13	< 0.01	< 0.01	0.14
							< 0.01	0.12	< 0.01	< 0.01	0.13
											0.13 (mean)
						70	< 0.01	0.11	< 0.01	< 0.01	0.12
							< 0.01	0.11	< 0.01	< 0.01	0.12
RV123-11DA, Elm Creek, Manitoba, Canada, 2011 (Speedway)	2	87	200	98	Fruit	0	0.37	< 0.05	< 0.01	< 0.01	0.42
	(7)	89	198	97			0.22	< 0.05	< 0.01	< 0.01	0.27
						1	0.20	< 0.05	< 0.01	< 0.01	0.25
							0.34	< 0.05	< 0.01	< 0.01	0.39
						7	0.35	0.053	0.017	< 0.01	0.40
							0.26	0.052	0.013	< 0.01	0.31
							0.30 (mean)				0.35 (mean)
						13	0.15	0.078	0.013	< 0.01	0.23
							0.17	0.066	0.012	< 0.01	0.23
						20	0.17	0.10	0.015	< 0.01	0.27
							0.13	0.090	0.011	< 0.01	0.22
						28	0.12	0.11	0.013	< 0.01	0.23

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							0.095	0.12	< 0.01	< 0.01	0.21
	1	51–60	410		Fruit	42	0.25	1.3	0.045	0.014	1.5
							0.25	1.7	0.040	0.019	1.9
						45	0.18	1.5	0.032	0.012	1.7
							0.19	1.4	0.034	0.013	1.6
							0.18 (mean)				1.6 (mean)
						50	0.069	0.68	0.011	0.01	0.76
							0.12	1.4	0.021	0.015	1.6
						59	0.077	0.79	0.014	0.01	0.88
							0.075	0.85	0.014	< 0.01	0.93
						69	0.056	0.71	< 0.01	< 0.01	0.77
							0.046	0.61	< 0.01	< 0.01	0.66
RV124-11DA, Uvalde, Texas, USA, 2011 (Taurus)	2	85	205	170	Fruit	0	0.15	< 0.05	< 0.01	< 0.01	0.20
	(5)	84	204	154			0.11	< 0.05	< 0.01	< 0.01	0.16
						1	0.083	< 0.05	< 0.01	< 0.01	0.13
							0.057	< 0.05	< 0.01	< 0.01	0.11
							0.070 (mean)				
						7	0.061	< 0.05	< 0.01	< 0.01	0.11
							0.057	< 0.05	< 0.01	< 0.01	0.11
						14	0.029	0.091	< 0.01	< 0.01	0.12
							0.05	0.051	< 0.01	< 0.01	0.10
						21	0.011	0.10	< 0.01	< 0.01	0.11
							0.018	0.13	< 0.01	< 0.01	0.14
						28	0.024	0.12	< 0.01	< 0.01	0.14
							0.025	0.27	< 0.01	< 0.01	0.29
											0.22 (mean)
	1	81	409		Fruit	40	< 0.01	0.32	< 0.01	< 0.01	0.33
							< 0.01	0.34	< 0.01	< 0.01	0.35
						45	< 0.01	0.12	< 0.01	< 0.01	0.13
							< 0.01	0.13	< 0.01	< 0.01	0.14
							< 0.01 (mean)				0.14 (mean)
						49	< 0.01	0.10	< 0.01	< 0.01	0.11
							< 0.01	0.13	< 0.01	< 0.01	0.14
						63	< 0.01	0.12	< 0.01	< 0.01	0.13
							< 0.01	0.069	< 0.01	< 0.01	0.079
						70	< 0.01	0.064	< 0.01	< 0.01	0.074
							< 0.01	0.081	< 0.01	< 0.01	0.091
RV125-11DA, Madera, California, USA, 2011 (Cyprus)	2	83	211	240	Fruit	0	0.21	0.083	< 0.01	< 0.01	0.29
	(7)	89	207	238			0.24	0.088	< 0.01	< 0.01	0.33
						1	0.18	< 0.05	< 0.01	< 0.01	0.23
							0.24	< 0.05	< 0.01	< 0.01	0.29
						7	0.32	< 0.05	0.021	< 0.01	0.37
							0.26	< 0.05	0.015	< 0.01	0.31
							0.29 (mean)				
						14	0.12	< 0.05	0.01	< 0.01	0.17
							0.12	< 0.05	< 0.01	< 0.01	0.17
						21	0.12	0.084	0.013	< 0.01	0.20
							0.14	0.26	0.015	< 0.01	0.40
						28	0.096	0.33	< 0.01	< 0.01	0.43
							0.11	0.34	0.013	< 0.01	0.46
											0.44 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
	1	68	394			40	0.011	0.13	< 0.01	< 0.01	0.14
							< 0.01	0.14	< 0.01	< 0.01	0.15
						45	< 0.01	0.11	< 0.01	< 0.01	0.12
							0.012	0.14	< 0.01	< 0.01	0.15
							0.011 (mean)				0.13 (mean)
						50	< 0.01	0.12	< 0.01	< 0.01	0.13
							< 0.01	0.068	< 0.01	< 0.01	0.078
						60	< 0.01	0.065	< 0.01	< 0.01	0.075
							< 0.01	0.079	< 0.01	< 0.01	0.089
						70	< 0.01	0.086	< 0.01	< 0.01	0.096
							< 0.01	0.086	< 0.01	< 0.01	0.096
RV126-11DA, Kerman, California, USA, 2011 (Red)	2	87	206	187	Fruit	0	0.55	< 0.05	< 0.01	< 0.01	0.60
	(7)	87	206	187			0.48	< 0.05	< 0.01	< 0.01	0.53
						1	0.55	< 0.05	< 0.01	< 0.01	0.60
							0.40	< 0.05	< 0.01	< 0.01	0.45
							0.47 (mean)				0.52 (mean)
						7	0.29	< 0.05	0.011	< 0.01	0.34
							0.33	< 0.05	0.012	< 0.01	0.38
						14	0.35	0.085	0.017	< 0.01	0.43
							0.24	0.085	0.013	< 0.01	0.33
						21	0.24	0.26	0.018	< 0.01	0.50
							0.18	0.20	0.015	< 0.01	0.38
						28	0.051	0.32	< 0.01	< 0.01	0.37
							0.069	0.26	< 0.01	< 0.01	0.33
	1	71	402			40	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						45	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	0.069	< 0.01	< 0.01	0.079
							< 0.01 (mean)				
						50	< 0.01	0.060	< 0.01	< 0.01	0.070
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						60	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						70	< 0.01	0.072	< 0.01	< 0.01	0.082
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
											0.071 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 105 Residues from foliar and soil drench applications of flupyradifurone to chilli peppers in the USA and Canada (Miller and Helfrich 2012, RARVY022)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 8-10 Foliar	2 (7)		205			1					
GAP, USA, Crop Group 8-10	1		409			45					

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Soil											
RV127-11DA, Richland, Iowa, USA, 2011 (Early Jalapeno)	2	76	207	141	Fruit	0	0.084	< 0.05	< 0.01	< 0.01	0.13
	(7)	89	209	204			0.19	< 0.05	< 0.01	< 0.01	0.24
						1	0.095	< 0.05	< 0.01	< 0.01	0.15
							0.078	< 0.05	< 0.01	< 0.01	0.13
						7	0.057	0.053	< 0.01	< 0.01	0.11
							0.078	0.057	< 0.01	< 0.01	0.14
						14	0.12	0.15	< 0.01	< 0.01	0.27
							0.12	0.093	< 0.01	< 0.01	0.21
							0.12 (mean)				
						21	0.11	0.25	< 0.01	< 0.01	0.35
							0.085	0.26	< 0.01	< 0.01	0.34
						28	0.059	0.34	< 0.01	< 0.01	0.40
							0.071	0.31	< 0.01	< 0.01	0.38
											0.39 (mean)
	1	64	408		Fruit	39	0.023	0.27	< 0.01	< 0.01	0.29
							0.020	0.16	< 0.01	< 0.01	0.18
						45	0.024	0.36	< 0.01	0.01	0.39
							0.021	0.46	< 0.01	< 0.01	0.48
						50	0.019	0.45	< 0.01	< 0.01	0.46
							0.020	0.46	< 0.01	< 0.01	0.48
						60	0.023	0.66	< 0.01	< 0.01	0.68
							< 0.01	0.30	< 0.01	< 0.01	0.31
						70	0.021	0.80	< 0.01	0.021	0.84
							0.027	0.95	< 0.01	0.014	0.99
							0.024 (mean)				0.92 (mean)
RV128-11HA, Levelland, Texas, USA, 2011 ("M")	2	89	211	191	Fruit	1	0.36	< 0.05	< 0.01	< 0.01	0.41
	(7)	89	207	187			0.38	< 0.05	< 0.01	< 0.01	0.43
							0.37 (mean)				0.42 (mean)
					Fruit,	1	0.81	0.080	0.046	0.01	0.90
					dried		0.89	0.10	0.045	< 0.01	0.99
							0.85 (mean)				0.94 (mean)
	1	59	415		Fruit	43	0.048	0.30	< 0.01	< 0.01	0.35
							0.046	0.33	< 0.01	< 0.01	0.37
							0.047 (mean)				0.36 (mean)
					Fruit,	43	0.18	0.98	0.024	< 0.01	1.2
					dried		0.16	1.0	0.019	< 0.01	1.2
							0.17 (mean)				1.2 (mean)
RV129-11HA, Jerome, Idaho, USA, 2011 (Jalapenos)	2	78	210	213	Fruit	1	0.083	< 0.05	< 0.01	< 0.01	0.13
	(7)	89	212	211			0.063	< 0.05	< 0.01	< 0.01	0.11
							0.073 (mean)				0.12 (mean)
					Fruit,	1	0.44	0.17	0.014	< 0.01	0.61

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
					dried		0.84	0.16	0.030	< 0.01	1.0
							0.64 (mean)				0.81 (mean)
	1	64	419		Fruit	44	< 0.01	0.17	< 0.01	< 0.01	0.18
							< 0.01	0.14	< 0.01	< 0.01	0.15
							< 0.01 (mean)				0.17 (mean)
					Fruit, dried	44	< 0.01	1.1	< 0.01	0.017	1.2
							< 0.01	1.1	< 0.01	0.018	1.1
							< 0.01 (mean)				1.1 (mean)
RV130-11DA, Porterville, California, USA, 2011 (Fresno Chilli)	2	87	211	255	Fruit	0	0.46	< 0.05	< 0.01	< 0.01	0.51
	(7)	89	204	298			0.37	< 0.05	< 0.01	< 0.01	0.42
						1	0.48	< 0.05	0.012	< 0.01	0.53
							0.58	< 0.05	0.014	< 0.01	0.63
							0.53 (mean)				
						7	0.23	0.05	0.017	< 0.01	0.28
							0.17	< 0.05	0.014	< 0.01	0.22
						14	0.22	0.14	0.031	< 0.01	0.36
							0.24	0.30	0.039	< 0.01	0.54
						21	0.14	0.67	0.040	< 0.01	0.81
							0.14	0.40	0.046	< 0.01	0.54
											0.68 (mean)
						28	0.14	0.30	0.036	< 0.01	0.44
							0.086	0.38	0.033	< 0.01	0.47
	1	39	409		Fruit	40	< 0.01	0.26	< 0.01	< 0.01	0.27
							< 0.01	0.37	< 0.01	< 0.01	0.38
						44	< 0.01	0.39	< 0.01	< 0.01	0.40
							< 0.01	0.36	< 0.01	< 0.01	0.37
							< 0.01 (mean)				
						49	< 0.01	0.44	< 0.01	< 0.01	0.45
							< 0.01	0.53	< 0.01	< 0.01	0.54
						60	< 0.01	0.86	< 0.01	< 0.01	0.87
							< 0.01	0.57	< 0.01	< 0.01	0.58
											0.72 (mean)
						70	< 0.01	0.79	< 0.01	< 0.01	0.80
							< 0.01	0.50	< 0.01	< 0.01	0.51

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Thirteen supervised trials were carried out on sweet corn (Table 106) in the USA and Canada during the 2010 growing season (Fischer 2012b, RARVY002). Two foliar applications of a 200 g/L SL formulation were made. Three of the trials also included plots to measure the magnitude of BYI 02960 residues following the planting of seed treated with BYI 02960 480 FS. Applications were made with ground-based equipment. An adjuvant was added in all foliar applications at 0.25% v/v.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in sweet corn kernels and cob with husk removed and in sweet corn forage and stover were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data were obtained for each analyte in each vegetable.

Residues in sweet corn forage and stover are shown in Table 137.

Table 106 Residues from foliar and soil drench applications of flupyradifurone to sweet corn in the USA and Canada (Fischer 2012b, RARVY002)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 15	2 (7)		205			7					
RV041-10HA, Germansville, Pennsylvania USA, 2010 (Extra Tender)	2 (6)	71	208	260	Kernels and cob, husked	6	0.017	0.21	< 0.01	0.039	0.27
		73	208	260			0.019	0.24	< 0.01	0.036	0.29
							0.018 (mean)				0.28 (mean)
	1 (Seed)	00	115		Kernels and cob, husked	72	< 0.01	0.11	< 0.01	< 0.01	0.12
							< 0.01	0.10	< 0.01	< 0.01	0.11
RV042-10DA, North Rose, New York USA, 2010 (Serendipity)	2 (7)	71	207	330	Kernels and cob, husked	0	0.014	0.091	< 0.01	< 0.01	0.10
		75	207	330			0.016	0.081	< 0.01	< 0.01	0.097
						3	0.016	0.13	< 0.01	0.012	0.16
							0.017	0.13	< 0.01	0.011	0.16
						7	0.030	0.14	< 0.01	0.017	0.19
							0.023	0.15	< 0.01	0.012	0.18
							0.026 (mean)				
						14	0.018	0.19	< 0.01	0.013	0.22
							0.020	0.17	< 0.01	0.01	0.20
											0.21 (mean)
						21	0.016	0.21	< 0.01	< 0.01	0.22
							0.01	0.16	< 0.01	< 0.01	0.17
RV043-10HA, Sycamore, Georgia USA, 2010 (Bi-Color)	2 (7)	73	208	190	Kernels and cob, husked	7	< 0.01	0.14	< 0.01	< 0.01	0.15
		75	207	190			< 0.01	0.087	< 0.01	< 0.01	0.097
							< 0.01(mean)				0.13 (mean)
RV044-10HA, High Springs, Florida USA, 2010 (Obsession)	2 (7)	65	201	240	Kernels and cob, husked	7	< 0.01	0.11	< 0.01	< 0.01	0.12
		73	204	240			< 0.01	0.11	< 0.01	< 0.01	0.12
							< 0.01(mean)				0.12 (mean)
RV045-10HA, Richland, Iowa USA, 2010 (Augusta)	2 (5)	71	207	180	Kernels and cob, husked	7	< 0.01	0.11	< 0.01	0.015	0.13
		71	205	160			< 0.01	0.12	< 0.01	< 0.01	0.13
							< 0.01(mean)				0.13 (mean)
	1 (Seed)	00	93		Kernels and cob, husked	76	< 0.01	0.061	< 0.01	< 0.01	0.071
							< 0.01	0.067	< 0.01	< 0.01	0.077
RV046-10HA, Stewardson, Illinois USA, 2010 (XTRA-tender 274A)	2 (7)	67	206	310	Kernels and cob, husked	7	< 0.01	0.17	< 0.01	0.012	0.19
		71	201	320			< 0.01	0.17	< 0.01	0.012	0.19
							< 0.01(mean)				0.19 (mean)
RV047-10HA, Rockwood, Ontario Canada, 2010 (Brocade TSW)	2 (7)	79	202	110	Kernels and cob, husked	7	< 0.01	0.089	< 0.01	< 0.01	0.099
		79	205	110			< 0.01	0.088	< 0.01	< 0.01	0.098
							< 0.01(mean)				0.099 (mean)
RV048-10HA,	2	63	207	190	Kernels	7	0.047	0.14	< 0.01	0.01	0.20

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Osceola, Nebraska USA, 2010 (Augusta)	(6)	69	206	190	and cob, husked		0.028	0.083	< 0.01	< 0.01	0.11
							0.038 (mean)				0.15 (mean)
	1 (Seed)	00	85		Kernels and cob, husked	73	0.012	0.099	< 0.01	< 0.01	0.11
							0.013	0.12	< 0.01	< 0.01	0.13
RV049-10DA, York, Nebraska USA, 2010 (Xtra-Tender 278A)	2 (7)	63 71	207 205	190 190	Kernels and cob, husked	0	0.022	0.051	< 0.01	< 0.01	0.073
							0.019	0.058	< 0.01	< 0.01	0.08
						3	0.014	0.10	< 0.01	< 0.01	0.12
							0.013	0.093	< 0.01	< 0.01	0.11
						7	0.017	0.19	< 0.01	< 0.01	0.21
							0.014	0.17	< 0.01	< 0.01	0.18
							0.016 (mean)				
						14	0.011	0.23	< 0.01	< 0.01	0.24
							0.015	0.25	< 0.01	< 0.01	0.26
											0.25 (mean)
						21	< 0.01	0.23	< 0.01	< 0.01	0.24
							0.01	0.25	< 0.01	< 0.01	0.26
RV050-10HA, Outlook, Saskatchewan Canada, 2010 (Jackpot)	2 (7)	71 71	205 207	200 200	Kernels and cob, husked	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01 (mean)				< 0.060 (mean)
RV051-10HA, Sanger, California USA, 2010 (Golden Queen)	2 (6)	71 75	206 209	360 270	Kernels and cob, husked	5	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01 (mean)				< 0.060 (mean)
RV052-10HA, Jerome, Idaho USA, 2010 (Jackpot)	2 (7)	71 75	208 207	190 180	Kernels and cob, husked	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01 (mean)				< 0.060 (mean)
RV053-10HA, Portland, Oregon USA, 2010 (Serendipity)	2 (7)	73 75	211 208	200 190	Kernels and cob, husked	7	< 0.01	0.051	< 0.01	< 0.01	0.061
							< 0.01	0.065	< 0.01	< 0.01	0.074
							< 0.01 (mean)				0.068 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Leafy vegetables (including Brassica leafy vegetables)

Eight supervised trials were carried out on mustard greens (Table 107) in the USA during the 2010 growing season (Netzband and Helrich 2012, RARVY042) to measure the magnitude of residues following two broadcast foliar spray applications of BYI 02960 200 SL. Residue reduction samples were collected in the decline trial and processed into mustard green, washed mustard green and cooked mustard green samples. All applications were made using ground-based equipment. An adjuvant was used in all applications (0.25% v/v).

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in mustard greens were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data were obtained for all analytes.

Table 107 Residues from foliar applications of flupyradifurone to mustard greens in the USA (Netzband and Helrich 2012, RARVY042)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 5	2 (7)		205			1					
RV278-10HA, Suffolk, Virginia, USA, 2010	2	46	211	124	Leaf	–2	4.4	0.19	0.035	0.042	4.7
	(5)	46	210	115			4.0	0.18	0.028	0.037	4.2
						1	9.9	0.39	0.065	0.069	10
							12	0.43	0.072	0.070	13
(Southern Giant Curled)							11 (mean)				12 (mean)
						3	8.0	0.47	0.075	0.071	8.5
							7.5	0.54	0.084	0.10	8.1
RV279-10HA, Chula, Georgia, USA, 2010 (Southern Giant Curled)	2	45	206	222	Leaf	–4	4.4	< 0.05	0.037	0.017	4.4
	(7)	46	204	215			4.4	< 0.05	0.024	0.012	4.5
						1	5.8	0.060	0.032	0.033	5.9
							6.3	0.072	0.029	0.035	6.4
							6.1 (mean)				6.2 (mean)
						3	3.5	0.10	0.027	0.037	3.6
							3.7	< 0.05	0.045	0.037	3.8
RV280-10HA, High Springs, Florida USA, 2010 (Southern Giant Curled	2	45	207	223	Leaf	–2	3.9	0.14	0.041	0.014	4.0
	(5)	46	207	220			3.8	0.18	0.030	0.018	4.0
						1	11	0.19	0.044	0.056	11
							9.8	0.22	0.039	0.052	10
							10 (mean)				11 (mean)
						3	4.9	0.33	0.042	0.055	5.2
							5.2	0.30	0.047	0.061	5.5
RV281-10HA, Cheneyville, Louisiana, USA, 2010 (Florida Broadleaf)	2	44	211	191	Leaf	–4	16	0.21	0.094	0.036	16
	(7)	48	206	185			17	0.20	0.14	0.036	17
						1	24	0.29	0.10	0.048	25
							24	0.27	0.10	0.054	25
							24 (mean)				25 (mean)
						3	19	0.34	0.11	0.048	19
							15	0.23	0.067	0.051	16
RV282-10HA, Clarence, Missouri, USA, 2010 (Southern Giant Curled)	2	17	206	190	Leaf	–4	4.1	0.17	0.045	0.060	4.4
	(7)	45	201	188			3.8	0.13	0.031	0.026	3.9
						1	7.4	0.31	0.048	0.064	7.8
							7.2	0.26	0.046	0.057	7.5
							7.3 (mean)				7.7 (mean)
						3	4.0	0.36	0.036	0.075	4.4
							4.3	0.49	0.043	0.069	4.8
RV283-10HA,	2	46	205	178	Leaf	–4	8.7	< 0.05	0.052	0.041	8.8
Wall, Texas,	(7)	49	206	182			9.7	< 0.05	0.042	0.028	9.8
USA, 2010						1	18	0.071	0.060	0.059	18
(Florida Broadleaf)							17	0.069	0.069	0.016	18
							18 (mean)				18 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
						3	13	0.088	0.081	0.039	14
							12	< 0.05	0.063	0.040	12
RV284-10HA,	2	46	205	261	Leaf	–4	8.8	0.23	0.060	0.025	9.0
Sanger, California,	(7)	49	202	246			8.2	0.094	0.069	0.023	8.3
USA, 2010						1	15	0.20	0.077	0.05	15
(Florida Broadleaf)							14	0.30	0.081	0.048	15
							15 (mean)				15 (mean)
						3	11	0.18	0.10	0.041	11
							16	0.37	0.081	0.062	17
RV285-10DA,	2	47	204	279	Leaf	–2	7.5	0.057	0.049	0.014	7.5
Hughson, California	(5)	49	205	280			7.8	0.080	0.037	0.014	7.9
USA, 2010						0	14	0.085	0.065	0.031	14
(Florida Broadleaf)							13	0.068	0.043	0.054	13
						1	11	< 0.05	0.051	0.046	12
							13	0.072	0.056	0.048	13
							12 (mean)				12 (mean)
						3	7.5	0.13	0.057	0.039	7.7
							6.4	0.063	0.055	0.052	6.6
						7	6.2	0.083	0.062	0.048	6.3
							2.9	0.15	0.029	0.060	3.1
						14	0.75	0.27	0.01	0.093	1.1
							1.0	0.33	< 0.01	0.13	1.5
					Leaf	1	11 (av.)	< 0.05 (av.)	0.072 (av.)	0.035 (av.)	11
					Leaf, washed	1	8.1 (av.)	0.071 (av.)	0.055 (av.)	0.037 (av.)	8.3
					Leaf, cooked	1	2.1 (av.)	< 0.05 (av.)	0.017 (av.)	0.014 (av.)	2.2

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Supervised trials were carried out on spinach (nine trials—Table 108), head lettuce (eight trials—Table 109) and leaf lettuce (nine trials—Table 110) in the USA and Canada during the 2011–12 growing seasons (Netzband and Niczyporowicz 2012, RARVY005) to measure the magnitude of residues in spinach, and head and leaf lettuce following two broadcast foliar spray applications of BYI 02960 200 SL. All applications were made using ground-based equipment. Additional samples of lettuce heads without wrapper leaves were taken from the head lettuce trials. From three trials, additional spinach samples were taken to evaluate potential residue reduction from washing and washing and cooking spinach. An adjuvant [non-ionic surfactant at 0.20–0.25% v/v, methylated seed oil at 0.25% v/v or crop oil concentrate 1.0% v/v] was used in all applications.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in spinach, head lettuce and leaf lettuce were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data were obtained for all analytes.

Table 108 Residues from foliar applications of flupyradifurone to spinach in the USA and Canada (Netzband and Niczyporowicz 2012, RARVY005)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 4	2 (7)		205			1					
RV018-11HA, Alton, New York USA, 2011 (Space F1)	2	47	207	236	Leaf	–6	4.1	0.055	0.076	0.037	4.1
	(7)	49	205	232			4.1	0.063	0.076	0.041	4.3
						1	7.3	0.28	0.068	0.039	7.6
							6.1	0.32	0.051	0.044	6.4
							6.7 (mean)				7.0 (mean)
					Leaf	1	5.4 (av.)	0.46 (av.)	0.070 (av.)	0.074 (av.)	6.0 (av.)
					Leaf, washed	1	2.9 (av.)	0.25 (av.)	0.037 (av.)	0.030 (av.)	3.1 (av.)
					Leaf, cooked	1	1.8 (av.)	0.18 (av.)	0.014 (av.)	0.018 (av.)	1.9 (av.)
RV019-11DA, Elm Creek, Manitoba, Canada, 2011 (Vancouver)	2	51	207	101	Leaf	–6	14	< 0.05	0.059	0.020	14
	(7)	59	194	95			7.8	< 0.05	0.033	0.013	7.8
						0	16	0.73	0.11	0.056	17
							15	0.63	0.12	0.047	15
						1	8.7	0.50	0.11	0.055	9.3
							7.1	0.53	0.089	0.057	7.7
							7.9 (mean)				8.5 (mean)
						7	3.4	0.64	0.047	0.070	4.1
							5.1	0.96	0.075	0.11	6.2
						14	3.0	1.1	0.045	0.16	4.1
							2.7	1.4	0.035	0.17	4.3
						20	1.3	0.82	0.020	0.16	2.3
							1.3	1.1	0.018	0.19	2.5
RV020-11HA, Uvalde, Texas, USA, 2012 (DMC66-07)	2	47	204	186	Leaf	–6	5.2	< 0.05	0.015	< 0.01	5.2
	(7)	49	206	165			8.2	< 0.05	0.020	< 0.01	8.2
						1	2.1	0.11	0.022	0.012	2.2
							1.9	0.099	0.018	< 0.01	2.0
							2.0 (mean) *c0.01*				2.1 (mean)
					Leaf	1	1.4 (av.)	0.099 (av.)	0.020 (av.)	0.01 (av.)	1.5
					Leaf, washed	1	0.83 (av.)	0.051 (av.)	0.011 (av.)	< 0.01 (av.)	0.88
					Leaf, cooked	1	0.82 (av.)	< 0.05 (av.)	< 0.01 (av.)	< 0.01 (av.)	0.87
RV021-11DA, Arroyo Grande, California, USA, 2011 (Falcon)	2	45	204	187	Leaf	–6	12	< 0.05	0.044	< 0.01	12
	(7)	45	208	189			9.7	< 0.05	0.048	< 0.01	9.7
						0	21	0.082	0.087	< 0.01	22
							18	0.083	0.079	< 0.01	18
						1	18	0.084	0.10	< 0.01	19
							16	0.072	0.10	< 0.01	16
							17 (mean)				18 (mean)
						7	12	0.083	0.12	< 0.01	12
							11	0.12	0.14	0.012	12
						14	5.2	0.18	0.073	< 0.01	5.4
							5.4	0.12	0.066	< 0.01	5.6
						21	1.5	0.089	0.018	< 0.01	1.6

Trial No., Location, Year (Variety)	Application No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)	Sample	DALA	Residues as parent (mg/kg) Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							1.7	0.16	0.022	0.012	1.9
						27	0.73	0.096	0.01	< 0.01	0.83
							0.78	0.15	0.01	< 0.01	0.92
RV022-11DA,	2	45	203	227	Leaf	–6	3.4	< 0.05	0.011	< 0.01	3.5
Chula, Georgia,	(7)	49	209	217			5.5	< 0.05	0.019	< 0.01	5.5
USA, 2011						0	10	0.051	0.035	< 0.01	10
(Vancouver)							12	0.058	0.032	< 0.01	12
						1	3.8	0.05	0.030	< 0.01	3.9
							3.7	0.064	0.040	< 0.01	3.8
							3.8 (mean)				3.8 (mean)
						6	1.5	0.081	0.043	0.011	1.6
							2.2	0.15	0.044	< 0.01	2.3
						14	0.82	0.20	0.011	< 0.01	1.0
							0.59	0.21	0.012	< 0.01	0.80
						20	0.36	0.29	< 0.01	< 0.01	0.65
							0.37	0.36	< 0.01	< 0.01	0.73
						26	0.20	0.28	< 0.01	< 0.01	0.48
							0.24	0.33	< 0.01	< 0.01	0.57
RV023-11DA, Portage la Prairie, Manitoba, Canada, 2011 (Longstand	2	45	207	176	Leaf	–6	12	< 0.05	0.062	< 0.01	12
	(7)	47	202	172			14	< 0.05	0.074	< 0.01	14
						0	19	0.060	0.074	< 0.01	19
							14	< 0.05	0.070	< 0.01	14
						1	11	0.055	0.082	< 0.01	11
Bloomsdale)							8.9	< 0.05	0.060	< 0.01	8.9
							9.8 (mean)				9.9 (mean)
						6	3.3	0.13	0.099	< 0.01	3.4
							2.9	0.099	0.095	< 0.01	3.0
						13	0.84	0.16	0.034	< 0.01	1.0
							0.94	0.18	0.039	0.012	1.1
						20	0.56	0.22	0.018	0.035	0.81
							0.35	0.19	0.012	0.021	0.55
						27	0.11	0.11	< 0.01	0.034	0.25
							0.079	0.093	< 0.01	0.020	0.19
RV024-11HA,	2	47	206	114	Leaf	–5	5.1	< 0.05	< 0.01	< 0.01	5.2
Eaton, Colorado,	(6)	49	209	123			6.2	< 0.05	0.012	< 0.01	6.3
USA, 2011						1	5.9	0.083	0.029	0.012	6.0
(Emu F1)							6.9	0.097	0.039	0.014	7.0
							6.4 (mean)				6.5 (mean)
RV025-11HA, Paso Robles, California, USA, 2011 (Emelia)	2	47	206	187	Leaf	–6	12	0.21	0.15	0.023	13
	(7)	47	204	186			12	0.23	0.15	0.034	12
						1	9.4	< 0.05	0.052	0.012	9.4
							8.3	< 0.05	0.047	< 0.01	8.3
							8.8 (mean)				8.9 (mean)
					Leaf	1	12 (av.)	0.50 (av.) c0.057	0.11 (av.)	0.016 (av.)	13
					Leaf, washed	1	7.8 (av.)	0.44 (av.) *c0.21*	0.090 (av.)	< 0.01 (av.)	8.3
					Leaf, cooked	1	6.6 (av.)	0.34 (av.) c 0.17	0.072 (av.)	< 0.01 (av.)	7.0
RV026-11HA,	2	47	209	202	Head	–6	12	< 0.05	0.057	< 0.01	12

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Abbotsford, British Columbia, Canada, 2011 (Unipack)	(7)	49	200	194			11	< 0.05	0.044	0.013	11
						1	18	0.16	0.19	0.016	18
							16	0.18	0.18	0.023	16
							17 (mean)				17 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 109 Residues from foliar applications of flupyradifurone to head lettuce in the USA and Canada (Netzband and Niczyporowicz 2012, RARVY005)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 4	2 (7)		205			1					
RV010-11HA, Oviedo, Florida, USA, 2011 (Great Lakes)	2	47	206	280	Head	–6	0.14	< 0.05	< 0.01	< 0.01	0.19
	(7)	49	201	274			0.19	< 0.05	< 0.01	< 0.01	0.24
						1	2.3	< 0.05	0.016	0.030	2.4
							2.3	< 0.05	0.020	0.030, 0.041	2.4
							2.3 (mean)				2.4 (mean)
					Head,	–6	0.11	< 0.05	< 0.01	< 0.01	0.16
					w/o		0.13	< 0.05	< 0.01	< 0.01	0.18
					wrapper	1	0.93	< 0.05	0.012	0.022	1.0
					leaves		0.78	< 0.05	< 0.01	0.022	0.85
											0.93 (mean)
RV011-11HA, Rockwood, Ontario, Canada, 2011 (Ithaca)	2	45	210	150	Head	–6	0.99	< 0.05	0.012	< 0.01	1.0
	(7)	48	204	146			1.1	< 0.05	0.016	< 0.01	1.2
						1	0.73	0.051	0.015	0.034	0.81
							0.65	0.052	0.015	0.033	0.73
							0.69 (mean)				0.77 (mean)
					Head,	–6	0.014	< 0.05	< 0.01	< 0.01	0.064
					w/o		0.012	< 0.05	< 0.01	< 0.01	0.062
					wrapper	1	0.012	< 0.05	< 0.01	0.031	0.093
					leaves		0.013	< 0.05	< 0.01	0.030	0.093
											0.093 (mean)
RV012-11HA, Corning, California, USA, 2011 (PYB 7101A M.T.)	2	45	204	235	Head	–6	0.20	< 0.05	< 0.01	0.013	0.26
	(7)	46	204	235			0.29	< 0.05	< 0.01	0.020	0.36
						1	0.39	< 0.05	0.01	0.021	0.46
							0.22	< 0.05	< 0.01	0.022	0.29
							0.31 (mean)				0.38 (mean)
					Head,	–6	0.011	< 0.05	< 0.01	< 0.01	0.061
					w/o		< 0.01	< 0.05	< 0.01	0.013	0.073

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
					wrapper	1	0.022	< 0.05	< 0.01	0.027	0.099
					leaves		0.039	< 0.05	< 0.01	0.026	0.11
											0.11 (mean)
RV013-11HA,	2	46	207	155	Head	–5	0.23	< 0.05	< 0.01	< 0.01	0.28
Sanger, California,	(6)	48	207	197			0.34	< 0.05	< 0.01	< 0.01	0.39
USA, 2011						1	1.0	< 0.05	0.015	0.013	1.1
(Great Lakes)							1.3	< 0.05	0.018	0.019	1.4
							1.2 (mean)				1.2 (mean)
					Head,	–5	0.032	< 0.05	< 0.01	< 0.01	0.082
					w/o		0.036	< 0.05	< 0.01	< 0.01	0.086
					wrapper	1	0.039	< 0.05	< 0.01	0.011	0.10
					leaves		0.051	< 0.05	< 0.01	< 0.01	0.10
											0.10 (mean)
RV014-11DA,	2	45	206	223	Head	–5	0.59	< 0.05	< 0.01	< 0.01	0.64
Elko, South	(6)	46	205	215			0.53	< 0.05	< 0.01	< 0.01	0.58
Carolina,						0	1.4	< 0.05	0.011	0.014	1.5
USA, 2011							1.5	< 0.05	< 0.01	0.020	1.5
(Great Lakes						1	0.51	< 0.05	< 0.01	0.018	0.57
Iceburg)							1.0	< 0.05	< 0.01	0.014	1.1
							0.76 (mean)				0.83 (mean)
						6	0.79	< 0.05	0.014	0.046	0.88
							0.46	< 0.05	< 0.01	0.033	0.54
						12	0.30	< 0.05	< 0.01	0.046	0.40
							0.31	< 0.05	< 0.01	0.037	0.40
						19	0.12	0.064	< 0.01	0.040	0.23
							0.15	0.080	< 0.01	0.047	0.28
						26	0.068	0.052	< 0.01	0.028	0.15
							0.081	0.093	< 0.01	0.039	0.21
					Head,	–5	0.016	< 0.05	< 0.01	< 0.01	0.066
					w/o		0.033	< 0.05	< 0.01	< 0.01	0.083
					wrapper	0	0.11	< 0.05	< 0.01	0.014	0.17
					leaves		0.051	< 0.05	< 0.01	0.016	0.12
						1	0.033	< 0.05	< 0.01	0.024	0.11
							0.034	< 0.05	< 0.01	0.025	0.11
						6	0.029	< 0.05	< 0.01	0.036	0.11
							0.043	< 0.05	< 0.01	0.040	0.13
						12	0.016	< 0.05	< 0.01	0.038	0.10
							0.025	< 0.05	< 0.01	0.045	0.12
						19	0.021	0.068	< 0.01	0.047	0.14
							0.016	0.069	< 0.01	0.044	0.13
						26	0.018	0.085	< 0.01	0.031	0.13
							0.018	0.10	< 0.01	0.041	0.16
											0.15 (mean)
RV015-11DA,	2	45	197	96	Head	0	3.7	< 0.05	0.028	0.055	3.8
	(6)	47	213	104			2.8	< 0.05	0.025	0.054	2.9
Elm Creek,						1	2.1	0.054	0.034	0.072	2.2

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Manitoba, Canada, 2011							1.9	< 0.05	0.037	0.068	2.0
(Summertime)							2.0 (mean)	c0.057			2.1 (mean)
						7	1.1	0.097	0.031	0.083	1.3
							0.83	0.082	0.018	0.070	0.98
						15	0.37	0.102	< 0.01	0.042	0.51
							0.25	0.099	< 0.01	0.042	0.39
						21	0.095	0.10	< 0.01	0.021	0.22
							0.039	0.058	< 0.01	0.011	0.11
						29	0.057	0.085	< 0.01	0.015	0.16
							0.048	0.078	< 0.01	< 0.01	0.13
					Head,	0	0.24	< 0.05	< 0.01	0.018	0.31
					w/o		0.17	< 0.05	< 0.01	0.028	0.25
					wrapper	1	0.12	< 0.05	< 0.01	0.027	0.20
					leaves		0.27	< 0.05	< 0.01	0.027	0.35
						7	0.35	< 0.05	< 0.01	0.049	0.45
							0.11	< 0.05	< 0.01	0.031	0.20
											0.32 (mean)
						15	0.018	0.063	< 0.01	0.023	0.10
							0.013	0.066	< 0.01	0.024	0.10
						21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	0.062	< 0.01	0.015	0.087
						29	0.035	0.060	< 0.01	0.011	0.11
							0.031	0.077	< 0.01	0.012	0.12
RV016-11DA,	2	47	208	236	Head	–6	0.27	< 0.05	< 0.01	< 0.01	0.32
Madera, California,	(7)	48	205	233			0.44	< 0.05	< 0.01	< 0.01	0.49
USA, 2011						0	2.0	< 0.05	0.013	0.016	2.1
(Vandenburg)							1.6	< 0.05	0.015	0.015	1.7
						1	1.6	< 0.05	0.018	0.019	1.7
							1.6	< 0.05	0.018	0.017	1.7
							1.6 (mean)				1.7 (mean)
						7	0.36	0.061	0.021	0.035	0.46
							0.32	0.062	0.018	0.035	0.42
						14	0.11	0.11	< 0.01	0.019	0.23
							0.15	0.11	0.013	0.036	0.30
						21	0.054	0.10	< 0.01	0.013	0.17
							0.095	0.18	< 0.01	0.022	0.29
						28	0.080	0.15	< 0.01	0.022	0.25
							0.070	0.18	< 0.01	0.019	0.27
					Head,	–6	0.066	< 0.05	< 0.01	< 0.01	0.12
					w/o		0.047	< 0.05	< 0.01	< 0.01	0.097
					wrapper	0	0.20	< 0.05	< 0.01	0.015	0.26
					leaves		0.11	< 0.05	< 0.01	< 0.01	0.16
						1	0.14	< 0.05	< 0.01	0.011	0.20
							0.14	< 0.05	< 0.01	0.015	0.21
						7	0.062	0.057	< 0.01	0.029	0.15
							0.057	< 0.05	< 0.01	0.018	0.13
						14	0.074	0.087	< 0.01	0.020	0.18

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							0.044	0.054	< 0.01	0.01	0.11
						21	0.038	0.099	< 0.01	0.011	0.15
							0.049	0.10	< 0.01	< 0.01	0.15
						28	0.045	0.16	< 0.01	0.011	0.22
							0.049	0.22	< 0.01	0.016	0.28
											0.25 (mean)
RV017-11DA, Porterville, California, USA, 2011 (Vandenburg)	2	46	206	189	Head	–6	1.9	< 0.05	0.017	0.020	2.0
	(7)	47	206	187			1.7	< 0.05	0.016	0.017	1.8
						0	1.7	< 0.05	0.020	0.037	1.8
							2.3	< 0.05	0.025	0.029	2.4
						1	1.6	< 0.05	0.018	0.034	1.6
							0.41	< 0.05	0.012	0.046	0.51
						7	1.9	< 0.05	0.026	0.054	2.0
							0.58	0.059	0.024	0.048	0.69
							1.3 (mean)				1.4 (mean)
						14	0.62	0.11	0.020	0.049	0.78
							0.31	0.088	0.015	0.048	0.45
						21	0.29	0.12	< 0.01	0.057	0.47
							0.11	0.093	< 0.01	0.031	0.23
						28	0.080	0.099	< 0.01	0.032	0.21
							0.048	0.089	< 0.01	0.030	0.17
					Head, w/o wrapper leaves	–6	0.23	< 0.05	< 0.01	0.012	0.30
							0.14	< 0.05	< 0.01	0.013	0.21
						0	0.035	< 0.05	< 0.01	0.028	0.11
							0.043	< 0.05	< 0.01	0.028	0.071
						1	0.032	< 0.05	< 0.01	0.033	0.12
							0.16	< 0.05	< 0.01	0.031	0.24
											0.18 (mean)
						7	0.044	< 0.05	< 0.01	0.059	0.15
							0.013	< 0.05	< 0.01	0.045	0.11
						14	0.014	0.090	< 0.01	0.048	0.15
							0.018	0.086	< 0.01	0.046	0.15
						21	< 0.01	0.077	< 0.01	0.029	0.12
							< 0.01	0.11	< 0.01	0.041	0.16
						28	< 0.01	0.15	< 0.01	0.035	0.19
							< 0.01	0.11	< 0.01	0.029	0.15

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 110 Residues from foliar applications of flupyradifurone to leaf lettuce in the USA and Canada (Netzband and Niczyporowicz 2012, RARVY005)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 4	2 (7)		205			1					

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RV001-11HA,	2	49	194	264	Leaf	–5	8.5	0.054	0.032	0.097	8.6
Oviedo,	(6)	49	202	276			6.7	0.058	0.035	0.10	6.9
Florida,						1	1.7	0.17	0.024	0.16	2.0
USA, 2011							1.9	0.17	0.032	0.19	2.3
(Nevada)							1.8 (mean)				2.1 (mean)
RV002-11HA,	2	47	206	188	Leaf	–6	3.9	< 0.05	0.051	0.031	4.0
Northwood,	(7)	47	203	186			4.3	< 0.05	0.035	0.031	4.4
North Dakota,						1	0.94	< 0.05	0.018	0.025	1.0
USA, 2011 (Romaine-Paris							0.80	< 0.05	0.015	0.038	0.89
Island)							0.87 (mean)				0.95 (mean)
RV003-11HA,	2	47	204	232	Leaf	–6	8.0	< 0.05	0.058	0.043	8.1
Madera,	(7)	49	205	233			7.9	< 0.05	0.05	0.069	8.0
California,						1	6.2	0.082	0.066	0.084	6.4
USA, 2011							6.3	0.080	0.064	0.099	6.5
(Black Seed Simpson)							6.3 (mean)				6.5 (mean)
RV004-11HA,	2	45	206	188	Leaf	–6	2.2	< 0.05	0.016	0.021	2.3
King City,	(7)	47	205	187			2.3	< 0.05	0.021	0.013	2.3
California,						1	2.1	< 0.05	0.031	0.060	2.3
USA, 2011							2.4	< 0.05	0.027	0.051	2.5
(Tehama)							2.3 (mean)				2.4 (mean)
RV005-11HA,	2	47	211	204	Leaf	–6	3.4	< 0.05	0.024	0.025	3.5
Abbotsford,	(7)	49	203	196			2.2	< 0.05	0.022	0.011	2.2
British Columbia,						1	4.2	< 0.05	0.055	0.058	4.3
Canada, 2011							3.4	< 0.05	0.056	0.052	3.6
(Bergram's Green)							3.8 (mean) c0.017				3.9 (mean)
RV006-11DA,	2	45	207	235	Leaf	–5	1.9	< 0.05	0.017	0.027	2.0
Alton,	(6)	47	206	234			1.7	< 0.05	0.015	0.024	1.8
New York,						0	3.8	< 0.05	0.023	0.039	3.9
USA, 2011							3.9	< 0.05	0.024	0.034	4.0
(Tropicana)						1	2.2	< 0.05	0.020	0.038	2.3
							2.0	< 0.05	0.017	0.037	2.1
							2.1 (mean)				2.2 (mean)
						7	0.30	< 0.05	< 0.01	0.032	0.39
							0.34	< 0.05	< 0.01	0.027	0.42
						14	0.24	< 0.05	< 0.01	0.019	0.31
							0.25	< 0.05	< 0.01	0.018	0.31
						21	0.14	0.05	< 0.01	0.011	0.20
							0.11	< 0.05	< 0.01	0.012	0.17
						28	0.093	0.072	< 0.01	0.011	0.18
							0.10	0.065	< 0.01	< 0.01	0.17
RV007-11DA,	2	45	206	238	Leaf	–6	6.1	< 0.05	0.040	0.030	6.1
Stewardson,	(7)	47	204	230			6.8	< 0.05	0.043	0.028	6.9

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Illinois, USA, 2011 (OakLeaf Royal)						0	6.8	0.062	0.052	0.055	7.0
							6.9	0.097	0.074	0.078	7.1
						1	6.7 c0.017	0.089	0.12	0.138	6.9
							7.9	0.077	0.082	0.096	8.0
							7.3 (mean)				7.5 (mean)
						7	1.8	0.17	0.064	0.099	2.1
							1.7	0.18	0.062	0.093	2.0
						12	0.54	0.28	0.040	0.12	0.94
							0.54	0.32	0.015	0.092	0.95
						21	0.093	0.50	< 0.01	0.048	0.64
							0.11	0.55	0.013	0.067	0.73
						28	0.10	0.36	0.021	0.040	0.50
							0.098	0.46	0.015	0.038	0.60
RV008-11DA, Porterville, California, USA, 2011 (Butter Crunch)	2 (7)	45 47	206 207	195 187	Leaf	–6	4.1	< 0.05	0.033	0.041	4.2
							4.4	< 0.05	0.037	0.046	4.5
						0	5.3	< 0.05	0.070	0.083	5.5
							4.4	< 0.05	0.065	0.072	4.5
						1	4.3	< 0.05	0.064	0.092	4.4
							1.1	< 0.05	0.062	0.11	1.2
							2.7 (mean)			c0.015	2.8 (mean)
						7	1.4	0.066	0.074	0.11	1.6
							1.3	0.059	0.076	0.11	1.5
						14	0.53	0.073	0.053	0.069	0.67
							0.56	0.088	0.064	0.093	0.74
						21	0.25	0.087	0.034	0.060	0.40
							0.23	0.10	0.029	0.052	0.39
						28	0.059	0.13	< 0.01	0.05	0.24
							0.038	0.099	< 0.01	0.036	0.17
RV009-11DA, Corning, California, USA, 2011 (Sun Valley)	2 (8)	49 49	204 206	189 189	Leaf	–7	1.2	< 0.05	0.015	0.024	1.3
							0.19	0.05	0.011	0.035	0.27
						0	4.7	< 0.05	0.025	0.056	4.8
							4.4	< 0.05	0.024	0.052	4.5
						1	1.1	0.053	0.025	0.061	1.2
							1.1	0.051	0.022	0.046	1.2
							1.1 (mean)				1.2 (mean)
						7	0.31	< 0.05	0.013	0.055	0.41
							0.46	0.066	0.020	0.060	0.58
						14	0.12	0.088	< 0.01	0.045	0.26
							0.076	0.079	< 0.01	0.035	0.19
						21	0.038	0.12	< 0.01	0.020	0.17
							0.039	0.11	< 0.01	0.032	0.18
						28	0.031	0.094	< 0.01	< 0.01	0.13
							0.028	0.082	< 0.01	< 0.01	0.11

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Legume vegetables

Supervised trials were carried out on common beans (eight trials—Table 111) and snow peas (six trials—Table 112) in the USA during the 2011 growing season (Beedle and Dallstream 2012, RARVY026) to measure the magnitude of residues in edible podded beans and peas following two broadcast foliar spray applications of BYI 02960 200 SL. All applications were made using ground-based equipment. An adjuvant [non-ionic surfactant at 0.20% v/v, methylated seed oil at 0.25% v/v or crop oil concentrate 1.0% v/v] was used in all applications.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in snap beans and snow peas were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data were obtained for all analytes.

Table 111 Residues from foliar applications of flupyradifurone to common beans in the USA and Canada (Beedle and Dallstream 2012, RARVY026)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 6	2 (10)		205			7					
RV148-11DA, Germansville, Pennslyvania USA, 2011 (Savannah)	2	55	207	270	Pod	0	2.3	0.47	0.048	0.62	3.4
	(9)	67	214	278			2.3	0.53	0.056	0.78	3.6
						7	0.24	0.84	0.039	0.68	1.8
							0.24	0.94	0.047	0.80	2.0
							0.24 (mean)				
						14	0.14	1.5	0.041	0.73	2.3
							0.11	1.4	0.039	0.66	2.2
						19	0.041	1.6	0.040	0.56	2.2
							0.045	1.7	0.059	0.67	2.4
						26	0.011	1.9	0.028	0.40	2.3
							0.01	2.0	0.035	0.53	2.5
											2.4 (mean)
RV149-11DA, Auburn, Alabama USA, 2011 (Jade)	2	67	205	158	Pod	0	1.4	0.087	0.048	0.21	1.7
	(8)	75	205	175			0.98	0.067	0.024	0.18	1.2
						7	0.81	0.16	0.11	0.55	1.5
							0.80	0.15	0.11	0.45	1.4
							0.81 (mean)				
						14	0.64	0.24	0.18	0.71	1.6
							0.47	0.22	0.16	0.53	1.2
						21	0.40	0.37	0.21	0.81	1.6
							0.38	0.40	0.24	0.77	1.5
											1.6 (mean)
						28	0.39	0.57	0.25	0.65	1.6
							0.26	0.60	0.18	0.77	1.6
RV150-11HA, Fairhope, Alabama USA, 2011 (Jade)	2	71	205	221	Pod	7	< 0.01	1.1	0.37	2.2	3.3
	(10)	84	205	218			0.014	0.98	0.40	1.7	2.7
							0.012 (mean)				3.0 (mean)
RV151-11HA, Rockwood, Ontario Canada, 2011 (24A Speculator)	2	55	204	141	Pod	6	0.22	0.63	0.033	0.81	1.7
	(10)	71	207	149			0.19	0.51	0.024	0.66	1.4
							0.21 (mean)				1.5 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RV152-11HA, Springfield, Nebraska USA, 2011 (Bush Blue Lake)	2	65	203	130	Pod	5	0.046	0.41	0.026	0.68	1.1
	(8)	71	212	138			0.080	0.46	0.040	0.90	1.4
							0.063 (mean)				1.3 (mean)
RV153-11DA,	2	69	209	145	Pod	0	0.39	0.14	0.016	0.17	0.70
Lenexa, Kansas,	(10)	73	207	147			0.30	0.15	0.020	0.24	0.69
USA, 2011						6	0.16	0.35	0.027	0.47	0.98
(Contenders)							0.15	0.36	0.025	0.55	1.1
							0.16 (mean)				
						12	0.090	0.56	0.029	0.59	1.2
							0.062	0.53	0.019	0.53	1.1
						19	0.038	0.77	0.022	0.78	1.6
							0.027	0.66	0.015	0.65	1.3
											1.5 (mean)
						26	0.01	0.92	< 0.01	0.35	1.3
							< 0.01	0.85	< 0.01	0.36	1.2
RV154-11DA, York, Nebraska USA, 2011 (Contenders)	2	67	209	189	Pod	0	0.35	0.20	0.011	0.31	0.86
	(8)	71	204	187			0.27	0.21	0.012	0.29	0.77
						7	0.17	0.42	0.035	0.53	1.1
							0.20	0.43	0.051	0.56	1.2
							0.18 (mean)				
						14	0.11	0.77	0.045	0.65	1.5
							0.11	0.71	0.043	0.64	1.5
						19	0.05	0.96	0.033	0.89	1.9
							0.048	0.87	0.036	0.66	1.6
											1.7 (mean)
						27	< 0.01	0.94	< 0.01	0.19	1.1
							0.011	0.99	< 0.01	0.25	1.3
RV155-11HA, Ephrata, Washington USA, 2011 (OSU 5630)	2	67	203	188	Pod	7	0.14	0.54	0.26	1.1	1.8
	(10)	74	204	188			0.12	0.52	0.19	0.99	1.6
							0.13 (mean)				1.7 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 112 Residues from foliar applications of flupyradifurone to snow peas in the USA and Canada (Beedle and Dallstream 2012, RARVY026)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 6	2 (10)		205			7					
RV156-11HA, Germansville, Pennslyvania USA, 2011 (Snow Sweet Nat II)	2	74	211	274	Pod	6	1.3	0.66	0.041	0.18	2.1
	(8)	77	211	274			1.1	0.95	0.053	0.21	2.3
							1.2 (mean)				2.2 (mean)
RV157-11DA, Rockwood, Ontario Canada, 2011 (231A Little Sweetie)	2	64	208	142	Pod	0	1.3	0.49	< 0.01	0.11	1.9
	(10)	69	208	150			1.3	0.37	< 0.01	0.092	1.7
						6	0.94	0.92	0.023	0.21	2.1
							0.99	1.0	0.030	0.23	2.2
						14	0.72	1.8	0.024	0.29	2.8
							0.53	1.5	0.021	0.27	2.3
						21	0.29	1.5	< 0.01	0.31	2.1
							1.1	1.7	0.016	0.56	3.4
						26	1.3	1.6	0.019	0.78	3.7
							1.0	1.2	0.015	0.63	2.9
							1.2 (mean)				3.3 (mean)
RV158-11HA, Atlantic, Iowa USA, 2011 (Sugar Pod)	2	65	201	190	Pod	7	1.0	0.44	0.020	0.11	1.6
	(10)	77	206	192			0.86	0.55	0.025	0.15	1.6
							0.95 (mean)				1.6 (mean)
RV159-11HA, Stewardson, Illinois USA, 2011 (Oregon Sugar Pod II)	2	67	240	236	Pod	7	0.65	0.98	0.016	0.19	1.8
	(7)	71	212	239			0.49	0.80	< 0.01	0.17	1.5
							0.57 (mean)				1.6 (mean)
RV160-11DA,	2	64	207	237	Pod	0	1.5	0.35	< 0.01	0.11	1.9
Payette, Idaho,	(10)	75	205	235			1.6	0.28	< 0.01	0.081	1.9
USA, 2011						7	0.59	0.74	0.01	0.20	1.5
							0.57	0.69	0.011	0.18	1.4
(Oregon Sugar Pod II)							0.58 (mean)				1.5 (mean)
						10	0.35	0.76	< 0.01	0.13	1.2
							0.33	0.89	< 0.01	0.14	1.4
						14	0.25	0.98	< 0.01	0.15	1.4
							0.34	1.2	0.011	0.17	1.7
						21	0.05	1.1	< 0.01	0.13	1.3
							0.084	1.2	< 0.01	0.17	1.4
RV161-11HA, Portland, Oregon USA, 2011 (Progress 9)	2	65	203	202	Pod	7	1.0	0.58 c0.12	0.026	0.23	1.8
	(10)	75	211	188			0.95	0.52 c0.12	0.022	0.23	1.7
							0.98 (mean)				1.8 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Supervised trials were carried out on <u>lima beans</u> (nine trials—Table 113) and <u>peas</u> (six trials—Table 114) in the USA during the 2011 growing season (Fischer 2012c, RARVY027) to measure the magnitude of residues in the succulent seed without pod (green seed) of lima beans and garden peas following two broadcast foliar spray applications of BYI 02960 200 SL. All applications were made using ground-based equipment. An adjuvant [non-ionic surfactant at 0.20–0.25% v/v, methylated seed oil at 0.25% v/v or crop oil concentrate 1.0% v/v] was used in all applications.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in lima beans and garden peas were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data were obtained for all analytes.

Table 113 Residues from foliar applications of flupyradifurone to lima beans in the USA and Canada (Fischer 2012c, RARVY027)

Trial No., Location, Year (Variety)	Application No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)	Sample	DALA	Residues as parent (mg/kg) Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 6	2 (10)		205			7					
RV170-11HA, North Rose, New York USA, 2011 (Eastland)	2 (10)	70 77	204 206	230 230	Seed, Green	7	< 0.01 < 0.01 < 0.01 (mean)	< 0.05 < 0.05	< 0.01 < 0.01	< 0.01 < 0.01	< 0.06 < 0.06 < 0.06 (mean)
RV171-11HA, Athens, Georgia USA, 2011 (Cangreen)	2 (10)	72 76	204 205	230 230	Seed, Green	7	0.11 0.094 0.10 (mean)	0.21 0.21	0.048 0.042	0.10 0.11	0.42 0.41 0.41 (mean)
RV172-11HA, Suffolk, Virginia USA, 2011 (Thorogreen)	2 (8)	71 80	210 206	130 130	Seed, Green	7	0.12 0.11 0.11 (mean)	0.18 0.19	0.10 0.11	0.26 0.25	0.56 0.56 0.56 (mean)
RV173-11DA, Chula, Georgia USA, 2011 (Cangreen)	2 (10)	75 75	204 204	170 170	Seed, Green	0	0.14 0.13	0.12 0.14	0.033 0.031	0.12 0.13	0.38 0.39
						7	0.067 0.065	0.31 0.33	0.033 0.035	0.15 0.14	0.52 0.54
						14	0.069 0.055 0.062 (mean)	0.40 0.44	0.051 0.042	0.19 0.16	0.66 0.66
						21	0.057 0.052	0.69 0.68	0.058 0.055	0.22 0.20	0.97 0.94 0.95 (mean)
						28	0.051 0.054	0.64 0.60	0.054 0.051	0.25 0.23	0.94 0.89
RV174-11HB, Ostrander, Ontario Canada, 2011 (IMP Kingston)	2 (8)	77 79	210 204	210 200	Seed, green	7	< 0.01 0.011 0.011 (mean)	< 0.05 < 0.05	0.020 0.028	0.077 0.089	0.14 0.15 0.14 (mean)
RV176-11DA, Geneva, Minnesota USA, 2011 (Fordhook 242 Bush)	2 (10)	75 79	206 204	200 190	Seed, green	0	0.016 0.014	< 0.05 < 0.05	< 0.01 < 0.01	0.034 0.025	0.10 0.089
						6	0.012 0.012 0.012 (mean)	< 0.05 < 0.05	0.013 0.012	0.066 0.055	0.13 0.12
						13	< 0.01 < 0.01	0.054 0.054	< 0.01 0.014	0.053 0.057	0.12 0.12
						20	< 0.01 < 0.01	0.078 0.074	0.013 0.012	0.071 0.060	0.16 0.14
						27	< 0.01	0.094	0.018	0.082	0.19

Trial No., Location, Year (Variety)	Application No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)	Sample	DALA	Residues as parent (mg/kg) Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							0.013	0.097	0.023	0.090	0.20
						33	< 0.01	0.12	0.018	0.078	0.20
							< 0.01	0.11	0.019	0.081	0.20
											0.20 (mean)
RV177-11DA, Delavan, Wisconsin	2 (10)	75	204	200	Seed, Green	0	0.070	< 0.05	0.034	0.072	0.19
		76	205	200			0.053	< 0.05	0.028	0.077	0.18
USA, 2011 (Henderson's Bush)						7	0.028	0.065	0.040	0.090	0.18
							0.025	0.059	0.035	0.075	0.16
							0.027 (mean)				
						14	0.011	0.081	0.031	0.067	0.16
							0.011	0.079	0.032	0.084	0.17
						21	< 0.01	0.060	0.027	0.061	0.13
							< 0.01	0.081	0.040	0.085	0.18
						28	< 0.01	0.084	0.024	0.061	0.16
							< 0.01	0.084	0.026	0.063	0.16
						34	< 0.01	0.12	0.023	0.060	0.19
							< 0.01	0.12	0.022	0.064	0.19
											0.19 (mean)
RV178-11DA, Sanger, California	2 (10)	82	205	280	Seed, green	0	0.020	< 0.05	< 0.01	0.013	0.083
		85	205	280			0.025	< 0.05	< 0.01	0.020	0.096
USA, 2011 (Fordhook)						7	0.022	< 0.05	0.01	0.030	0.10
							0.018	< 0.05	< 0.01	0.028	0.095
						14	0.029	< 0.05	0.019	0.036	0.11
							0.020	< 0.05	0.012	0.025	0.095
							0.025 (mean)				0.10 (mean)
						21	0.023	< 0.05	0.012	0.040	0.11
							0.016	< 0.05	0.012	0.025	0.091
						28	0.015	< 0.05	< 0.01	0.039	0.10
							0.013	< 0.05	< 0.01	0.028	0.091
						35	0.016	< 0.05	0.012	0.026	0.092
							0.017	< 0.05	0.014	0.036	0.10
RV179-11HA, Payette, Idaho	2 (9)	72	201	230	Seed, green	7	< 0.01	< 0.05	< 0.01	0.075	0.14
USA, 2011 (Fordhook 242)		75	207	240			< 0.01	< 0.05	< 0.01	0.072	0.13
							< 0.01 (mean)				0.13 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 3 Residues from foliar applications of flupyradifurone to peas in the USA (Fischer 2012c, RARVY027)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 6	2 (10)		205			7					
RV163-11DA,	2	71	208	270	Seed,	0	0.62	0.11	0.022	0.055	0.78
Germansville,	(8)	75	206	270	Green		0.60	0.13	0.022	0.061	0.79
Pennslyvania						7	0.77	0.31	0.074	0.21	1.3

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
USA, 2011							0.77	0.30	0.075	0.22	1.3
(Strike)						14	0.68	0.73	0.037	0.38	1.8
							0.70	0.75	0.037	0.36	1.8
						20	1.4	1.4	0.053	0.73	3.5
							1.4	1.4	0.053	0.82	3.7
						28	1.5	1.5	0.059	0.79	3.8
							1.4	1.5	0.058	0.75	3.6
						33	1.5	1.5	0.058	0.83	3.9
							1.6	1.5	0.060	0.89	3.9
							1.5 (mean)				3.9 (mean)
RV164-11DA, Jeffersonville, Georgia USA, 2011 (Thomas Laxton)	2	64	206	220	Seed, green	0	0.16	0.41	< 0.01	0.22	0.79
	(10)	65	206	230			0.15	0.39	< 0.01	0.22	0.76
						7	0.52	0.93	0.017	0.44	1.9
							0.51	0.91	0.017	0.42	1.8
							0.51 (mean)			c0.044	1.9 (mean)
						14	0.016	1.7	< 0.01	0.23	2.0
							0.20	1.0	< 0.01	0.37	1.6
						21	0.14	1.3	< 0.01	0.32	1.7
RV165-11HA, Fitchburg, Wisconsin USA, 2011 (Kalamo)	2	69	207	240	Seed, green	7	0.12	0.47	< 0.01	0.18	0.77
	(10)	77	208	200			0.13	0.59	< 0.01	0.24	0.95
							0.12 (mean)				0.86 (mean)
RV166-11HA, St George, Wisconsin USA, 2011 (Spring)	2	75	204	200	Seed, green	7	0.79	0.62	0.036	0.31	1.7
	(8)	80	218	210			0.76	0.59	0.033	0.33	1.7
							0.77 (mean)			c0.043	1.7 (mean)
RV167-11DA, Northwood, North Dakota USA, 2011 (Maestro)	2	75	206	190	Seed, green	0	0.56	0.083	0.016	0.15	0.80
	(9)	79	207	190			0.58	0.084	0.016	0.15	0.82
						7	0.59	0.14	0.024	0.24	0.98
							0.64	0.14	0.028	0.28	1.1
							0.62 (mean)				
						15	0.48	0.23	0.032	0.34	1.1
							0.47	0.29	0.029	0.33	1.1
						21	0.38	0.32	0.027	0.39	1.1
							0.35	0.30	0.024	0.35	0.99
											1.0 (mean)
						28	0.25	0.38	0.027	0.34	0.98
							0.25	0.40	0.032	0.40	1.0
RV168-11DA, Rupert, Idaho USA, 2011 (Progress 9)	2	62	201	170	Seed, green	0	0.15	0.086	< 0.01	0.067	0.30
	(10)	76	206	170			0.14	0.082	< 0.01	0.067	0.29
						7	0.24	0.25	< 0.01	0.21	0.69
							0.26	0.27	< 0.01	0.20	0.73
							0.25 (mean)	c0.051			
						14	0.23	0.54	< 0.01	0.27	1.0
							0.19	0.58	< 0.01	0.28	1.1
						21	0.048	1.2	< 0.01	0.28	1.5
							0.042	1.2	< 0.01	0.27	1.6
						28	< 0.01	1.3	< 0.01	0.17	1.5
							< 0.01	1.3	< 0.01	0.16	1.5
						35	< 0.01	1.9	< 0.01	0.089	2.0

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							< 0.01	1.7	< 0.01	0.094	1.8
											1.9 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Pulses

Supervised trials were carried out on <u>beans</u> (nine trials—Table 115) and <u>peas</u> (ten trials—Table 116) in the USA during the 2011 growing season (Hoag, Arthur and Woodard 2012, RARVY028) to measure the magnitude of residues in the dried seeds of beans and peas and in bean and pea forage and hay following two broadcast foliar spray applications of BYI 02960 200 SL. All applications were made using ground-based equipment. An adjuvant [non-ionic surfactant at 0.20–0.25% v/v, methylated seed oil at 0.25% v/v or crop oil concentrate 1.0% v/v] was used in all applications.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in dried seeds, forage and hay of beans and peas were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data were obtained for all analytes.

Residues in pea vines and hay are shown in Table 132 and in bean forage and hay in Table 133.

Table 115 Residues from foliar applications of flupyradifurone to beans (dry) in the USA and Canada (Hoag, Arthur and Woodard 2012, RARVY028)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 6	2 (10)		205			7					
RV190-11HA, Marysville, Ohio USA, 2011 (Vista)	2	80	205	185	Seed,	7	< 0.01	< 0.05	< 0.01	0.060	0.12
	(10)	86	206	179	dry		< 0.01	< 0.05	< 0.01	0.068	0.13
							< 0.01 (mean)				0.12 (mean)
RV191-11HA, Lenexa, Kansas USA, 2011 (Pink Eye Purple hull)	2	70	207	147	Seed,	6	0.030	< 0.05	0.40	0.69	0.77
	(9)	75	202	143	dry		0.043	< 0.05	0.47	0.87	0.96
							0.036 (mean)				0.87 (mean)
RV192-11HA, Geneva, Minnesota USA, 2011 (Great Northern)	2	82	206	190	Seed,	6	0.019	< 0.05	0.012	0.68	0.75
	(9)	85	208	195	dry		0.019	< 0.05	0.01	0.47	0.54
							0.019 (mean)			c0.016	0.64 (mean)
RV193-11HA, Taber, Alberta Canada, 2011 (AC Redbond)	2	81	206	176	Seed,	7	0.063	< 0.05	0.013	1.0	1.1
	(10)	85	207	175	dry		0.077	< 0.05	0.019	1.2	1.3
							0.070 (mean)				1.2 (mean)
RV195-11HA, Jerome, Idaho USA, 2011 (Othello)	2	86	206	206	Seed,	7	0.012	< 0.05	0.012	0.50	0.56
	(8)	88	208	206	dry		0.01	< 0.05	0.014	0.84	0.90
							0.011 (mean)				0.73 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RV196-11DA,	2	83	206	196	Seed,	0	0.078	< 0.05	< 0.01	0.35	0.47
Atlantic, Iowa	(9)	87	208	212	dry		0.054	< 0.05	< 0.01	0.29	0.39
USA, 2011						7	0.069	< 0.05	< 0.01	0.18	0.30
(Black Turtle)							0.059	< 0.05	< 0.01	0.18	0.29
						14	0.092	< 0.05	< 0.01	0.23	0.37
							0.085	< 0.05	< 0.01	0.24	0.37
						21	0.19	< 0.05	< 0.01	0.31	0.55
							0.21	< 0.05	< 0.01	0.32	0.57
						28	0.25	< 0.05	< 0.01	0.17	0.47
							0.24	< 0.05	< 0.01	0.22	0.51
							0.24 (mean)				
						35	0.21	< 0.05	< 0.01	0.40	0.66
							0.26	< 0.05	< 0.01	0.49	0.80
											0.73 (mean)
RV197-11DA, Eldridge, North Dakota USA, 2011 (Navigator)	2	80	211	144	Seed,	0	0.096	< 0.05	0.041	0.37	0.52
	(9)	87	208	144	dry		0.17	< 0.05	0.045	0.55	0.76
						7	0.045	< 0.05	0.068	1.5	1.6
							0.041	< 0.05	0.076	1.2	1.3
							0.043 (mean)				1.5 (mean)
						14	0.023	< 0.05	0.035	0.63	0.70
							0.012	< 0.05	0.035	0.91	0.98
						21	0.028	< 0.05	0.052	1.3	1.4
							0.031	< 0.05	0.062	1.5	1.6
						27	0.017	< 0.05	0.053	1.5	1.6
							0.037	< 0.05	0.042	1.2	1.2
						33	0.022	< 0.05	0.057	1.4	1.4
							0.026	< 0.05	0.053	1.3	1.4
RV198-11DA,	2	73	205	187	Seed,	0	0.073	< 0.05	0.032	0.042	0.16
Kerman, California	(10)	89	204	187	dry		0.073	< 0.05	0.048	0.044	0.17
USA, 2011						7	0.092	< 0.05	0.055	0.078	0.22
(Blue Lake 274)							0.15	< 0.05	0.044	0.051	0.25
							0.12 (mean)				
						14	0.090	< 0.05	0.075	0.18	0.32
							0.10	< 0.05	0.047	0.036	0.19
						21	0.047	< 0.05	0.046	0.049	0.15
							0.12	< 0.05	0.047	0.055	0.22
						28	0.018	< 0.05	0.012	0.15	0.22
							0.018	< 0.05	0.019	0.026	0.095
						35	0.038	0.085	0.056	0.52	0.64
							0.036	0.062	0.046	0.24	0.33
											0.49 (mean)
RV199-11DA,	2	78	202	148	Seed,	0	0.096	0.24	0.047	5.2	5.5
Rupert, Idaho	(10)	81	202	159	dry		0.095	0.27	0.05	5.2	5.5
USA, 2011						7	0.029	0.26	0.051	5.5	5.8
(Bill Z)							0.030	0.24	0.049	4.9	5.2
						14	0.019	0.22	0.041	5.8	6.1
							0.027	0.21	0.042	5.1	5.4
						21	0.029	0.26	0.045	5.1	5.4
							0.025	0.28	0.038	6.4	6.7
						28	0.020	0.24	0.041	4.6	4.9

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							0.033	0.29	0.045	5.9	6.2
						35	0.037	0.34	0.054	7.1	7.4
							0.036	0.32	0.052	7.1	7.4
							0.036 (mean)				7.4 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 116 Residues from foliar applications of flupyradifurone to peas (dry) in the USA and Canada (Hoag, Arthur and Woodard 2012, RARVY028)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 6	2 (10)		205			7					
RV180-11HA, Rockwood, Ontario Canada, 2011 (Meadow)	2	80	203	141	Seed,	7	0.58	0.25	< 0.01	0.45	1.3
	(9)	85	206	146	dry		0.76	0.32	< 0.01	0.40	1.5
							0.67 (mean)				1.4 (mean)
RV181-11HA, Rupert, Idaho USA, 2011 (Progress 9)	2	77	206	162	Seed,	7	0.51	0.22	< 0.01	0.27	1.0
	(10)	82	205	151	dry		0.38	0.12	< 0.01	0.15	0.66
							0.45 (mean)				0.83 (mean)
RV182-11HA, Jerome, Idaho USA, 2011 (FMK 888-0132*N14)	2	86	205	202	Seed,	7	0.020	< 0.05	< 0.01	< 0.01	0.070
	(9)	88	210	212	dry		0.014	< 0.05	< 0.01	< 0.01	0.064
							0.017 (mean)				0.067 (mean)
RV183-11HA, Payette, Idaho USA, 2011 (Austrian Winter Pea)	2	80	207	235	Seed,	7	1.5	0.64	0.013	0.38	2.5
	(10)	84	206	234	dry		0.86	0.49	< 0.01	0.34	1.7
							1.2 (mean)				2.1 (mean)
RV184-11HA, Waldheim, Saskatchewan Canada, 2011 (Admiral)	2	82	208	111	Seed,	7	0.13	0.12	< 0.01	0.19	0.44
	(9)	88	206	111	dry		0.13	0.11	< 0.01	0.16	0.41
							0.13 (mean)				0.42 (mean)
RV185-11HA, Blaine Lake, Saskatchewan Canada, 2011 (Admiral)	2	79	204	108	Seed,	7	0.53	0.12	0.019	0.26	0.91
	(9)	87	206	109	dry		0.41	0.10	0.014	0.21	0.72
							0.47 (mean)				0.82 (mean)
RV186-11DA, Ephrata, Washington USA, 2011 (Austrian Winter	2	72	207	188	Seed,	0	1.0	1.3	< 0.01	0.62	2.9
	(10)	82	206	187	dry		0.94	1.8	< 0.01	0.50	3.2
						7	1.4	3.	< 0.01	0.76	5.3
							1.3	2.1	< 0.01	0.73	4.1
Pea)							1.3	c0.57		c0.012	

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							(mean)				
						14	1.2	3.7	< 0.01	0.90	5.8
							1.2	4.5	< 0.01	0.92	6.6
											6.2 (mean)
						21	1.2	2.3	< 0.01	0.63	4.2
							1.2	2.4	< 0.01	0.75	4.3
						28	1.0	3.4	< 0.01	0.88	5.3
							0.98	2.9	< 0.01	0.93	4.8
						35	1.2	2.7	< 0.01	0.81	4.7
							0.95	2.5	< 0.01	0.85	4.2
RV187-11DA,	2	79	206	188	Seed,	0	0.51	0.26	0.026	0.25	1.0
Parkdale, Oregon	(10)	81	208	192	dry		0.51	0.24	0.029	0.25	1.0
USA, 2011						7	0.79	0.26	0.030	0.35	1.4
(Progress 9)							0.87	0.36	0.046	0.53	1.8
						13	0.98	0.30	0.039	0.34	1.6
							0.89	0.46	0.044	0.71	1.6
						21	1.0	0.53	0.061	0.55	2.1
							0.93	0.50	0.055	0.54	2.0
											2.0 (mean)
						28	1.2	0.49	0.051	0.57	2.2
							0.93	0.41	0.045	0.58	1.9
							1.0 (mean)				
						35	0.99	0.47	0.055	0.66	2.1
							0.82	0.44	0.043	0.50	1.8
RV188-11DA,	2	80	204	108	Seed,	0	0.68	0.12	< 0.01	0.20	1.0
Rosthern,	(10)	88	206	111	dry		0.62	0.12	< 0.01	0.18	0.91
Saskatchewan						7	0.20	0.074	< 0.01	0.11	0.39
Canada, 2011							0.22	0.075	< 0.01	0.12	0.42
(Meadow)						12	0.35	0.10	< 0.01	0.16	0.61
							0.41	0.14	< 0.01	0.22	0.77
							0.38 (mean)				0.69 (mean)
						21	0.37	0.11	< 0.01	0.11	0.59
							0.39	0.12	< 0.01	0.15	0.66
						26	0.27	0.064	< 0.01	0.086	0.41
							0.26	0.068	< 0.01	0.10	0.42
						33	0.21	0.060	< 0.01	0.084	0.35
							0.18	0.052	< 0.01	0.075	0.30
RV189-11DA,	2	79	204	108	Seed,	0	0.84	0.17	< 0.01	0.24	1.3
Wakaw,	(10)	86	206	111	dry		0.92	0.18	0.01	0.24	1.3
Saskatchewan						7	0.57	0.25	< 0.01	0.36	1.2
Canada, 2011							0.60	0.27	< 0.01	0.41	1.3
(Meadow)						12	0.71	0.30	< 0.01	0.38	1.4
							0.66	0.33	< 0.01	0.37	1.4
						21	0.75	0.34	< 0.01	0.40	1.5
							0.84	0.41	< 0.01	0.51	1.8
						26	0.70	0.33	< 0.01	0.43	1.5
							0.76	0.36	< 0.01	0.40	1.5
						33	0.81	0.46	0.012	0.46	1.7
							0.81	0.44	0.011	0.50	1.8
							0.81 (mean)				1.7 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Twenty supervised trials were carried out on soya beans (Table 115) in the USA and Canada during the 2010-2011 growing seasons (Sturdivant 2012, RARVY011) to measure the magnitude of residues in/on soya bean seed, forage and hay following two broadcast foliar spray applications of BYI 02960 200 SL, and in three trials, by planting seed treated with BYI 02960 480 FS. All foliar applications were made using ground-based equipment. Adjuvants were used in all foliar applications at a rate of 0.25% v/v.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in soya bean seed, forage and hay were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data (mean recoveries, RSDs) were obtained for all analytes.

Residues in soya bean forage and hay are shown in Table 134.

Table 117 Residues from foliar or seed treatment applications of flupyradifurone to soya beans in the USA and Canada (Sturdivant 2012, RARVY011)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Soya bean seed	2 (10)		205			21					
RV132-10DA,	2	80	202	192	Seed,	9	0.019	< 0.05	0.014	0.048	0.12
Seven Springs,	(10)	85	204	198	dry		0.016	< 0.05	0.023	0.086	0.15
North Carolina						14	< 0.01	< 0.05	0.013	0.044	0.10
USA, 2010							< 0.01	< 0.05	0.016	0.05	0.11
(AG5605)						21	0.013	< 0.05	0.016	0.069	0.13
							< 0.01	< 0.05	0.019	0.072	0.13
							0.012 (mean)			c0.027	0.13 (mean)
						28	0.011	< 0.05	0.016	0.051	0.11
							0.01	< 0.05	0.018	0.064	0.12
						35	0.011	< 0.05	0.019	0.073	0.13
							0.013	< 0.05	0.020	0.063	0.13
RV133-10HA, Tallassee, Alabama USA, 2010 (Stine 4782-4)	2	77	207	167	Seed,	21	0.24	0.069	0.078	0.87	1.2
	(10)	81	205	230	dry		0.27	0.069	0.10	0.91	1.2
							0.25 (mean)				1.2 (mean)
	1	00	32		Seed,	131	< 0.01	0.75	< 0.01	0.34	1.1
					dry		< 0.01	0.87	< 0.01	0.44	1.3
RV134-10DA,	2	79	205	188	Seed,	10	0.56	0.29	0.18	2.0	2.8
Proctor, Arkansas	(10)	85	204	188	dry		0.56	0.27	0.13	1.8	2.7
USA, 2010						15	0.77	0.38	0.21	3.0	4.2
(Armor 47G7)							0.85	0.36	0.18	2.2	3.4
						21	0.62	0.30	0.18	2.7	3.6
							0.60	0.39	0.23	3.1	4.1
							0.61 (mean)				3.9 (mean)
						28	0.37	0.26	0.10	2.2	2.9
							0.37	0.28	0.11	2.2	2.8
						35	0.48	0.26	0.12	2.0	2.7
							0.40	0.22	0.098	1.7	2.3

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RV135-10HA, Fisk, Missouri USA, 2010 (Stine 4782-4)	2	92	204	187	Seed,	20	< 0.01	< 0.05	< 0.01	0.020	0.080
	(8)	96	204	186	dry		0.020	< 0.05	< 0.01	0.020	0.090
							0.015 (mean)			c0.019	0.085 (mean)
	1	00	51		Seed,	138	< 0.01	0.48	< 0.01	0.054	0.55
					dry		< 0.01	0.47	< 0.01	0.052	0.54
RV136-10HA, Pollard, Arkansas USA, 2010 (Pioneer 94M80)	2	91	206	188	Seed,	20	0.089	0.19	0.10	1.5	1.8
	(8)	81	205	188	dry		0.075	0.19	0.10	1.6	1.8
							0.082 (mean)				1.8 (mean)
RV137-10DB,	2	77	207	296	Seed,	8	0.20	0.30	0.12	1.6	2.1
Branchton,	(9)	88	221	336	dry		0.16	0.29	0.098	1.8	2.2
Ontario						14	0.21	0.46	0.15	1.9	2.6
Canada, 2010							0.22	0.51	0.16	2.4	3.1
(Secan RCAT						21	0.23	0.54	0.18	2.3	3.0
Matrix)							0.25	0.49	0.17	2.2	2.9
										c0.021	
						28	0.38	0.51	0.21	2.4	3.3
							0.33	0.65	0.24	4.0	5.0
							0.36 (mean)				4.1 (mean)
						35	0.31	0.54	0.20	2.3	3.1
							0.26	0.40	0.17	2.0	2.7
RV138-10HA, Springfield, Nebraska USA, 2010 (NC+3051R)	2	79	211	149	Seed,	19	0.072	0.088	0.10	1.5	1.6
	(8)	79	204	148	dry		0.065	0.083	0.091	1.3	1.4
							0.068 (mean)			c0.041	1.5 (mean)
	1	00	49		Seed,	134	< 0.01	0.12	< 0.01	0.038	0.16
					dry		< 0.01	0.11	< 0.01	0.025	0.14
RV148-10HA,	2	77	208	134	Seed,	19	0.075	0.38	0.055	1.2	1.7
Springfield, Nebraska	(8)	82	206	133	dry		0.063	0.36	0.053	1.3	1.7
US, 2011							0.069				1.7
(S28-B4)							(mean)				(mean)
RV139-10HA,	2	75	198	172	Seed,	20	0.032	0.10	0.051	1.6	1.8
York, Nebraska	(8)	89	205	191	dry		0.036	0.11	0.053	1.6	1.7
USA, 2010							0.034				1.8
(NC+2751R)							(mean)				(mean)
RV150-10HA,	2	80	205	186	Seed,	21	< 0.01	< 0.05	< 0.01	0.20	0.26
York, Nebraska	(8)	93	206	191	dry		< 0.01	< 0.05	< 0.01	0.17	0.23
USA, 2011							< 0.01			c0.065	0.25
(16501RR)							(mean)				(mean)
RV140-10HA,	2	83	206	130	Seed,	20	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Rockwood, Ontario	(7)	85	209	132	dry		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Canada, 2010							< 0.01				< 0.060
(90M01)							(mean)				(mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RV141-10HA,	2	79	206	188	Seed,	22	0.16	0.49	0.074	2.9	3.6
Campbell, Minnesota	(8)	92	205	187	dry		0.15	0.52	0.073	3.1	3.8
USA, 2010							0.15			c0.015	3.7
(AG 0808)							(mean)				(mean)
RV142-10HA,	2	77	202	180	Seed,	21	0.19	0.11	0.045	0.36	0.66
Clarence, Missouri	(10)	79	207	195	dry		0.24	0.10	0.044	0.30	0.65
USA, 2010							0.22				0.66
(Asgrow 3803)							(mean)				(mean)
RV143-10HA,	2	83	207	145	Seed,	20	0.018	< 0.05	< 0.01	0.041	0.11
Breslau, Ontario	(7)	85	204	127	dry		0.020	< 0.05	< 0.01	0.038	0.11
Canada, 2010							0.019			c0.037	0.11
(DKBOO-99)							(mean)				(mean)
RV144-10HA,	2	79	203	163	Seed,	21	0.017	< 0.05	0.015	0.19	0.26
Richland, Iowa	(10)	88	206	154	dry		0.013	< 0.05	0.014	0.20	0.26
USA, 2010							0.015			c0.013	0.26
(Pioneer 92Y80)							(mean)				(mean)
RV145-10HA,	2	83	205	129	Seed,	20	< 0.01	< 0.05	< 0.01	0.036	0.096
Cambridge, Ontario	(7)	85	204	131	dry		< 0.01	< 0.05	< 0.01	0.025	0.085
Canada, 2010							< 0.01			c0.012	0.090
(90M40)							(mean)				(mean)
RV146-10DA,	2	79	203	183	Seed,	10	< 0.01	< 0.05	< 0.01	0.021	0.081
Northwood,	(10)	79	208	190	dry		< 0.01	< 0.05	< 0.01	0.022	0.082
North Dakota						15	< 0.01	< 0.05	< 0.01	0.022	0.082
USA, 2010							< 0.01	< 0.05	< 0.01	0.021	0.081
(Asgrow						21	< 0.01	< 0.05	< 0.01	0.019	0.079
AG00901)							< 0.01	< 0.05	< 0.01	0.018	0.078
							< 0.01			c0.012	
							(mean)				
						28	< 0.01	< 0.05	< 0.01	0.017	0.077
							< 0.01	< 0.05	< 0.01	0.022	0.082
						35	< 0.01	< 0.05	< 0.01	0.022	0.082
							< 0.01	< 0.05	< 0.01	0.021	0.081
											0.082 (mean)
RV147-10HA,	2	77	204	184	Seed,	20	1.1	1.7	1.0	7.5	10
Stafford, Kansas	(9)	79	208	193	dry		0.94	1.5	0.90	8.5	11
USA, 2011							1.0 (mean)			8.0 (mean)	11 (mean)
(Pioneer 93Y70)										c0.12	
RV149-10HA,	2	77	209	148	Seed,	19	0.28	0.25	0.096	1.3	1.9
Lenexa, Kansas	(10)	79	202	147	dry		0.27	0.27	0.12	1.6	2.2
USA, 2011							0.28			c0.060	2.0
(Willcross RR2428N)							(mean)				(mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RV151-10HA,	2	79	208	305	Seed,	21	0.045	0.092	0.12	0.54	0.67
Kimballton, Iowa	(10)	80	199	293	dry		0.061	0.090	0.13	0.57	0.72
USA, 2011							0.053				0.70
(Stine 2862-4)							(mean)				(mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Root and tuber vegetables

Seventeen supervised trials were carried out on carrots (10 trials—Table 116) and radishes (seven trials—Table 119) in the USA and Canada during the 2011 growing season (Murphy 2012, RARVY039) to measure the magnitude of residues in/on carrots and radishes, following two broadcast foliar spray applications of BYI 02960 200 SL. All of them were made using ground-based equipment. All applications included an adjuvant (non-ionic surfactant at 0.20% v/v, crop oil concentrate at 1.0% v/v or methylated seed oil at 0.25% v/v). Additional samples of carrot were collected in three trials at a PHI of 7 days and washed and cooked to evaluate potential residue reduction resulting from common food practices.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in carrots and radishes were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data (mean recoveries, RSDs) were obtained for all analytes.

Table 118 Residues from the foliar application of flupyradifurone to carrots in the USA and Canada (Murphy 2012, RARVY039)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Subgroup 1B	2 (10)		205			7					
RV204-11DA,	2	48	209	238	Root	0	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Alton,	(7)	49	207	235			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
New York						7	0.022	0.056	< 0.01	0.016	0.094
USA, 2011							0.011	0.058	< 0.01	0.017	0.085
(Vitana F1)							0.017 (mean)				
						14	< 0.01	0.076	< 0.01	0.017	0.10
							0.014	0.082	< 0.01	0.017	0.11
											0.11 (mean)
						21	< 0.01	0.078	< 0.01	0.015	0.10
							< 0.01	0.067	< 0.01	0.011	0.088
						28	0.011	0.071	< 0.01	0.011	0.093
							< 0.01	0.088	< 0.01	0.020	0.12
						35	< 0.01	0.077	< 0.01	0.013	0.10
							< 0.01	0.078	< 0.01	0.013	0.10
RV205-11HA,	2	46	205	205	Root	7	0.015	< 0.05	< 0.01	0.012	0.077
Madison,	(10)	48	204	206			0.013	< 0.05	< 0.01	0.013	0.075
Florida USA, 2011							0.014 (mean)				0.076 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
(Maverick)					RAC	7	0.019 (av.)	< 0.05 (av.)	< 0.01 (av.)	0.013 (av.)	0.082
					Root, washed	7	0.012 (av.)	< 0.05 (av.)	< 0.01 (av.)	0.011 (av.)	0.073
					Root, cooked	7	0.013 (av.)	< 0.05 (av.)	< 0.01 (av.)	0.011 (av.)	0.074
RV206-11DA,	2	45	206	188	Root	0	0.14	< 0.05	< 0.01	< 0.01	0.19
Lime Springs,	(8)	47	208	189			6.8	0.065	0.044	0.01	6.9
Iowa						5	0.87	0.090	0.026	0.013	0.97
USA, 2011							0.21	0.063	< 0.01	< 0.01	0.27
(Nantindo F1)						12	1.0	0.076	0.030	0.01	1.1
							0.19	0.065	< 0.01	< 0.01	0.25
							0.60 (mean)				0.68 (mean)
						19	0.049	< 0.05	< 0.01	< 0.01	0.099
							0.067	< 0.05	< 0.01	< 0.01	0.12
						26	0.024	< 0.05	< 0.01	< 0.01	0.074
							0.025	< 0.05	< 0.01	< 0.01	0.075
						33	0.018	< 0.05	< 0.01	< 0.01	0.068
							0.022	0.056	< 0.01	< 0.01	0.078
RV207-11DA, Northwood, North Dakota USA, 2011 (Kuroda)	2	45	203	186	Root	0	0.016	< 0.05	< 0.01	< 0.01	0.066
	(9)	45	205	187			0.024	< 0.05	< 0.01	< 0.01	0.074
						7	0.018	< 0.05	< 0.01	< 0.01	0.068
							0.023	0.060	< 0.01	0.012	0.095
							0.021 (mean)				
						14	0.017	0.072	< 0.01	0.016	0.11
							0.019	0.078	< 0.01	0.01	0.11
											0.11 (mean)
						21	0.013	0.080	< 0.01	0.016	0.11
							0.014	0.063	< 0.01	0.012	0.089
						28	0.018	0.068	< 0.01	0.011	0.097
							0.013	0.071	< 0.01	0.014	0.098
						35	0.015	0.078	< 0.01	0.01	0.10
							0.011	0.071	< 0.01	0.011	0.093
RV208-11HA, Fremont, Michigan USA, 2011 (Canada)	2	47	202	210	Root	7	0.014	0.15	< 0.01	0.026	0.19
	(10)	48	202	212			0.026	0.19	< 0.01	0.029	0.24
							0.020 (mean)				0.21 (mean)
					RAC	7	0.011 (av.)	0.16 (av.)	< 0.01 (av.)	0.027 (av.)	0.19
					Root, washed	7	< 0.01 (av.)	0.16 (av.)	< 0.01 (av.)	0.026 (av.)	0.20
					Root, cooked	7	< 0.01 (av.)	0.13 (av.)	< 0.01 (av.)	0.019 (av.)	0.16
RV209-11HA, Elm Creek, Manitoba Canada, 2011 (Danvers)	2	46	192	94	Root	7	< 0.01	0.19	< 0.01	0.026	0.23
	(8)	47	208	101			< 0.01	0.23	< 0.01	0.019	0.26
							< 0.01 (mean)				0.24 (mean)
RV210-11HA,	2	46	204	210	Root	7	< 0.01	0.44	< 0.01	0.019	0.47
Hinton, Oklahoma	(9)	47	210	177			< 0.01	0.42	< 0.01	0.021	0.45
USA, 2011							< 0.01				0.46
(Nantes Scarlet)							(mean)				(mean)
RV211-11DA, Madera, California	2	45	207	235	Root	0	0.046	0.19	< 0.01	< 0.01	0.23
	(10)	49	208	241			0.061	0.22	< 0.01	< 0.01	0.28
						7	0.048	0.44	< 0.01	0.018	0.50

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
USA, 2011 (Danvers 126)							0.025	0.45	< 0.01	0.016	0.49
							0.037 (mean)				
						14	0.018	0.46	< 0.01	0.01	0.48
							0.026	0.55	< 0.01	0.019	0.59
						21	0.015	0.54	< 0.01	< 0.01	0.56
							0.019	0.57	< 0.01	< 0.01	0.59
						28	0.018	0.53	< 0.01	< 0.01	0.55
							0.017	0.63	< 0.01	< 0.01	0.65
											0.60 (mean)
						35	< 0.01	0.40	< 0.01	< 0.01	0.41
							< 0.01	0.45	< 0.01	< 0.01	0.45
RV212-11HA, Sanger, California USA, 2011 (Vitana)	2	47	221	201	Root	7	0.061	< 0.05	< 0.01	0.016	0.13
	(11)	48	206	187			0.056	< 0.05	< 0.01	0.018	0.12
							0.059 (mean)				0.13 (mean)
					RAC	7	0.05 (av.)	< 0.05 (av.)	< 0.01 (av.)	0.018 (av.)	0.12
					Root, washed	7	0.040 (av.)	< 0.05 (av.)	< 0.01 (av.)	0.015 (av.)	0.11
					Root, cooked	7	0.041 (av.)	< 0.05 (av.)	< 0.01 (av.)	0.015 (av.)	0.11
RV213-11HA,	2	48	203	269	Root	6	0.028	0.17	< 0.01	0.022	0.22
Porterville, Oregon	(9)	48	207	250			0.025	0.19	< 0.01	0.025	0.24
USA, 2011							0.027				0.23
(Danver's No.126)							(mean)				(mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 119 Residues from the foliar application of flupyradifurone to radishes in the USA and Canada (Murphy 2012, RARVY039)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Subgroup 1B	2 (10)		205			7					
RV214-11HA, North Rose, New York USA, 2011 (Agora)	2	13	207	236	Tuber	7	0.036	0.12	< 0.01	0.040	0.20
	(10)	43	206	234			0.025	0.12	< 0.01	0.036	0.18
							0.031 (mean)				0.19 (mean)
RV215-11DA, Alachua, Florida USA, 2011 (Cherriette)	2	49	202	252	Tuber	0	0.12	0.13	< 0.01	0.041	0.29
	(10)	49	203	253			0.14	0.14	< 0.01	0.034	0.31
						7	0.054	0.19	< 0.01	0.053	0.29
							0.037	0.18	< 0.01	0.054	0.27
							0.046 (mean)				0.28 (mean)
						14	< 0.01	0.14	< 0.01	0.031	0.18
							< 0.01	0.12	< 0.01	0.026	0.16
						21	< 0.01	0.12	< 0.01	0.019	0.15
							0.015	0.11	< 0.01	0.017	0.14
						28	0.011	0.093	< 0.01	0.016	0.12

Trial No., Location, Year (Variety)	Application No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)	Sample	DALA	Residues as parent (mg/kg) Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							0.012	0.11	< 0.01	0.014	0.14
						35	< 0.01	0.062	< 0.01	0.01	0.082
							< 0.01	0.081	< 0.01	0.012	0.10
RV216-11HA,	2	14	200	276	Tuber	6	0.055	0.21	< 0.01	0.057	0.32
Oviedo, Florida	(10)	42	202	279			0.031	0.21	< 0.01	0.063	0.30
USA, 2011 (Pink Beauty OG)							0.043 (mean)				0.31 (mean)
RV217-11DA,	2	41	206	201	Tuber	0	0.043	< 0.05	< 0.01	0.017	0.11
Stewardson, Illinois	(8)	49	209	240			0.064	0.053	< 0.01	0.017	0.13
USA, 2011						7	0.046	0.053	< 0.01	0.026	0.13
(Early Scarlet Globe)							0.027	< 0.05	< 0.01	0.026	0.10
							0.037 (mean)				
						14	0.033	< 0.05	< 0.01	0.014	0.098
							0.023	< 0.05	< 0.01	0.018	0.091
						20	0.039	0.056	< 0.01	0.018	0.11
							0.033	0.056	< 0.01	0.017	0.11
											0.11 (mean)
						27	0.028	0.058	< 0.01	0.012	0.098
							0.0 26	0.062	< 0.01	0.013	0.10
						33	0.016	0.085	< 0.01	0.011	0.13
							0.014	0.061	< 0.01	< 0.01	0.074
RV218-11HA,	2	42	211	142	Tuber	6	0.024	0.063	< 0.01	0.020	0.11
Lenexa, Kansas	(8)	47	210	147			0.023	0.059	< 0.01	0.022	0.10
USA, 2011							0.024				0.11
(Champion)							(mean)				(mean)
RV219-11HA,	2	12	205	175	Tuber	7	0.030	< 0.05	< 0.01	0.021	0.10
Portage la Prairie, Manitoba	(8)	41	206	174			0.028	< 0.05	< 0.01	0.025	0.10
Canada, 2011							0.029				0.10
(Champion)							(mean)				(mean)
RV220-11HA,	2	44	204	237	Tuber	7	0.039	0.060	< 0.01	0.018	0.12
Nipomo, California	(10)	49	205	244			0.041	0.062	< 0.01	0.015	0.12
USA, 2011							0.040				0.12
(Red Satin)							(mean)				(mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Twenty-six supervised trials were carried out on potatoes (Table 120) in the USA and Canada during the 2010 growing season (Krolski and Harbin 2012b, RARVY015) to measure the magnitude of residues in/on potatoes following two broadcast foliar spray applications of BYI 02960 200 SL. All applications were made using ground-based equipment. All applications included a non-ionic surfactant adjuvant at a rate of 0.25% v/v.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in potatoes were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data (mean recoveries, RSDs) were obtained for all analytes.

Table 120 Residues from the foliar application of flupyradifurone to potatoes in the USA and Canada (Krolski and Harbin 2012b, RARVY015)

Trial No., Location, Year (Variety)	Application No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)	Sample	DALA	Residues as parent (mg/kg) Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Subgroup 1C	2 (7)		205			7					
RV178-10DA,	2	91	208	280	Tuber	0	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Alton, New York	(8)	93	206	280			0.011	< 0.05	0.012	< 0.01	0.061
USA, 2010						3	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Superior)							0.015	< 0.05	< 0.01	< 0.01	0.065
						7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							0.011	< 0.05	< 0.01	< 0.01	0.061
						21	0.012	< 0.05	< 0.01	< 0.01	0.062
							0.012	< 0.05	< 0.01	< 0.01	0.062
							0.012 (mean)				0.062 (mean)
RV200-10HA,	2	91	205	280	Tuber	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Alton, New York	(8)	93	205	280			0.014	< 0.05	< 0.01	< 0.01	0.064
USA, 2010							0.012				0.062
(Reba)							(mean) c0.01				(mean)
RV179-10HA,	2	91	201	280	Tuber	6	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
North Rose, New York	(8)	91	201	280			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(Carola)							(mean)				(mean)
RV182-10HA,	2	89	207	340	Tuber	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
North Rose, New York	(7)	91	208	350			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(NorDonna)							(mean)				(mean)
RV183-10HA,	2	89	205	340	Tuber	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
North Rose, New York	(7)	91	209	350			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(NY-129)							(mean)				(mean)
RV180-10HA,	2	81	212	190	Tuber	8	< 0.01	0.087	< 0.01	< 0.01	0.097
Germansville, Pennsylvania	(7)	89	205	190			< 0.01	0.085	< 0.01	< 0.01	0.095
USA, 2010							< 0.01				0.096
(Dark Red Norland)							(mean)				(mean)
RV181-10HA,	2	85	221	300	Tuber	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Frenchtown, New Jersey	(8)	91	211	260			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(Dark Red Norland)							(mean)				(mean)
RV184-10HA,	2	71	200	260	Tuber	7	< 0.01	0.083	< 0.01	< 0.01	0.093
Pikeville, North Carolina	(7)	74	206	280			< 0.01	0.057	< 0.01	< 0.01	0.067
USA, 2010							< 0.01				0.080
(Snowden)							(mean)				(mean)
RV185-10HA,	2	91	205	280	Tuber	7	0.021	< 0.05	< 0.01	< 0.01	0.071
Oviedo, Florida	(7)	93	206	280			0.018	< 0.05	< 0.01	< 0.01	0.068
USA, 2010							0.020				0.070
(Kennebec)							(mean)				(mean)
RV186-10HA,	2	93	206	180	Tuber	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Richland, Iowa	(7)	93	204	190			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(Kennebec)							(mean)				(mean)
RV187-10HA,	2	45	204	190	Tuber	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Clarence, Missouri	(7)	45	204	190			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(Kennebec)							(mean)				(mean)
RV188-10HA,	2	93	200	210	Tuber	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Stewardson, Illinois	(8)	95	209	240			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(Kennebec)							(mean)				(mean)
RV189-10HA,	2	91	206	120	Tuber	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Rockwood, Ontario	(6)	93	207	130			0.01	< 0.05	< 0.01	< 0.01	0.060
Canada, 2010							0.01				0.060
(Russet Burbank)							(mean)				(mean)
RV190-10HA,	2	49	211	140	Tuber	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Taber, Alberta	(8)	49	203	140			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Canada, 2010							< 0.01				< 0.060
(Russet Burbank)							(mean)				(mean)
RV191-10HA,	2	91	206	210	Tuber	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Jerome, Idaho	(7)	93	201	200			< 0.01	< 0.05	< 0.01	0.022	0.082
USA, 2010							< 0.01				0.071
(Ranger Russet)							(mean)				(mean)
RV192-10HA,	2	85	202	370	Tuber	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Sanger, California	(7)	91	201	370			0.034	< 0.05	< 0.01	< 0.01	0.084
USA, 2010							0.022				0.072
(Red La Soda)							(mean)				(mean)
RV193-10HA,	2	91	206	280	Tuber	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Payette, Idaho	(6)	91	211	290			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(Dark Red Norland)							(mean)				(mean)
RV195-10DA,	2	91	206	280	Tuber	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Payette, Idaho	(7)	95	207	280			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(Russet Norkotah)							(mean)				(mean)
RV194-10HA,	2	93	209	160	Tuber	6	< 0.01	< 0.05	< 0.01	0.019	0.079
Rupert, Idaho	(8)	95	212	160			< 0.01	< 0.05	< 0.01	0.028	0.088
US, 2010							< 0.01				0.083
(Russet Burbank)							(mean) c 0.011				(mean)
RV202-10DA,	2	47	205	110	Tuber	0	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Rupert,	(7)	91	206	110			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Idaho						3	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Ranger Russet)						7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						14	< 0.01	0.073	< 0.01	0.016	0.10
							0.01	0.10	< 0.01	0.023	0.14
							0.01 (mean)				0.12 (mean)
						21	< 0.01	< 0.05	< 0.01	0.013	0.073
							< 0.01	0.11	< 0.01	0.021	0.14
RV196-10HA,	2	48	204	230	Tuber	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Ephrata, Washington	(7)	48	209	240			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(Umatilla)							(mean)				(mean)
RV197-10HA,	2	47	208	190	Tuber	7	0.057	< 0.05	< 0.01	< 0.01	0.11
Ephrata, Washington	(7)	48	204	190			0.016	< 0.05	< 0.01	< 0.01	0.066
USA, 2010							0.037				0.088
(Norkotah)							(mean)				(mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RV198-10HA,	2	42	213	200	Tuber	7	0.046	< 0.05	< 0.01	0.037	0.13
Abbotsford, British Columbia	(7)	43	206	200			0.027	< 0.05	< 0.01	0.027	0.10
Canada, 2010							0.037				0.12
(Russet Burbank)							(mean)				(mean)
RV199-10HA,	2	89	209	160	Tuber	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Minto, Manitoba	(7)	91	212	160			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Canada, 2010							< 0.01				< 0.060
(Norland)							(mean)				(mean)
RV201-10DA,	2	91	209	150	Tuber	0	0.040	< 0.05	< 0.01	< 0.01	0.090
Gardner,	(7)	93	211	150			0.021	< 0.05	< 0.01	< 0.01	0.071
Kansas						3	0.012	< 0.05	< 0.01	< 0.01	0.062
USA, 2010							0.016	< 0.05	< 0.01	< 0.01	0.066
(Norland Red)						7	0.018	< 0.05	< 0.01	< 0.01	0.068
							0.019	< 0.05	< 0.01	< 0.01	0.069
						14	0.029	< 0.05	< 0.01	< 0.01	0.079
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							0.020 (mean)				0.070 (mean)
						21	0.016	< 0.05	< 0.01	< 0.01	0.066
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV203-10DA,	2	49	209	100	Tuber	0	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Josephburg,	(7)	49	207	100			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Alberta						3	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Canada, 2010							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Russet Nacota)						6	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01				< 0.060
							(mean)				(mean)
						13	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						19	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Stalk and stem vegetables

Ten supervised trials were carried out on celery (Table 121) in the USA and Canada during the 2011 growing season (Netzband and Niczyporowicz 2012, RARVY005) to measure the magnitude of residues in celery stalks and trimmed celery stalks following two broadcast foliar spray applications of BYI 02960 200 SL. All applications were made using ground-based equipment. An adjuvant [non-ionic surfactant at 0.20% v/v, methylated seed oil at 0.25% v/v or crop oil concentrate 1.0% v/v] was used in all applications.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in celery stalk and trimmed celery stalk were determined using LC-MS/MS method 01304. Acceptable validation and concurrent recovery data were obtained for all analytes.

Table 121 Residues from foliar application of flupyradifurone to celery in the USA and Canada (Netzband and Niczyporowicz 2012, RARVY005)

Trial No., Location,	Application	Sample	DALA	Residues as parent (mg/kg)

Year (Variety)	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 4	2 (7)		205			1					
RV027-11DA,	2	45	206	281	Stalk	–6	0.59	< 0.05	< 0.01	0.094	0.73
Gregory, Michigan,	(7)	45	206	266			1.1	< 0.05	0.014	0.05	1.2
USA, 2011						0	1.2	0.05	0.01	< 0.01	1.3
(Tall Utah)							0.10	< 0.05	< 0.01	< 0.01	0.15
						1	0.62 c1.2	< 0.05 c0.063	< 0.01 c0.012	< 0.01	0.67
							0.49 c1.2	< 0.05 c0.063	< 0.01 c0.012	< 0.01	0.54
						7	0.32	0.059	< 0.01	< 0.01	0.38
							0.33	< 0.05	< 0.01	< 0.01	0.38
						14	0.16	0.078	< 0.01	< 0.01	0.24
							0.19	0.066	< 0.01	< 0.01	0.25
						21	0.040	0.12	< 0.01	< 0.01	0.16
							0.16	0.14	< 0.01	< 0.01	0.30
						28	0.049	0.153	< 0.01	< 0.01	0.20
							0.049	0.128	< 0.01	< 0.01	0.18
					Stalk,	–6	0.081	< 0.05	< 0.01	< 0.01	0.13
					Trimmed		0.091	< 0.05	< 0.01	< 0.01	0.14
						1	0.11	< 0.05	< 0.01	< 0.01	0.16
							0.11	< 0.05	< 0.01	< 0.01	0.16
RV028-11DA,	2	43	207	176	Stalk	–6	0.055	< 0.05	< 0.01	< 0.01	0.10
Sparta, Michigan,	(7)	45	207	175			0.057	< 0.05	< 0.01	< 0.01	0.11
USA, 2011						0	1.7	< 0.05	< 0.01	< 0.01	1.8
(Green Bay)							2.2	< 0.05	< 0.01	< 0.01	2.3
						1	0.27	< 0.05	< 0.01	< 0.01	0.32
							0.17	< 0.05	< 0.01	< 0.01	0.22
							0.22 (mean)				0.27 (mean)
						7	0.065	< 0.05	< 0.01	< 0.01	0.12
							0.059	< 0.05	< 0.01	< 0.01	0.11
						14	0.029	< 0.05	< 0.01	< 0.01	0.079
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						21	0.032	< 0.05	< 0.01	< 0.01	0.082
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						28	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
					Stalk,	–1	0.018	< 0.05	< 0.01	< 0.01	0.068
					Trimmed		0.022	< 0.05	< 0.01	< 0.01	0.072
						1	0.14	< 0.05	< 0.01	< 0.01	0.19
							0.13	< 0.05	< 0.01	< 0.01	0.18
											0.19 (mean)
RV029-11DA,	2	45	206	311	Stalk	–6	2.1	< 0.05	< 0.01	< 0.01	2.1
Porterville, California,	(7)	45	204	263			2.4	< 0.05	< 0.01	< 0.01	2.4
USA, 2011						0	3.8	< 0.05	0.023	< 0.01	3.8
(Command)							2.4	< 0.05	0.015	< 0.01	2.5
						1	2.3	< 0.05	0.021	< 0.01	2.4
							2.4	< 0.05	0.020	< 0.01	2.5
							2.4 (mean)				2.4 (mean)
						7	0.99	< 0.05	0.019	< 0.01	1.0
							1.6	< 0.05	0.020	< 0.01	1.6

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
						14	0.68	< 0.05	0.012	< 0.01	0.73
							0.67	< 0.05	0.011	< 0.01	0.72
						21	0.55	< 0.05	0.01	< 0.01	0.60
							0.60	< 0.05	< 0.01	< 0.01	0.65
						28	0.46	< 0.05	< 0.01	< 0.01	0.51
							0.37	< 0.05	< 0.01	< 0.01	0.42
					Stalk,	–6	0.35	< 0.05	< 0.01	< 0.01	0.40
					Trimmed		0.37	< 0.05	< 0.01	< 0.01	0.42
						1	0.32	< 0.05	< 0.01	< 0.01	0.37
							0.28	< 0.05	< 0.01	< 0.01	0.33
											0.35 (mean)
RV030-11DB,	2	45	206	187	Stalk	–6	1.5	< 0.05	< 0.01	< 0.01	1.6
Guadulupe, California,	(7)	47	209	190			1.4	< 0.05	< 0.01	< 0.01	1.5
USA, 2011						0	4.3	< 0.05	0.025	< 0.01	4.3
(Conquistador)							3.2	< 0.05	0.020	< 0.01	3.3
						1	3.2	< 0.05	0.024	< 0.01	3.2
							3.2	< 0.05	0.024	< 0.01	3.2
							3.2 (mean)				3.2 (mean)
						6	1.5	< 0.05	0.015	< 0.01	1.6
							1.8	< 0.05	0.022	< 0.01	1.9
						14	0.68	< 0.05	< 0.01	< 0.01	0.73
							1.2	< 0.05	0.013	< 0.01	1.2
						21	0.58	< 0.05	0.01	< 0.01	0.63
							0.37	< 0.05	< 0.01	< 0.01	0.42
						28	0.26	< 0.05	< 0.01	< 0.01	0.31
							0.29	< 0.05	< 0.01	< 0.01	0.34
					Stalk,	–6	0.23	< 0.05	< 0.01	< 0.01	0.28
					Trimmed		0.60	< 0.05	< 0.01	< 0.01	0.65
						1	0.58	< 0.05	< 0.01	< 0.01	0.63
							0.84	< 0.05	0.01	< 0.01	0.89
											0.76 (mean)
RV031-11HA,	2	49	202	275	Stalk	–6	2.4	< 0.05	< 0.01	< 0.01	2.5
Oviedo, Florida,	(7)	49	203	277			1.6	< 0.05	< 0.01	< 0.01	1.7
USA, 2011						1	1.7	< 0.05	< 0.01	< 0.01	1.7
(Tango)							2.6	< 0.05	0.016	< 0.01	2.6
							2.1 (mean)				2.2 (mean)
					Stalk,	–6	0.44	< 0.05	< 0.01	< 0.01	0.49
					Trimmed		0.38	< 0.05	< 0.01	< 0.01	0.43
						1	0.86	< 0.05	< 0.01	< 0.01	0.91
							0.54	< 0.05	< 0.01	< 0.01	0.59
											0.75 (mean)
RV032-11HA, Elm Creek, Manitoba, Canada, 2011 (–)	2	47	198	97	Stalk	1	1.8	< 0.05	0.019	0.016	1.9
	(7)	49	204	99			2.1	< 0.05	0.016	0.012	2.2
							2.0 (mean)			c0.020	2.0 (mean)
					Stalk,	1	0.066	< 0.05	< 0.01	< 0.01	0.12
					Trim		0.035	< 0.05	< 0.01	< 0.01	0.085

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
					med						
											0.10 (mean)
RV033-11HC,	2	45	203	212	Stalk	–6	1.7	< 0.05	< 0.01	< 0.01	1.8
Manchester,	(7)	47	210	217			1.7	< 0.05	< 0.01	< 0.01	1.8
Michigan,						1	0.97	< 0.05	< 0.01	< 0.01	1.0
USA, 2011							1.2	< 0.05	< 0.01	< 0.01	1.3
(Tall Utah)							1.1 (mean)				1.1 (mean)
					Stalk,	–6	0.052	< 0.05	< 0.01	< 0.01	0.10
					Trimmed		0.071	< 0.05	< 0.01	< 0.01	0.12
						1	0.045	< 0.05	< 0.01	< 0.01	0.095
							0.10	< 0.05	< 0.01	< 0.01	0.15
											0.12 (mean)
RV034-11HA,	2	49	205	175	Stalk	–4	2.9	< 0.05	0.014	< 0.01	2.9
St Eustache,	(5)	49	209	178			2.1	< 0.05	< 0.01	< 0.01	2.2
Manitoba,						1	3.7	< 0.05	0.027	< 0.01	3.7
Canada, 2011							3.4	< 0.05	0.022	< 0.01	3.4
(Utah Salt Lake)							3.5 (mean)				3.6 (mean)
					Stalk,	–4	0.62	< 0.05	< 0.01	< 0.01	0.67
					trimmed		0.99	< 0.05	< 0.01	< 0.01	1.0
						1	0.74	< 0.05	< 0.01	< 0.01	0.79
							0.74	< 0.05	< 0.01	< 0.01	0.79
											0.79 (mean)
RV035-11HA,	2	49	208	238	Stalk	–5	5.0	< 0.05	0.030	< 0.01	5.0
Madera, California,	(6)	49	207	238			4.7	< 0.05	0.028	0.012	4.8
USA, 2011						1	6.7	0.064	0.054	0.028	6.8
(Sonora)							5.3	0.058	0.046	0.025	5.4
							6.0 (mean)				6.1 (mean)
					Stalk,	–5	0.76	< 0.05	< 0.01	< 0.01	0.81
					trimmed		0.96	< 0.05	< 0.01	< 0.01	1.0
						1	0.75	< 0.05	< 0.01	< 0.01	0.80
							0.83	< 0.05	< 0.01	< 0.01	0.88
											0.84 (mean)
RV036-11HA,	2	45	208	187	Stalk	–7	5.6	< 0.05	0.026	0.013	5.7
Corning, California,	(8)	47	207	188			5.9	< 0.05	0.021	< 0.01	6.0
USA, 2011						1	2.2	< 0.05	0.038	0.016	2.2
(Sonora)							2.2	< 0.05	0.029	0.022	2.3
							2.2 (mean)				2.2 (mean)
					Stalk,	–7	0.69	< 0.05	< 0.01	< 0.01	0.74
					trimmed		0.60	< 0.05	< 0.01	< 0.01	0.65
						1	0.53	< 0.05	< 0.01	0.013	0.59
							0.39	< 0.05	< 0.01	< 0.01	0.44
											0.51 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Cereal grains

Twenty supervised trials were carried out on barley (Table 122) in the USA during the 2010–2011 growing seasons (Hoag 2012a, RARVY001) to measure the magnitude of residues in/on barley grain, barley hay and barley straw following two broadcast foliar spray applications of BYI 02960 200 SL, or by planting seed treated with BYI 02960 480 FS. Barley seed was treated at a nominal rate of 0.25 kg/100 kg seed. All foliar applications were made using ground-based equipment. Adjuvants were used in all foliar applications at a rate of 0.25% v/v.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) were determined in barley grain, hay and straw using LC/MS/MS Method 01304. Acceptable concurrent recovery data were obtained for all analytes.

Residues in barley hay and straw are shown in Table 135.

Table 122 Residues from foliar or seed treatment application of flupyradifurone to barley in the USA and Canada (Hoag 2012a, RARVY001)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 15	2 (7)		205			21					
RV001-10HA,	2	77	207	175	Grain	16	0.85	0.34	0.14	0.17	1.4
Germansville,	(6)	85	207	175			0.77	0.40	0.16	0.18	1.3
Pennsylvania							0.81				1.4
USA, 2010 (AC Minoa)							(mean)				(mean)
RV002-10HA,	2	71	208	137	Grain	19	0.069	1.1	0.035	0.24	1.4
Springfield,	(5)	83	206	134			0.061	1.2	0.034	0.30	1.5
Nebraska USA, 2010 (Robust)							0.065 (mean)				1.5 (mean)
	1	0	123		Grain	92	< 0.01	0.47	< 0.01	< 0.01	0.48
	(Seed)						< 0.01	0.52	< 0.01	< 0.01	0.53
RV003-10DA,	2	75	209	255	Grain	10	3.7	0.14	0.96	0.16	4.0
Carlyle, Illinois	(7)	83	208	248			4.6	0.13	0.10	0.21	5.0
USA, 2010						15	2.2	0.075	0.028	0.037	2.3
(–)							1.8	0.063	0.029	0.024	1.9
						20	1.3	0.084	0.025	0.015	1.4
							1.1	0.078	0.025	0.013	1.1
							1.2 (mean)				1.3 (mean)
						28	0.95	0.057	0.011	0.011	1.0
							0.99	0.071	0.017	0.01	1.1
						35	0.57	0.067	0.016	< 0.01	0.63
							0.69	0.080	0.016	0.026	0.80
RV004-10HA,	2	83	202	109	Grain	21	2.2	0.082	0.034	0.093	2.3
Rockwood, Ontario	(7)	85	206	114			2.4	0.089	0.043	0.097	2.5
Canada, 2010							2.3				2.4
(Dignity)							(mean)				(mean)
RV005-10HA,	2	75	207	190	Grain	22	0.42	0.28	0.023	0.097	0.79

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Grand Island, Nebraska USA, 2010 (Robust)	(7)	85	205	190			0.47	0.24	0.025	0.083	0.79
							0.44 (mean)				0.79 (mean)
	1 (Seed)	0	202		Grain	110	< 0.01	0.69	< 0.01	< 0.01	0.70
							< 0.01	0.69	< 0.01	< 0.01	0.70
RV006-10HA,	2	77	204	187	Grain	10	0.50	0.43	0.14	0.25	1.2
Carrington, North Dakota	(5)	83	204	186			0.51	0.41	0.15	0.19	1.1
USA, 2010						15	0.52	0.46	0.11	0.23	1.2
(Pinneacle)							0.59	0.44	0.13	0.20	1.2
						21	0.34	0.30	0.13	0.072	0.71
							0.35	0.32	0.14	0.070	0.74
						29	0.44	0.31	0.15	0.092	0.84
							0.49	0.33	0.13	0.10	0.92
							0.46 (mean)				0.88 (mean)
						35	0.28	0.22	0.083	0.080	0.57
							0.22	0.20	0.067	0.067	0.49
RV007-10HA,	2	83	203	187	Grain	21	0.48	0.17	0.10	0.083	0.73
Oberon, North Dakota	(5)	85	209	190			0.47	0.19	0.11	0.084	0.74
USA, 2010							0.48				0.74
(Pinneacle)							(mean)				(mean)
RV008-10HA,	2	83	208	189	Grain	19	0.59	0.084	0.038	0.047	0.72
Jamestown, North Dakota	(5)	85	208	191			0.76	0.11	0.047	0.068	0.94
USA, 2010							0.68				0.83
(Tradition)							(mean)				(mean)
RV009-10HA,	2	83	207	188	Grain	20	0.76	< 0.05	0.035	0.016	0.82
Jerome, Idaho	(7)	87	210	192			0.93	< 0.05	0.031	0.019	1.0
USA, 2010							0.84				0.91
(Harrington)							(mean)				(mean)
RV010-10HA,	2	85	204	289	Grain	21	1.9	0.082	0.051	0.081	2.0
Sanger, California	(7)	87	207	300			1.5	0.073	0.043	0.069	1.6
USA, 2010							1.7				1.8
(UC937)							(mean)				(mean)
RV011-10HA,	2	85	205	280	Grain	21	0.80	< 0.05	0.048	0.020	0.87
Ephrata, Washington	(7)	87	207	282			0.63	< 0.05	0.055	0.020	0.70
USA, 2010							0.71				0.78
(AC Metcalfe)							(mean)				(mean)
RV012-10HA, Payette, Idaho USA, 2010 (Champion)	2	73	205	233	Grain	21	0.21	0.34	0.068	0.042	0.59
	(7)	77	209	238			0.21	0.27	0.058	0.036	0.52
							0.21 (mean)				0.55 (mean)
	1	00	195		Grain	101	< 0.01	0.23	< 0.01	< 0.01	0.24
	(Seed						< 0.01	0.22	< 0.01	< 0.01	0.23

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RV013-10DA,	2 (8)	73	206	101	Grain	10	0.47	0.59	0.073	0.17	1.2
Josephburg, Alberta		85	206	99			0.38	0.59	0.077	0.14	1.1
Canada, 2010						13	0.28	0.63	0.071	0.13	1.0
(Coalition)							0.24	0.65	0.073	0.14	1.0
						19	0.29 c0.023	0.70	0.064	0.13	1.1
							0.29	0.68	0.058	0.13	1.1
						27	0.31	0.66	0.062	0.15	1.1
							0.30	0.66	0.057	0.14	1.1
						34	0.36	0.85	0.068	0.20	1.4
							0.25	0.79	0.068	0.15	1.2
							0.31 (mean)				1.3 (mean)
RV014-10DA,	2	65	211	207	Grain	9	0.35	0.43	0.046	0.13	0.91
Alvena,	(5)	72	195	192			0.35	0.41	0.045	0.12	0.88
Saskatchewan						15	0.31	0.60	0.048	0.16	1.1
Canada, 2010							0.35	0.55	0.05	0.13	1.0
(Metcalf)						20	0.22	0.43	0.037	0.076	0.73
							0.31	0.51	0.042	0.097	0.92
							0.27 (mean)				0.82 (mean)
						28	0.18	0.36	0.031	0.06	0.60
							0.14	0.33	0.028	0.04	0.51
						34	0.13	0.39	0.035	0.020	0.54
							0.13	0.40	0.031	0.021	0.55
RV015-10HA,	2	58	201	198	Grain	21	0.043	0.54	0.017	0.12	0.70
Wakaw, Saskatchewan	(6)	64	201	197			0.033	0.51	< 0.01	0.080	0.63
Canada, 2010							0.038				0.67
(Ranger)							(mean)				(mean)
RV016-10HA,	2	83	204	158	Grain	21	0.26	0.50	0.10	0.041	0.80
Minto, Manitoba	(7)	87	204	158			0.34	0.58	0.11	0.061	0.98
Canada, 2010							0.30				0.89
(CDC Copeland)							(mean)				(mean)
RV017-10HA,	2	77	204	159	Grain	21	0.084	0.97	0.041	0.13	1.2
Boissevain, Manitoba	(7)	83	209	162			0.11	1.2	0.055	0.14	1.5
Canada, 2010							0.096				1.3
(Metcalfe)							(mean)				(mean)
RV018-10HA,	2	65	203	198	Grain	21	0.25	0.43	0.062	0.042	0.72
Wellwood, Manitoba	(7)	71	198	193			0.23	0.37	0.063	0.048	0.65
Canada, 2010							0.24				0.69
(Tradition)							(mean)				(mean)
RV019-10HA,	2	77	204	100	Grain	19	0.76	0.33	0.092	0.12	1.2
Fort Saskatchewan, Alberta	(8)	85	199	98			0.61	0.34	0.075	0.10	1.0
Canada, 2010							0.68				1.1

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
(Coalition)							(mean)				(mean)
RV020-10HA,	2	59	207	201	Grain	20	0.23	0.24	0.028	0.043	0.51
Waldheim, Saskatchewan	(7)	77	210	204			0.27	0.24	0.025	0.042	0.55
Canada, 2010							0.25				0.53
(Metcalf)							(mean)				(mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Twenty-nine supervised trials were carried out on wheat (Table 123) in the USA and Canada during the 2010 growing season (Fischer and Niczyporowicz 2012, RARVY003) to measure the magnitude of residues in/on wheat grain, forage, hay and straw following two broadcast foliar spray applications of BYI 02960 200 SL, or, in three trials, by planting seed treated with BYI 02960 480 FS. Wheat seed was treated at a nominal rate of 105 g/100 kg seed. All foliar applications were made using ground-based equipment. Adjuvants were used in all foliar applications at a rate of 0.25% v/v.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) were determined in wheat grain, forage, hay and straw using LC/MS/MS Method 01304. Acceptable concurrent recovery data were obtained for all analytes.

Residues in wheat forage, hay and straw are shown in Table 136.

Table 123 Residues from foliar or seed treatment application of flupyradifurone to wheat in the USA and Canada (Fischer and Niczyporowicz 2012, RARVY003)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 15	2 (7)		205			21					
RV054-10HA,	2	75	209	290	Grain	20	0.586	0.103	< 0.01	0.030	0.72
Seven Springs, North Carolina	(6)	83	206	290			0.626	0.161	< 0.01	0.037	0.82
USA, 2010							0.61				0.77
(Pioneer 26R15)							(mean)				(mean)
RV055-10HA,	2	77	207	180	Grain	21	0.167	0.116	< 0.01	0.013	0.30
Cheneyville, Louisiana	(7)	85	206	180			0.125	0.118	< 0.01	0.012	0.25
USA, 2011							0.15				0.27
(Terral Brand LA821)							(mean)				(mean)
RV056-10HA, Stafford, Kansas USA, 2010 (Found Juniper)	2	69	209	190	Grain	21	0.088	0.257	< 0.01	0.012	0.36
	(6)	75	209	190			0.118	0.282	< 0.01	0.013	0.41
							0.10 (mean)				0.38 (mean)
	1	0	128			252	< 0.01	0.538	< 0.01	< 0.01	0.55
	(Seed)						< 0.01	0.641	< 0.01	< 0.01	0.65
RV057-10HA,	2	77	201	140	Grai	21	0.331	0.438	< 0.01	0.018	0.79

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
					n						
Gardner, Kansas	(7)	83	206	150			0.342	0.413	< 0.01	0.022	0.78
USA, 2010							0.34				0.78
(Winter Hawk)							(mean)				(mean)
RV058-10HA,	2	83	197	110	Grain	21	0.586	0.278	< 0.01	0.034	0.90
Rockwood, Ontario	(7)	85	205	110			0.583	0.289	< 0.01	0.036	0.91
Canada, 2010							0.58				0.90
(Glenn (Hard Red, Spring))							(mean)				(mean)
RV059-10HA,	2	77	205	190	Grain	21	0.078	0.943	< 0.01	0.061	1.1
Campbell, Minnesota	(7)	83	205	190			0.101	1.03	< 0.01	0.082	1.2
USA, 2010							0.090				1.1
(RB07)							(mean)				(mean)
RV060-10DA,	2 (6)	77	206	190	Grain	10	0.186	1.19	< 0.01	0.033	1.4
Fisk, Missouri		83	206	190			0.196	1.13	< 0.01	0.038	1.4
USA, 2010						15	0.119	1.56	< 0.01	0.073	1.8
(Beretta)							0.082	1.51	< 0.01	0.064	1.7
						21	0.169	1.52	< 0.01	0.101	1.8
							0.153	1.35	< 0.01	0.091	1.6
							0.16 (mean)				
						28	0.136	1.31	< 0.01	0.052	1.5
							< 0.01	1.11	< 0.01	0.039	1.2
						35	0.157	1.60	< 0.01	0.055	1.8
							0.172	1.72	< 0.01	0.059	1.9
											1.9 (mean)
RV061-10HA,	2	85	207	270	Grain	21	0.259	0.079	< 0.01	< 0.01	0.34
East Bernard, Texas	(7)	87	203	270			0.203	< 0.05	< 0.01	< 0.01	0.25
USA, 2010							0.23				0.30
(Fannin)							(mean)				(mean)
RV062-10HA, Grand Island, Nebraska USA, 2010 (Traverse)	2	71	206	190	Grain	22	0.038	1.37	< 0.01	0.10	1.5
	(7)	73	207	190			0.030	1.47	< 0.01	0.11	1.6
							0.034 (mean)				1.6 (mean)
	1	00	113		Grain	103	< 0.01	0.276	< 0.01	< 0.01	0.29
	(Seed)						< 0.01	0.266	< 0.01	< 0.01	0.28
RV066-10DA,	2 (7)	71	205	190	Grain	9	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Grand Island, Nebraska		83	205	180			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2011						14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Overland HRW)							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						20	0.118	0.330	< 0.01	0.013	0.46
							0.174	0.332	< 0.01	0.013	0.52
							0.15 (mean)				

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
						27	0.138	0.485	< 0.01	0.013	0.64
							0.152	0.522	< 0.01	0.018	0.69
											0.66 (mean)
						35	0.099	0.375	< 0.01	< 0.01	0.47
							0.089	0.397	< 0.01	0.011	0.50
RV063-10HA,	2	73	204	190	Grain	21	0.058	0.811	< 0.01	0.036	0.90
Carrington, North Dakota	(5)	77	205	190			0.060	0.863	< 0.01	0.041	0.96
USA, 2010							0.059				0.93
(Faller)							(mean)				(mean)
RV064-10HA,	2	77	205	190	Grain	20	0.171	0.446	< 0.01	0.019	0.64
Jamestown, North Dakota	(6)	83	207	180			0.158	0.517	< 0.01	0.024	0.70
USA, 2010							0.16				0.67
(Oklee)							(mean)				(mean)
RV065-10HA,	2	73	197	180	Grain	21	0.074	0.596	< 0.01	0.028	0.70
Oberon, North Dakota	(5)	77	206	190			0.074	0.604	< 0.01	0.025	0.70
USA, 2010							0.074				0.70
(Faller)							(mean)				(mean)
RV067-10HA,	2	83	204	140	Grain	30	0.018	< 0.05	< 0.01	< 0.01	0.068
Taber, Alberta	(7)	87	197	130			0.019	< 0.05	< 0.01	< 0.01	0.069
Canada, 2010											
(Supurb)											
RV068-10HA,	2	69	208	190	Grain	21	0.05	< 0.05	< 0.01	< 0.01	0.10
Meadow, Texas	(6)	77	208	190			0.051	< 0.05	< 0.01	< 0.01	0.10
USA, 2011							0.05				0.10
(Hatcher)							(mean)				(mean)
RV069-10HA,	2	83	201	180	Grain	21	0.232	0.315	< 0.01	0.015	0.56
Hinton, Oklahoma	(7)	83	207	180			0.288	0.345	0.01	0.013	0.65
USA, 2010							0.26				0.60
(Jagger)							(mean)				(mean)
RV070-10HA, Levelland, Texas USA, 2010 (TAM 111)	2	71	205	190	Grain	21	0.041	< 0.05	< 0.01	< 0.01	0.091
	(8)	83	206	190			0.026	< 0.05	< 0.01	< 0.01	0.076
							0.033 (mean)				0.083 (mean)
	1	0	102		Grain	205	< 0.01	0.069	< 0.01	< 0.01	0.079
	(Seed)						< 0.01	0.069	< 0.01	< 0.01	0.079
RV071-10HA,	2 (7)	85	205	180	Grain	21	0.048	< 0.05	< 0.01	< 0.01	0.098
Wall, Texas		87	201	170			0.033	< 0.05	< 0.01	< 0.01	0.083
USA, 2010							0.040				0.090
(Coronado)							(mean)				(mean)
RV073-10DA,	2	85	201	180	Grain	10	0.105	< 0.05	< 0.01	< 0.01	0.16
Wall, Texas	(7)	85	201	180			0.102	< 0.05	< 0.01	< 0.01	0.15
USA, 2011						15	0.107	< 0.05	< 0.01	< 0.01	0.16
(Doans)							0.075	< 0.05	< 0.01	< 0.01	0.12

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
						21	0.069	< 0.05	< 0.01	< 0.01	0.12
							0.083	< 0.05	< 0.01	< 0.01	0.13
						28	0.066	< 0.05	< 0.01	< 0.01	0.12
							0.078	< 0.05	< 0.01	< 0.01	0.13
						34	0.344	1.03	< 0.01	0.095	1.5
							0.074	< 0.05	< 0.01	< 0.01	0.12
							0.21 (mean)				0.79 (mean)
RV072-10HA,	2	77	201	190	Grain	20	0.163	0.051	< 0.01	< 0.01	0.21
Uvalde, Texas	(7)	87	201	180			0.205	0.053	< 0.01	< 0.01	0.26
USA, 2011							0.18				0.24
(TAM 203)							(mean)				(mean)
RV074-10HA,	2	77	209	200	Grain	20	0.012	0.541	< 0.01	0.014	0.57
Parkdale, Oregon	(7)	77	205	190			0.021	0.547	< 0.01	0.014	0.58
USA, 2010							0.016				0.57
(Penawara)							(mean)				(mean)
RV075-10HA,	2	51	204	200	Grain	21	0.749	2.03	0.029	0.069	2.8
Alvena, Saskatchewan	(5)	59	212	210			0.708	1.88	0.026	0.066	2.7
Canada, 2010							0.73				2.7
(Infinity)							(mean)				(mean)
RV076-10HA,	2	83	205	160	Grain	21	0.083	0.075	0.079	0.081	0.24
Minto, Manitoba	(7)	85	200	160			0.256	0.894	< 0.01	0.084	1.2
Canada, 2010							0.17				0.74
(Infinity)							(mean)				(mean)
RV077-10HA,	2	75	208	160	Grain	21	0.028	0.264	< 0.01	0.018	0.31
Boissevain, Manitoba	(6)	83	206	160			0.020	0.264	< 0.01	0.014	0.30
Canada, 2010							0.024				0.30
(Glenn)							(mean)				(mean)
RV078-10HA,	2	71	199	190	Grain	21	0.032	0.179	< 0.01	< 0.01	0.21
Wellwood, Manitoba	(5)	73	201	200			0.030	0.175	< 0.01	< 0.01	0.21
Canada, 2010							0.031				0.21
(Glenn)							(mean)				(mean)
RV079-10HA,	2	57	206	200	Grain	21	0.361	2.07	0.026	0.037	2.5
Wakaw, Saskatchewan	(5)	64	211	210			0.375	2.27	0.029	0.038	2.7
Canada, 2010							0.37				2.6
(Harvest)							(mean)				(mean)
RV080-10HA,	2	65	206	200	Grain	20	0.251	1.00	0.019	0.024	1.3
Waldheim, Saskatchewan	(7)	73	210	200			0.196	0.958	0.015	0.021	1.2
Canada, 2010							0.22				1.2
(Infinity)							(mean)				(mean)
RV081-10HA,	2	75	201	99	Grain	21	0.102	0.695	< 0.01	0.029	0.83
Fort Saskatchewan, Alberta	(7)	85	209	100			0.099	0.649	0.011	0.022	0.77

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Canada, 2010							0.10				0.80
(Superb)							(mean)				(mean)
RV082-10DA,	2	83	213	100	Grain	10	0.40	0.536	0.017	0.027	0.96
Josephburg, Alberta	(7)	83	211	100			0.376	0.471	0.016	0.028	0.87
Canada, 2010						14	0.425	0.792	0.014	0.038	1.3
(Superb)							0.285	0.535	< 0.01	0.025	0.84
						21	0.082	0.675	< 0.01	0.025	0.78
							0.097	0.759	< 0.01	0.023	0.88
							0.090 (mean)				0.83 (mean)
						28	0.061	0.715	< 0.01	0.046	0.82
							0.056	0.72	< 0.01	0.042	0.82
						35	0.071	0.644	< 0.01	0.052	0.77
							0.067	0.686	< 0.01	0.05	0.80

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Nine supervised trials were carried out on sorghum (Table 124) in the USA during the 2010 growing season (Krolski and Dallstream 2012, RARVY004). Two foliar applications of a 200 g/L SL formulation were made. Three of the trials also included plots to measure the magnitude of BYI 02960 residues following the planting of seed treated with BYI 02960 480 FS (flowable concentrate). All applications were made using ground-based equipment. Adjuvant was added in all foliar applications at 0.25% v/v.

Residues in sorghum grain, forage and stover (fodder) were quantitated as flupyradifurone, difluoroacetic acid (DFA) and difluoroethylaminofuranone (DFEAF) using LC/MS/MS Method 01304. In addition, residues of 6-chloronicotinic acid (6-CNA) were measured. Acceptable concurrent recovery data (mean recoveries, RSDs) were obtained for all analytes.

Residues in sorghum forage and stover (fodder) are shown in Table 139.

Table 124 Residues from foliar or seed treatment application of flupyradifurone to sorghum in the USA (Krolski and Dallstream 2012, RARVY004)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 15	2 (7)		205			21					
RV083-10HA,	2	85	208	96	Grain	21	1.2	< 0.05	0.021	0.061	1.4
Proctor, Arkansas	(7)	85	206	95			1.5	< 0.05	0.022	0.085	1.6
USA, 2010							1.4				1.5
(Pioneer 85Y40)							(mean)				(mean)
RV084-10HA, Gardner, Kansas	2	85	207	149	Grain	21	0.97	< 0.05	0.019	0.029	1.1
USA, 2010	(5)	85	208	151			0.75	< 0.05	0.014	0.023	0.82
(B-7B47)							0.86 (mean)				0.94 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
	1	00	26		Grain	116	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(See d)						< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV085-10HA,	2	75	205	188	Grain	21	0.39	0.14	0.062	0.082	0.60
Dudley, Missouri	(7)	85	202	184			0.53	0.12	0.065	0.084	0.74
USA, 2010							0.46				0.67
(DKS54-00)							(mean)				(mean)
RV086-10DA,	2	87	204	184	Grain	10	1.7	< 0.05	0.022	0.024	1.7
Springfield,	(6)	87	203	174			1.3	< 0.05	0.015	0.023	1.4
Nebraska						13	1.3	< 0.05	0.018	0.026	1.4
USA, 2010							1.4	< 0.05	0.019	0.028	1.5
(NC+371)						19	0.78	< 0.05	0.015	0.030	0.86
							0.83	< 0.05	0.015	0.026	0.91
						26	1.3	< 0.05	0.014	0.040	1.3
							1.8	0.052	0.016	0.057	1.9
							1.5 (mean)				1.6 (mean)
						33	0.82	< 0.05	0.017	0.029	0.90
							0.81	0.053	0.020	0.036	0.90
RV087-10HA, Uvalde, Texas USA, 2010 (Asgrow A571)	2	85	204	186	Grain	21	0.56	0.14	0.051	0.066	0.76
	(7)	85	205	160			0.46	0.12	0.051	0.062	0.64
							0.51 (mean)			c0.012	0.70 (mean)
	1	00	14			119	< 0.01	< 0.05	< 0.01	0.017	0.077
	(See d)						< 0.01	< 0.05	< 0.01	0.013	0.073
RV088-10HA, Raymondville, Texas	2	85	211	96	Grain	20	0.87	0.061	0.044	0.043	0.97
	(7)	85	209	95			0.72	0.052	0.036	0.041	0.82
USA, 2010							0.79				0.89
(Dekalb: DKS 3707)							(mean)				(mean)
RV089-10HA,	2	85	206	188	Grain	21	0.32	0.053	0.039	0.023	0.40
Grand Island, Nebraska	(7)	85	206	191			0.35	0.055	0.034	0.024	0.43
USA, 2010							0.34				0.41
(7B47)							(mean)				(mean)
RV090-10HA, Levelland, Texas USA, 2010 (F-270E)	2	85	211	190	Grain	21	0.49	< 0.05	< 0.01	0.030	0.57
	(6)	85	204	184			0.50	< 0.05	< 0.01	0.040	0.59
							0.50 (mean)			c0.012	0.58 (mean)
	1	00	20		Grain	124	< 0.01	< 0.05	< 0.01	0.014	0.074
	(See d)						< 0.01	< 0.05	< 0.01	0.015	0.075
RV091-10HA, Wall, Texas USA, 2010 (Garst: 5515)	2	85	203	181	Grain	21	0.39	< 0.05	< 0.01	0.030	0.47
	(7)	87	203	179			0.52	< 0.05	< 0.01	0.033	0.61
							0.46				0.54
							(mean)				(mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Twenty supervised trials were carried out on field corn (Table 125) in the USA and Canada during the 2010 growing season (Fischer 2012, RARVY002). Two foliar applications of a 200 g/L SL formulation were made. Three of the trials also included plots to measure the magnitude of BYI 02960 residues following the planting of seed treated with BYI 02960 480 FS. All applications were made using ground equipment. Adjuvant was added in all foliar applications at 0.25% v/v.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in field corn kernels, forage and stover (fodder) were determined using LC-MS/MS method 01304. Acceptable concurrent recovery data were obtained for each analyte in each vegetable.

Residues in field corn forage and stover are shown in Table 137.

Table 125 Residues from foliar or seed treatment application of flupyradifurone to field corn in the USA and Canada (Fischer 2012, RARVY002)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Crop Group 15	2 (7)		205			21					
RV021-10HA, Alton, New York, USA, 2010 (Hyland Seeds HL 2093)	2	79	205	280	Kernel	20	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(6)	83	204	280			< 0.01	0.195	< 0.01	< 0.01	0.21
							< 0.01				0.13
							(mean)				(mean)
RV022-10HA, Blackville, South Carolina, USA, 2010 (DKC69-72)	2	89	203	130	Kernel	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(6)	89	205	130			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01				< 0.060
							(mean)				(mean)
RV023-10HA, Richland, Iowa, USA, 2010 (09HYBK110HOER)	2	87	207	160	Kernel	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(7)	87	205	170			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01				< 0.060
							(mean)				(mean)
	1 (Seed)	00	119		Kernel	133	< 0.01	0.118	< 0.01	< 0.01	0.13
							< 0.01	0.105	< 0.01	< 0.01	0.12
RV024-10HA, Gardner, Kansas, USA, 2010 (09HYBK110HOER)	2	85	204	150	Kernel	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(5)	85	201	150			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01				< 0.060
							(mean)				(mean)
	1 (Seed)	00	54			119	< 0.01	0.056	< 0.01	< 0.01	0.066
							< 0.01	0.089	< 0.01	< 0.01	0.099
RV033-10HA, Gardner, Kansas, USA, 2010 (83R38-3000GT)	2	85	206	150	Kernel	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(5)	85	207	150			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01				< 0.060
							(mean)				(mean)
RV025-10HA,	2	85	206	190	Kernel	20	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Northwood, North Dakota,	(6)	85	206	190			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(Decalb DKC35-19)							(mean)				(mean)
RV026-10HA, Brant, Ontario,	2	85	207	310	Kernel	22	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Canada, 2010	(7)	85	209	310			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Decalb 3832							< 0.01				< 0.060
Non-BT)							(mean)				(mean)
RV029-10HA, Brant, Ontario,	2	87	205	310	Kernel	22	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Canada, 2010	(7)	87	206	300			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Dekalb 4660)							< 0.01				< 0.060
							(mean)				(mean)
RV027-10HA, Stafford, Kansas,	2	85	205	190	Kernel	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010	(7)	87	199	180			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(A:09HYBK105							< 0.01				< 0.060
HOER)							(mean)				(mean)
	1 (Seed)	00	118		Kernel	131	< 0.01	0.174	< 0.01	< 0.01	0.18
							< 0.01	0.168	< 0.01	< 0.01	0.18
RV035-10HA, Stafford, Kansas,	2	85	207	190	Kernel	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010	(7)	87	209	190			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Pioneer 32B34)							< 0.01				< 0.060
							(mean)				(mean)
RV028-10HA, Clarence,	2	85	202	180	Kernel	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Missouri,	(7)	87	209	190			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(MFA Trophy)							(mean)				(mean)
RV030-10HA, York, Nebraska,	2	87	204	190	Kernel	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010	(7)	87	204	180			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Channel 207-							< 0.01				< 0.060
03VT)							(mean)				(mean)
RV031-10HA, Perry, Iowa,	2	79	201	190	Kernel	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010	(7)	87	205	190			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(P1162XR)							< 0.01				< 0.060
							(mean)				(mean)
RV032-10HA, Rockwood,	2	85	206	130	Kernel	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Ontario,	(7)	87	207	140			0.011	< 0.05	< 0.01	< 0.01	0.061
Canada, 2010							0.011				0.061
(25T87)							(mean)				(mean)
RV034-10HA, Breslau, Ontario,	2	87	202	200	Kernel	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Canada, 2010	(7)	87	204	200			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(20T16)							< 0.01				< 0.060
							(mean)				(mean)
RV036-10DA, Atlantic, Iowa,	2	85	211	300	Kernel	10	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
USA, 2010 (Cgarst 85R08-3000GT)	(7)	85	210	300			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						22	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01				< 0.060
							(mean)				(mean)
						28	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						34	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV037-10DA, Branchton, Ontario, Canada, 2010 (Maizex)	2	85	203	280	Kernel	10	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(7)	85	199	300			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						19	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01				< 0.060
							(mean)				(mean)
						27	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						33	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV038-10DA, Springfield, Nebraska, USA, 2010 (N38B4)	2	87	204	180	Kernel	10	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(6)	87	202	170			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						13	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							0.01	< 0.05	< 0.01	< 0.01	0.060
						19	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01				< 0.060
							(mean)				(mean)
						26	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						33	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV039-10DA, Campbell, Minnesota, USA, 2010 (Dekalb 38-89)	2	85	205	190	Kernel	10	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(7)	85	205	190			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						15	0.012	< 0.05	< 0.01	< 0.01	0.062
							0.01	< 0.05	0.011	< 0.01	0.060
						21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01				< 0.060
							(mean)				(mean)
						28	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						35	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV040-10HA, Raymondville, Texas USA, 2010 (H6284162)	2	87	211	96	Kernel	20	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(7)	87	212	96			< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
								c0.203			

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Tree nuts

Supervised trials were carried out on almonds (five trials—Table 126) and pecans (five trials—Table 127) in the USA during the 2010 growing season (Niczyporowicz and Netzband 2012, RARVY016). Two foliar applications of a 200 g/L SL formulation were made in either concentrated or dilute spray applications. Applications were made to plots using ground equipment. Adjuvant was added in all applications at 0.25% v/v.

The almonds and pecans sampled were shelled to produce the commodity of nutmeat. Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) were determined in almonds and pecan nutmeat and almond hulls, using LC/MS/MS Method 01304. Acceptable concurrent recovery data were obtained for all analytes.

Residues in almond hulls are shown in Table 142.

Table 126 Residues from the foliar application of flupyradifurone to almonds in the USA (Niczyporowicz and Netzband 2012, RARVY016)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RV204-10DA,	2	78	204	423	Nutmeat w/o	0	< 0.01	< 0.05	< 0.01	0.014	0.074
Madera,	(14)	79	205	425	shell		0.01	< 0.05	< 0.01	< 0.01	0.060
California,						3	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Non-Pareil)						7	< 0.01	0.073	< 0.01	< 0.01	0.083
							< 0.01	0.055	< 0.01	< 0.01	0.065
							< 0.01 (mean)				0.074 (mean)
						14	< 0.01	0.104	< 0.01	< 0.01	0.11
							< 0.01	0.092	< 0.01	< 0.01	0.10
							< 0.01 (mean)				0.11 (mean)
						21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	2	78	210	2131	Nutmeat w/o	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(14)	79	211	2147	shell		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01 (mean)				< 0.060 (mean)
RV205-10DA,	2 (14)	78	204	94	Nutmeat w/o	0	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Orland,		89	206	94	shell		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
California,						3	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Non-Pareil)						7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01 (mean)				< 0.060 (mean)
						14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	2 (14)	78	204	1869	Nutmeat w/o	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
		89	204	1871	shell		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							< 0.01 (mean)				< 0.060 (mean)
RV206-10HA,	2 (14)	85	203	379	Nutmeat w/o	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Hickman,		89	205	382	shell		0.015	< 0.05	< 0.01	< 0.01	0.065
California,							0.013 (mean)				0.063 (mean)
USA, 2010	2 (14)	85	201	2217	Nutmeat w/o	7	0.015	< 0.05	< 0.01	< 0.01	0.065
(Sonora)		89	205	2185	shell		0.014	< 0.05	< 0.01	< 0.01	0.064
							0.015 (mean)				0.065 (mean)
RV207-10HA,	2 (14)	85	205	358	Nutmeat w/o	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Lost Hills,		85	205	353	shell		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
California,							< 0.01 (mean)				< 0.060 (mean)
USA, 2010	2 (14)	85	206	2296	Nutmeat w/o	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Monterey)		85	206	2216	shell		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01 (mean)				< 0.060 (mean)
RV208-10HA,	2 (14)	79	201	417	Nutmeat w/o	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Kerman,		85	202	420	shell		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
California,							< 0.01 (mean)				< 0.060 (mean)
USA, 2010	2 (14)	79	206	2364	Nutmeat w/o	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Padre)		85	209	2391	shell		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01 (mean)				< 0.060 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 127 Residues from the foliar application of flupyradifurone to pecans in the USA (Niczyporowicz and Netzband 2012, RARVY016)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Tree Nuts	2 (14)		205			7					
RV209-10DA,	2	79	205	202	Nutmeat	0	0.011	< 0.05	< 0.01	< 0.01	0.061
Chula,	(14)	89	205	197	w/o		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Georgia,					shell	3	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Sumner)						7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01 (mean)				< 0.060 (mean)
						14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	2	79	208	2323	Nutmeat	7	0.013	< 0.05	< 0.01	< 0.01	0.063

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
	(14)	89	205	2311	w/o		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
					shell		0.012 (mean)				0.062 (mean)
RV210-10DA,	2	79	205	202	Nutmeat	0	0.048	< 0.05	< 0.01	< 0.01	0.095
Ocilla,	(14)	89	205	197	w/o		0.015	< 0.05	< 0.01	< 0.01	0.065
Georgia,					shell	3	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Sumner)						7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01 (mean)				< 0.060 (mean)
						14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	2	79	208	2324	Nutmeat	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(14)	89	205	2308	w/o		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
					shell		< 0.01 (mean)				< 0.060 (mean)
RV211-10HA,	2	95	210	248	Nutmeat	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Alexandria,	(14)	97	207	301	w/o		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Louisiana, USA, 2010					shell		< 0.01 (mean)				< 0.060 (mean)
(Creek)	2	95	209	2048	Nutmeat	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(14)	97	210	1791	w/o		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
					shell		< 0.01 (mean)				< 0.060 (mean)
RV212-10HA,	2	87	208	381	Nutmeat	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
D'Hanis,	(13)	87	204	419	w/o		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Texas, USA, 2010					shell		< 0.01 (mean)				< 0.060 (mean)
(Cheyenne)	2	87	201	1851	Nutmeat	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
	(13)	87	205	2041	w/o		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
					shell		< 0.01 (mean)				< 0.060 (mean)
RV213-10HA,	2	85	211	478	Nutmeat	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Pumpkin Center,	(15)	89	209	489	w/o shell		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Oklahoma,							< 0.01 (mean)				< 0.060 (mean)
USA, 2010	2	85	205	2133	Nutmeat	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Kiowa and	(15)	89	205	2225	w/o		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Washita)					shell		< 0.01 (mean)				< 0.060 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Oilseeds

Supervised trials were carried out on <u>cotton</u> (12 trials—Table 128) in the USA during the 2010 growing season (Timberlake and Harbin 2012, RARVY009). Two foliar applications of a 200 g/L SL formulation were made using ground equipment. Adjuvant was added in all spray applications at 0.25% v/v. Three of these field trials also included plots to measure residues in the same matrices following the planting of seed treated with BYI 02960 480 FS.

All seed cotton samples were ginned to generate cottonseed samples (undelinted seed) for analysis. Additionally, six trials included the collection of cotton gin by-products (gin trash) from cotton harvested mechanically using either a picker or stripper (three trials each).

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) were determined in cotton undelinted seed and cotton gin by-products using LC-MS/MS method 01304. In addition, residues of were measured. Acceptable concurrent recovery data were obtained for all analytes in both seed and gin by-products.

Residues in cotton gin by-products are shown in Table 143.

Table 128 Residues in cotton seed from the foliar application of flupyradifurone to cotton in the USA (Timberlake and Harbin 2012, RARVY009)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Cotton	2 (10)		205			14					
RV108-10HA,	2	83	210	283	Seed,	14	< 0.01	< 0.05	< 0.01	0.10	0.16
Fresno, California,	(9)	85	215	289	undelinted		0.018	< 0.05	< 0.01	0.024	0.092
USA, 2010							0.014				0.13
(PHY755 WRF Acala)							(mean)				(mean)
RV109-10HA,	2	83	207	285	Seed,	14	0.10	< 0.05	< 0.01	0.035	0.19
Paso Robles, California,	(8)	85	205	272	undelinted		0.16	< 0.05	< 0.01	0.023	0.24
USA, 2010							0.13				0.21
(DP353)							(mean)				(mean)
RV110-10HA,	2	85	205	195	Seed,	14	0.049	< 0.05	< 0.01	0.069	0.17
Cheneyville, Louisiana,	(8)	87	209	161	undelinted		0.112	< 0.05	< 0.01	0.12	0.29
USA, 2010							0.081 (mean)				0.23 (mean)
(Phytogen 485 WRF)							c0.011				
RV111-10HA,	2	87	205	186	Seed,	19	0.027	< 0.05	< 0.01	0.11	0.19
Suffolk, Virginia,	(9)	88	206	188	undelinted		0.039	< 0.05	< 0.01	0.014	0.10
USA, 2010											
(PHY375WRF)											
RV112-10HA,	2	87	206	143	Seed,	13	0.16	< 0.05	< 0.01	0.027	0.24
Wall, Texas,	(8)	88	207	142	undelinted		0.63	< 0.05	< 0.01	0.021	0.70
USA, 2010							0.40				0.47
(FM 1740 B2F)							(mean)				(mean)
RV113-10HA,	2	82	203	308	Seed,	14	0.016	< 0.05	< 0.01	0.01	0.076
Sanger,	(9)	87	204	305	undelinted		0.019	< 0.05	< 0.01	< 0.01	0.069
California,							0.018 (mean)				0.072 (mean)
USA, 2010	1 (Seed)	0	47		Seed,	179	< 0.01	< 0.05	< 0.01	0.020	0.080
(Acala Daytona RF)					undelinted		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
RV114-10HA,	2	84	204	172	Seed,	13	0.053	0.093	< 0.01	0.22	0.37
Uvalde, Texas,	(7)	87	206	183	undelinted		0.024	< 0.05	< 0.01	0.092	0.17
							0.039 (mean)				0.27 (mean)
USA, 2010	1 (Seed)	0	61		Seed,	136	< 0.01	0.057	< 0.01	0.045	0.11

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
(FM1740B2F)					undelinted		< 0.01	0.076	< 0.01	0.033	0.12
RV118-10DA,	2	88	202	170	Seed,	0	0.57	< 0.05	< 0.01	0.032	0.65
Uvalde,	(9)	89	202	184	undelinted	6	0.81	< 0.05	< 0.01	0.038	0.90
Texas,						14	0.26	< 0.05	< 0.01	0.042	0.35
USA, 2010							0.41	< 0.05	< 0.01	0.055	0.51
(Stoneville 5458)							0.33 (mean)				
						19	0.49	< 0.05	< 0.01	0.090	0.63
						28	0.34	< 0.05	< 0.01	0.11	0.49
RV115-10HA,	2	84	207	187	Seed,	14	0.081	< 0.05	< 0.01	< 0.01	0.13
Levelland,	(7)	87	203	183	undelinted		0.067	< 0.05	< 0.01	< 0.01	0.12
Texas,							0.074 (mean)				0.12 (mean)
USA, 2010	1 (Seed)	0	60		Seed,	158	< 0.01	0.39	< 0.01	< 0.01	0.40
(FM 9180 B2 F)					undelinted		< 0.01	0.29	< 0.01	< 0.01	0.30
RV116-10DA,	2	88	206	112	Seed,	0	0.44	< 0.05	< 0.01	0.030	0.52
Greenville,	(8)	89	206	111	Undelinted	6	0.23	< 0.05	< 0.01	0.032	0.31
Mississippi,						14	0.17	< 0.05	< 0.01	0.039	0.26
USA, 2010							0.19	< 0.05	< 0.01	0.038	0.28
(ST 5458BIIRF)							0.18 (mean)				0.27 (mean)
						21	0.058	< 0.05	< 0.01	0.046	0.15
						27	0.080	< 0.05	< 0.01	0.040	0.17
RV117-10DA,	2	88	205	95	Seed,	0	0.42	< 0.05	< 0.01	0.090	0.56
Proctor,	(10)	89	205	95	undelinted	7	0.064	< 0.05	< 0.01	0.057	0.17
Arkansas,						13	0.060	< 0.05	< 0.01	0.058	0.17
USA, 2010							0.082	< 0.05	< 0.01	0.046	0.18
(DynaGro2400RF)							c 0.024				
						21	0.12	< 0.05	< 0.01	0.066	0.24
						28	0.020	0.060	< 0.01	0.084	0.16
RV119-10DA,	2	89	204	283	Seed,	0	0.36	< 0.05	< 0.01	0.42	0.83
Hinton,	(9)	89–99	207	286	undelinted	7	0.22	< 0.05	< 0.01	0.51	0.78
Oklahoma,						13	0.17	< 0.05	< 0.01	0.41	0.63
USA, 2010							0.24	< 0.05	< 0.01	0.57	0.86
(FM9063B2F)							0.20 (mean)				0.74 (mean)
						28	0.18	< 0.05	< 0.01	0.56	0.79

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Supervised trials were carried out on peanuts (12 trials—Table 129) in the USA during the 2010 growing season (Krolski and Harbin 2012a, RARVY010). Two foliar applications of a 200 g/L SL formulation were made with ground-based equipment. Adjuvant was added in all applications at 0.25% v/v.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in peanuts nut without shell and peanut hay were determined using LC/MS/MS Method 01304. Acceptable concurrent recovery data (mean recoveries, RSDs) were obtained for all analytes.

Residues in peanut hay are shown in Table 144.

Table 129 Residues in peanut nutmeat from the foliar application of flupyradifurone to peanuts in the USA (Krolski and Harbin 2012a, RARVY010)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Peanuts	2 (10)		205			7					
RV120-10HA,	2	83	205	130	Nut m	6	0.034	< 0.05	< 0.01	< 0.01	0.084
Elko, South Carolina,	(11)	86	206	140	eat		0.020	< 0.05	< 0.01	0.019	0.090
USA, 2010							0.027				0.087
(Gregory)							(mean)				(mean)
RV121-10HA,	2	79	206	270	Nut m	7	0.018	< 0.05	< 0.01	0.011	0.079
Athens, Georgia,	(10)	79	204	280	eat		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							0.014				0.069
(Georgia-06G)							(mean)				(mean)
RV122-10HA,	2	87	210	120	Nut m	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Suffolk, Virginia,	(10)	88	211	120	eat		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(Champs)							(mean)				(mean)
RV123-10HA,	2	89	204	230	Nut m	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Tallassee, Alabama,	(10)	89	206	190	eat		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(Georgia Greener)							(mean)				(mean)
RV124-10HA,	2	85	201	190	Nut m	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Seven Springs, North Carolina,	(10)	87	205	200	eat		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(Perry)							(mean)				(mean)
RV128-10DA,	2	88	208	200	Nut	0	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Seven Springs,	(10)	89	205	200	m eat		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
North						3	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Carolina,							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010						7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Champs)							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01				
							(mean)				
						14	< 0.01	0.066	< 0.01	< 0.01	0.076
							< 0.01	0.077	< 0.01	< 0.01	0.087
											0.082 (mean)
						21	< 0.01	0.054	< 0.01	< 0.01	0.064
							< 0.01	0.052	< 0.01	< 0.01	0.062
RV125-10HA,	2	88	205	220	Nut m	8	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Cuthbert, Georgia,	(10)	88	205	230	eat		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							< 0.01				< 0.060
(Georgia Green)							(mean)				(mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RV126-10HA,	2	87	208	190	Nut m	3	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Greenville, Florida,	(7)	89	205	190	eat		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010											
(GA-06)											
RV127-10HA,	2	84	198	180	Nut m	6	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Hinton, Oklahoma,	(10)	85	199	200	eat		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010							c0.074				
(Tamnut 0L06)											
RV129-10DA,	2	88	206	230	Nut m	0	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Tifton, Georgia,	(10)	88	205	230	eat		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010						3	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Georgia 06G)							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						8	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							0.011	< 0.05	< 0.01	< 0.01	0.061
							0.011				
							(mean)				
						21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	0.060	< 0.01	< 0.01	0.070
											0.065 (mean)
RV130-10DA,	2	84	205	180	Nut m	0	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Madill, Oklahoma,	(10)	89	206	180	eat		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010						3	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Tamrun)							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							c 0.071				
						14	0.019	< 0.05	< 0.01	< 0.01	0.069
							0.011	< 0.05	< 0.01	< 0.01	0.061
						21	0.023	< 0.05	< 0.01	< 0.01	0.073
							0.011	< 0.05	< 0.01	< 0.01	0.061
							0.017 (mean)				0.067 (mean)
RV131-10DA,	2	85	207	180	Nut m	0	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
Wellington, Texas,	(10)	89	208	180	eat		< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
USA, 2010						3	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
(Florida 07)							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01				< 0.060
							(mean)				(mean)
						14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
						21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.060

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Seeds for beverages and sweets

Supervised trials were carried out on coffee (four trials—Table 130) in Guatemala and Mexico during the 2011 growing season to determine residues after a single soil drench application followed by three broadcast spray applications of flupyradifurone (Hoag 2012b, RARVP074). Other trials were conducted in Brazil in 2011 (Resende 2012, M-427469-03-2; I11-008 Trials I11-008-01, 02, 04 and 05) and in 2012 (Anon. 2013, M-481362-01-1; summary data, trials I12-006-01 to I12-006-05). Foliar applications were made with ground equipment. Adjuvant was added in all applications at 0.25% v/v.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in coffee beans were determined using LC/MS/MS Method 01304. Acceptable concurrent recovery data were obtained for all analytes.

Table 130 Residues in green coffee beans from the foliar application of flupyradifurone to coffee in Guatemala, Mexico and Brazil (various studies)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RV232-11DA,	1 + 3	78	600	227	Bean,	0	0.085	0.13	< 0.01	< 0.01	0.22
Cuilapa, Santa Rosa,	(91, 13,	79	199	394	green		0.079	0.23	< 0.01	< 0.01	0.31
Guatemala, 2011	12)	80	210	412		7	0.098	0.14	0.013	< 0.01	0.24
(Catuai)		88	201	367			0.11	0.094	0.015	< 0.01	0.20
						14	0.11	0.053	0.015	< 0.01	0.17
							0.13	0.063	0.015	< 0.01	0.19
						21	0.12	0.10	0.014	< 0.01	0.22
							0.11	0.097	0.018	< 0.01	0.21
						28	0.14	0.12	0.022	< 0.01	0.26
							0.13	0.089	0.020	< 0.01	0.22
RV233-11DA,	1 + 3	78	600	230	Bean,	0	0.047	0.10	< 0.01	< 0.01	0.15
Barberena, Santa	(90, 14,	81	199	401	green		0.055	0.12	< 0.01	< 0.01	0.18
Rosa,	14)	81	199	406		7	0.045	0.11	< 0.01	< 0.01	0.15
Guatemala, 2011		88	199	370			0.040	0.097	< 0.01	< 0.01	0.14
(Caturra)						14	0.061	0.12	< 0.01	< 0.01	0.18
							0.046	0.080	< 0.01	< 0.01	0.13
						21	0.063	0.14	< 0.01	< 0.01	0.20
							0.067	0.13	< 0.01	< 0.01	0.19
						28	0.052	0.12	< 0.01	< 0.01	0.17
							0.05	0.10	< 0.01	< 0.01	0.15
RV234-11DA,	1 + 3	73	605	126	Bean,	0	0.21	0.35	0.012	0.011	0.57
Zentla, Veracruz	(86, 14,	77	199	394	green		0.19	0.67	0.017	< 0.01	0.85
Mexico, 2011	14)	79	197	395		7	0.16	0.65	0.023	0.013	0.82
(Costa Rica)		81	199	402			0.16	0.75	0.019	0.014	0.92
						14	0.10	0.22	0.015	< 0.01	0.33
							0.13	0.40	< 0.01	0.011	0.54
						21	0.14	0.50	< 0.01	< 0.01	0.65
							0.14	0.33	0.019	< 0.01	0.47
						28	0.12	0.33	0.015	< 0.01	0.45
							0.11	0.51	0.019	< 0.01	0.63
RV246-11DA,	1 + 3	72	609	195	Bean,	0	0.12	0.12	0.014	< 0.01	0.24
La Union,	(89, 12,	81	197	397	green		0.12	0.11	0.014	< 0.01	0.23
Zihuateutla,	13)	81	195	393		7	0.25	0.13	0.028	< 0.01	0.37
Puebla,		85	203	414			0.24	0.13	0.030	< 0.01	0.38
Mexico, 2011						13	0.44	0.11	0.055	< 0.01	0.55
(Caturra)							0.36	0.10	0.043	< 0.01	0.46
						20	0.46	0.12	0.064	0.01	0.59

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
							0.44	0.12	0.060	0.012	0.58
						26	0.59	0.31	0.090	0.020	0.91
							0.52	0.28	0.095	0.020	0.82
I11-008-01,	1 + 3	81	600		Bean,	0	0.03	< 0.05	< 0.01	< 0.01	0.08
Ribeirao Preto, São		85	202	400	green		0.04	< 0.05	< 0.01	< 0.01	0.09
Paulo,		88	208	400		7	0.03	< 0.05	< 0.01	< 0.01	0.08
Brazil, 2011		89	186	400			< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
(Catuai)						14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
						21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
						28	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
I11-008-02,	1 + 3	75	596		Bean,	0	0.04	< 0.05	< 0.01	< 0.01	0.09
Paulinia, São Paulo,		88	212	400	green		0.04	< 0.05	< 0.01	< 0.01	0.09
Brazil, 2011		88	200	400		7	0.04	< 0.05	< 0.01	< 0.01	0.09
(Catuai-Vermelho)		89	192	400			0.03	< 0.05	< 0.01	< 0.01	0.08
						14	0.03	< 0.05	< 0.01	< 0.01	0.08
							0.04	< 0.05	< 0.01	< 0.01	0.09
						21	0.02	< 0.05	< 0.01	< 0.01	0.07
							0.02	< 0.05	< 0.01	0.01	0.08
						28	0.07	0.09	0.01	0.01	0.17
							0.08	0.10	0.01	0.02	0.20
I11-008-04,	1 + 3	73	598		Bean,	0	0.02	< 0.05	< 0.01	< 0.01	0.07
Londrina, Paraná,		85	206	400	green		0.02	< 0.05	< 0.01	< 0.01	0.07
Brazil, 2011		88	170	400		7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
(Catuai)		89	214	400			< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
						14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
							0.01	< 0.05	< 0.01	< 0.01	0.06
						21	0.05	< 0.05	< 0.01	< 0.01	0.10
							0.05	< 0.05	< 0.01	< 0.01	0.10
						28	0.03	< 0.05	< 0.01	< 0.01	0.08
							0.03	< 0.05	< 0.01	< 0.01	0.08
I11-008-05,	1 + 3	85	606		Bean,	0	0.02	< 0.05	< 0.01	< 0.01	0.07
Cristais Paulista,		87	212	400	green		0.02	< 0.05	< 0.01	< 0.01	0.07
São Paulo,		88	200	400		7	0.02	< 0.05	< 0.01	< 0.01	0.07
Brazil, 2011		89	202	400			0.01	< 0.05	< 0.01	< 0.01	0.06
(Mundo Novo)						14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
						21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
						28	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
							< 0.01	< 0.05	< 0.01	< 0.01	< 0.06
I12-006-01,	1 + 3	75	600	200	Bean,	0	0.20	< 0.05	< 0.01	< 0.01	0.25
Paulinia		81	200	400	green	7	0.23	< 0.05	0.01	< 0.01	0.28
Brazil, 2012		83	200	400		14	0.20	0.10	< 0.01	< 0.01	0.30
(Catuai Vermelho)		85	198	400		22	0.17	0.10	< 0.01	< 0.01	0.27
						28	0.20	0.09	< 0.01	0.01	0.30
						35	0.19	0.07	< 0.01	< 0.01	0.26
I12-006-02,	1 + 3	73	601	200	Bean,	0	0.08	< 0.05	0.01	< 0.01	0.13
Campinas		85	208	400	green	8	0.08	< 0.05	0.01	< 0.01	0.13
Brazil, 2012		85	195	400		14	0.08	< 0.05	< 0.01	< 0.01	0.13
(Catuai Vermelho)		85	196	400		20	0.08	< 0.05	< 0.01	< 0.01	0.13
						28	0.07	< 0.05	< 0.01	< 0.01	0.12
						35	0.22	0.08	0.02	< 0.01	0.30
I12-006-03,	1 + 3	73	602	200	Bean,	0	0.04	0.10	< 0.01	< 0.01	0.14
Londrina		79	191	400	green	7	0.02	0.10	< 0.01	< 0.01	0.12

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
Brazil, 2012		80	201	400		14	0.03	0.12	< 0.01	< 0.01	0.15
(Catuai)		81	200	400		19	0.02	< 0.05	< 0.01	< 0.01	0.07
						28	0.02	0.07	< 0.01	< 0.01	0.09
						33	0.02	0.08	< 0.01	< 0.01	0.10
I12-006-04,	1 + 3	81	639	200	Bean,	0	0.01	< 0.05	< 0.01	< 0.01	0.06
Ribirao Preto		81	206	400	green	7	0.05	< 0.05	< 0.01	0.01	0.11
Brazil, 2012		81	200	400		14	0.08	< 0.05	< 0.01	< 0.01	0.13
(Catuai)		81	195	400		21	0.21	0.14	0.01	0.02	0.37
						28	0.27	0.12	0.01	0.01	0.40
						35	0.60	0.15	0.01	0.02	0.77
I12-006-05,	1 + 3	75	617	200	Bean,	0	0.04	< 0.05	< 0.01	< 0.01	0.09
Cristais Paulista		81	196	400	green	7	0.24	0.06	< 0.01	0.02	0.32
Brazil, 2012		81	204	400		14	0.25	0.06	0.01	0.01	0.32
(Mundo Novo)		85	192	400		21	0.35	0.12	0.02	0.02	0.49
						28	0.18	0.09	< 0.01	< 0.01	0.27
						35	0.26	0.10	< 0.01	< 0.01	0.36

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Hops

Supervised trials were carried out on hops (three trials—Table 131) in the USA during the 2011 growing season to determine residues after a single foliar application (Krolski 2012, RARVY008). Applications were made to plots using ground equipment. Adjuvant was added in all applications at 0.2% v/v (RV047-11HA, non-ionic surfactant, 1.0% v/v (RV048-11HA, crop-oil concentrate) or 0.25% (RV048-11HA, methylated seed oil). Fresh hops were kiln-dried on the day of harvest to generate the RAC of dried hop cones.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in dried hop cones were determined using LC/MS/MS Method 01304. Acceptable concurrent recovery data were obtained for all analytes.

Table 131 Residues in kiln-dried hop cones from the foliar application of flupyradifurone to hops in the USA (Krolski 2012, RARVY008)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP, USA, Hops	1		153			21					
RV047-11HA,	1	85	156	422	Cone,	21	2.41	0.90	0.011	0.092	3.4
Wilder, Idaho,					kiln-dried					c 0.064	
USA, 2011	1	85	155	1178	Cone,	21	2.18	0.96	0.006	0.089	3.2
(Apollo)					kiln-dried					c 0.064	
RV048-11HA,	1	85	155	421	Cone,	21	4.63	3.32	0.037	0.19	8.1
Ephrata, Washington,					kiln-dried					c 0.017	
USA, 2011	1	85	154	974	Cone,	21	4.72	2.97	0.07	0.24	7.9
(Cascade)					kiln-dried					c 0.017	
RV049-11HA,	1	85	154	315	Cone,	21	2.26	0.80	0.004	0.051	3.1

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
St. Paul, Oregon,					kiln-dried					c 0.016	
USA, 2011	1	85	152	595	Cone,	21	2.7	0.64	0.008	0.047	3.4
(Nugget)					kiln-dried					c 0.016	

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Animal feeds

Table 132 Residues in pea vines and hay from foliar applications of flupyradifurone to peas in the USA and Canada (Hoag, Arthur and Woodard 2012, RARVY028)

Trial No., Location, Year (Variety)	Application				Sample	DALA (% DM)	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	Parent + DFA	6-CNA
GAP, USA, Crop Group 6	2 (10)		205			7					
RV180-11HA, Rockwood, Ontario Canada, 2011 (Meadow)	2	61	206	144	Green	7	4.4	0.72	0.046	5.1	0.11
	(8)	67	209	146	material	(22.6)	4.8	0.69	0.058	5.5	0.11
										5.3 (mean)	0.11 (mean)
					Hay	7	9.8	2.1	0.084	12	0.43
						(40.5)	9.9	2.2	0.14	12	0.41
							9.9 (mean)			12 (mean)	0.42 (mean)
RV181-11HA, Rupert, Idaho USA, 2011 (Progress 9)	2	16	205	163	Green	7	1.4	0.65	0.031	2.1	0.05
	(10)	62	200	167	material	(15.9)	1.6	0.69	0.043	2.3	0.054
										2.2 (mean)	0.052 (mean)
					Hay	7	6.8	3.1	0.16	9.9	0.37
						(87.0)	6.8	3.0	0.11	9.7	0.34
							6.8 (mean) c0.025			9.8 (mean)	0.35 (mean)
RV182-11HA, Jerome, Idaho USA, 2011 (FMK 888-0132*N14)	2	37	202	184	Green	5	4.9	0.28	0.051	5.2	0.11
	(10)	59	205	179	material	(12.4)	5.1	0.30	0.052	5.4	0.11
										5.3 (mean)	0.11 (mean)
					Hay	5	18	2.4	0.33	21	0.79
						(57.1)	12	4.4	0.15	16	1.0
							15 (mean)			19 (mean)	0.91 (mean)
RV183-11HA, Payette, Idaho USA, 2011 (Austrian Winter Pea)	2	61	203	231	Green	7	3.4	0.90	0.031	4.3	0.15
	(9)	65	203	231	material	(17.6)	3.4	1.0	0.041	4.4	0.21
										4.4 (mean)	0.18 (mean)
					Hay	7	7.3	4.1	0.11	11	1.2
						(76.1)	9.2	2.4	0.28	12	0.74
							8.2 (mean) c0.011			11 (mean)	0.96 (mean)
RV184-11HA,	2	62	206	111	Green	6	4.3	0.73	0.048	5.0	0.13

Trial No., Location, Year (Variety)	Application				Sample	DALA (% DM)	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	Parent + DFA	6-CNA
Waldheim, Saskatchewan Canada, 2011 (Admiral)	(10)	69	208	111	material	(18.6)	4.4	0.55	0.036	4.9	0.10
										5.0 (mean)	0.12 (mean)
					Hay	6	5.7	2.3	0.025	8.0	1.3
						(39.7)	7.1	2.5	0.028	9.6	1.3
							6.4 (mean) c0.32			8.8 (mean)	1.3 (mean)
RV185-11HA, Blaine Lake, Saskatchewan Canada, 2011 (Admiral)	2	64	204	109	Green	6	3.6	0.35	0.041	3.9	0.053
	(10)	69	207	112	material	(19.3)	3.4	0.38	0.033	3.8	0.055
										3.8 (mean)	0.054 (mean)
					Hay	6	4.6	1.0	0.027	5.6	0.30
						(48.2)	4.8	1.3	0.022	6.1	0.36
							4.7 (mean) c0.12			5.9 (mean)	0.33 (mean)
RV186-11DA, Ephrata, Washington	2	61	206	187	Green	0	8.1	1.1	0.032	9.2	0.11
	(10)	69	206	187	material	(17.8)	7.3	1.2	0.041	8.5	0.13
						7	2.5	1.6	0.041	4.1	0.22
						(24.0)	2.5	1.5	0.029	4.1	0.19
USA, 2011 (Austrian Winter Pea)							c0.016	c0.28		4.1 (mean)	
						14	1.8	2.4	0.024	4.2	0.35
						(29.3)	2.2	1.8	0.019	3.9	0.36
						21	1.0	4.1	0.015	5.1	0.64
						(48.3)	1.5	3.9	0.016	5.3	0.75
						28	2.8	5.1	0.039	7.9	1.4
						(83.5)	4.2	4.0	0.053	8.2	1.2
						35	2.4	4.9	0.05	7.3	1.4
						(82.2)	3.1	4.2	0.055	7.2	1.7
											1.6 (mean)
					Hay	0	19	3.2	0.22	22	0.65
						(68.5)	21	3.8	0.23	25	0.77
						7	7.6	4.4	0.11	12	0.86
						(90.3)	8.5	4.7	0.097	13	0.99
							8.0 (mean) c0.028	c 0.36		13 (mean)	
						14	5.1	4.4	0.067	9.5	1.0
						(86.5)	4.6	4.9	0.056	9.5	1.1
						21	6.0	4.3	0.082	10	1.7
						(90.6)	4.3	4.7	0.055	9.0	1.4
											1.6 (mean)
						28	4.8	4.3	0.075	9.1	0.99
						(87.2)	5.4	3.3	0.075	8.7	1.1
						35	3.6	4.2	0.068	7.8	1.6
						(91.0)	3.2	4.0	0.054	7.2	1.3
RV187-11DA, Parkdale, Oregon USA, 2011 (Progress 9)	2	33	206	189	Green	0	15	0.20	0.13	15	0.051
	(10)	34	204	168	material	(13.8)	16	0.15	0.12	16	0.048
						7	3.6	0.21	0.083	3.8	0.058
						(13.8)	3.7	0.21	0.11	3.9	0.049
										3.9	0.054

Trial No., Location, Year (Variety)	Application				Sample	DALA (% DM)	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	Parent + DFA	6-CNA
										(mean)	(mean)
						14	1.3	0.25	0.026	1.6	0.049
						(14.9)	1.4	0.31	0.025	1.7	0.044
						21	0.39	0.23	< 0.01	0.61	0.024
						(14.3)	0.14	0.21	< 0.01	0.35	0.020
						28	0.077	0.18	< 0.01	0.25	0.012
						(14.5)	0.082	0.17	< 0.01	0.25	0.014
						35	0.13	0.24	< 0.01	0.37	0.036
						(15.7)	0.083	0.21	< 0.01	0.30	0.020
					Hay	0	43	1.0	0.66	44	0.72
						(38.4)	40	1.1	0.64	41	0.65
						7	10	0.95	0.26	11	0.39
						(30.5)	10	0.71	0.24	11	0.33
							10 (mean)			11 (mean)	0.36 (mean)
						14	7.3	1.4	0.16	8.7	0.25
						(78.6)	5.9	1.1	0.14	7.0	0.26
						21	0.55	0.77	0.012	1.3	0.074
						(38.2)	0.67	0.59	0.011	1.3	0.090
						28	0.096	0.42	< 0.01	0.51	0.036
						(34.6)	0.19	0.56	< 0.01	0.76	0.042
						35	0.22	0.60	< 0.01	0.81	0.070
						(38.4)	0.11	0.50	< 0.01	0.61	0.046
RV188-11DA, Rosthern, Saskatchewan Canada, 2011	2	65	205	110	Green	0	6.1	0.32	0.025	6.4	0.076
	(10)	69	207	112	material	(14.3)	5.8	0.25	0.025	6.1	0.069
						6	3.3	0.48	0.023	3.8	0.15
						(15.0)	3.5	0.43	0.030	3.9	0.13
(Meadow)						13	3.1	0.65	0.017	3.7	0.28
						(20.2)	2.2	0.56	0.012	2.8	0.20
										3.3 (mean)	0.24 (mean)
						20	3.3	0.62	0.016	3.9	0.25
						(31.7)	2.4	0.62	< 0.01	3.1	0.26
						28	4.8	0.90	0.014	5.7	0.55
						(63.1)	3.4	0.85	0.012	4.3	0.51
						35	5.3	1.3	0.033	6.5	0.68
						(85.6)	6.2	1.4	0.033	7.6	0.89
										7.0 (mean)	
					Hay	0	21	3.1	0.16	24	2.4
						(33.1)	15	2.4	0.13	18	2.0
						6	8.0	1.8	0.05	9.8	1.3
						(49.0)	10	2.1	0.068	12	1.6
							9.1 (mean) *c0.13*			11 (mean)	1.5 (mean)
						13	8.6	1.5	0.058	10	0.82
						(62.9)	7.0	1.8	0.028	8.8	1.1
						20	6.0	1.6	0.019	7.6	1.1
						(81.5)	7.3	1.5	0.041	8.8	1.0
						28	6.1	1.0	0.024	7.1	0.57
						(85.6)	5.0	1.2	0.022	6.2	0.69
						35	5.2	1.1	0.016	6.4	0.80
						(88.1)	5.6	1.3	0.027	6.9	0.91
RV189-11DA, Wakaw,	2	62	202	108	Green	0	8.7	0.29	0.041	9.0	0.058
	(10)	67	205	111	material	(19.3)	9.7	0.32	0.039	10	0.062

Trial No., Location, Year (Variety)	Application				Sample	DALA (% DM)	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	Parent + DFA	6-CNA
Saskatchewan USA, 2011 (Meadow)						6	4.3	0.54	0.061	4.9	0.10
						(19.9)	4.9	0.46	0.058	5.4	0.084
						13	5.5	0.75	0.041	6.3	0.14
						(23.3)	3.3	0.61	0.031	3.9	0.11
										5.1 (mean)	
						20	2.1	0.62	0.011	2.7	0.24
						(26.1)	3.0	0.87	0.016	3.9	0.34
											0.29 (mean)
						28	4.1	1.2	0.020	5.4	0.54
						(58.2)	3.7	1.5	0.028	5.2	0.63
						35	4.2	1.4	0.028	5.6	0.72
						(83.4)	4.1	1.3	0.030	5.4	0.71
					Hay	0	8.8	3.7	0.12	12	2.0
						(28.5)	10	3.7	0.14	14	2.1
						6	5.3	1.9	0.048	7.1	0.74
						(41.1)	4.8	1.8	0.031	6.5	0.84
							5.0 (mean)			6.8 (mean)	0.79 (mean)
						13	3.8	1.6	0.028	5.4	0.71
						(49.8)	3.1	1.3	0.015	4.4	0.49
						20	4.9	2.1	0.018	7.1	1.2
						(80.7)	4.9	2.1	0.019	7.0	1.1
						28	3.5	1.8	0.017	5.3	0.80
						(82.0)	4.0	1.6	0.019	5.5	0.84
						35	3.8	1.8	0.033	5.6	1.1
						(86.5)	3.3	1.7	0.023	5.0	0.98

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.) in pea vines and hay

Table 133 Residues in bean forage and hay from foliar applications of flupyradifurone to beans in the USA and Canada (Hoag, Arthur and Woodard 2012, RARVY028)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
GAP, USA, Crop Group 6	2 (10)		205			7					
RV190-11HA, Marysville, Ohio USA, 2011 (Vista)	2	35	205	177	Forage	7	0.078	0.29	0.088	0.37	0.38
	(10)	39	205	173		(24.6)	0.032	0.15	0.036	0.18	0.17
										0.27 (mean)	0.28 (mean)
	2	80	205	185	Hay	7	< 0.040	< 0.20	< 0.040	< 0.24	< 0.01
	(10)	86	206	179		(80.9)	< 0.040	< 0.20	< 0.040	< 0.24	< 0.01
							< 0.040 (mean)			< 0.24 (mean)	< 0.01 (mean)
RV191-11HA, Lenexa, Kansas	2	12	210	203	Forage	7	1.3	0.38	0.45	1.7	0.52

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
USA, 2011 (Pink Eye Purple hull)	(9)	15	211	147		(12.2)	1.3	0.44	0.45	1.8	0.61
										1.7 (mean)	0.56 (mean)
	2	70	207	147	Hay	6	8.7	2.8	2.8	12	3.7
	(9)	75	202	143		(67.3)	7.0	2.8	2.6	9.8	3.9
							7.9 (mean)			11 (mean)	3.8 (mean)
RV192-11HA, Geneva, Minnesota USA, 2011 (Great Northern)	2	24	210	156	Forage	6	0.28	1.5	0.40	1.8	0.42
	(8)	34	207	172		(14.3)	0.27	1.6	0.41	1.8	0.58
										1.8 (mean)	0.50 (mean)
	2	82	206	190	Hay	7	2.6	1.1	0.16	3.6	1.3
	(9)	85	208	195		(71.4)	3.4	1.2	0.16	4.6	1.9
							3.0 (mean)			4.1 (mean)	1.6 (mean)
RV193-11HA, Taber, Alberta Canada, 2011 (AC Redbond)	2	25	203	198	Forage	7	0.42	0.60	1.7	1.0	0.61
	(9)	29	201	197		(12.6)	0.36	0.66	1.4	1.0	0.58
										1.0 (mean)	0.59 (mean)
	2	81	206	176	Hay	7	1.8	0.68	0.51	2.5	2.6
	(10)	85	207	175		(84.7)	3.7	1.4	0.96	5.0	2.8
							2.7 (mean)			3.7 (mean)	2.7 (mean)
RV194-11HA, Levelland, Texas USA, 2011 (Pinto)	2	39	208	188	Forage	7	2.2	1.8	0.82	4.1	0.39
	(8)	39	208	188		(19.6)	2.5	1.7	1.1	4.1	0.46
										4.1 (mean)	0.43 (mean)
RV195-11HA, Jerome, Idaho USA, 2011 (Othello)	2	25	209	198	Forage	7	1.3	0.53	0.86	1.8	0.43
	(8)	39	205	192		(14.1)	0.99	0.57	0.88	1.6	0.51
										1.7 (mean)	0.47 (mean)
	2	86	206	206	Hay	7	3.4	0.11	0.19	3.5	0.36
	(8)	88	208	206		(78.1)	0.93	0.33	0.16	1.3	1.2
							2.2 (mean)			2.4 (mean)	0.79 (mean)
RV196-11DA, Atlantic, Iowa USA, 2011 (Black Turtle)	2	30	204	192	Forage	0	15	0.13	0.39	15	0.18
	(10)	35	208	193		(13.9)	21	0.16	0.42	21	0.20
						7	0.70	0.45	1.1	1.1	0.54
						(13.1)	0.72	0.48	1.1	1.2	0.47
										1.2 (mean)	0.51 (mean)
						14	0.024	0.47	0.11	0.49	0.16
						(13.2)	0.029	0.56	0.12	0.59	0.20
						21	< 0.01	0.35	< 0.01	0.36	0.067
						(13.7)	0.011	0.37	0.014	0.38	0.072
						28	< 0.01	0.33	< 0.01	0.34	0.10
						(15.9)	< 0.01	0.35	< 0.01	0.36	0.061
						35	< 0.01	0.26	< 0.01	0.27	0.029
						(18.3)	< 0.01	0.38	< 0.01	0.39	0.05
	2	83	206	196	Hay	0	6.9	1.6	0.34	8.5	1.4
	(9)	87	208	212		(73.4)	5.3	1.2	0.35	6.5	1.0
						7	4.4	1.2	0.17	5.5	0.98

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
						(80.0)	5.0	1.3	0.18	6.3	0.94
							4.7 (mean)			5.9 (mean)	0.96 (mean)
						14	2.5	1.1	0.046	3.6	0.87
						(61.9)	2.4	0.91	< 0.040	3.3	0.48
						21	2.6	1.2	0.043	3.8	0.68
						(76.0)	2.2	1.6	0.047	3.8	0.95
						28	1.5	1.7	0.044	3.2	0.79
						(73.6)	3.1	1.4	0.058	4.5	0.76
						35	2.3	1.9	0.054	4.1	0.80
						(82.5)	2.1	2.1	0.055	4.1	0.97
RV197-11DA, Eldridge, North Dakota	2	24	207	140	Forage	0	8.5	0.26	0.38	8.8	0.23
	(10)	50	208	142		(12.8)	7.9	0.27	0.42	8.2	0.24
						7	0.95	0.60	0.74	1.5	0.50
						(14.8)	0.68	0.64	0.62	1.3	0.49
USA, 2011 (Navigator)										1.4 (mean)	0.50 (mean)
						14	0.039	0.66	0.10	0.70	0.34
						(14.8)	0.027	0.63	0.12	0.65	0.32
						21	0.01	0.70	0.015	0.72	0.15
						(15.1)	0.015	0.79	0.028	0.81	0.26
						28	< 0.01	0.78	< 0.01	0.79	0.17
						(17.5)	< 0.01	0.81	< 0.01	0.82	0.21
						35	< 0.01	0.93	< 0.01	0.94	0.24
						(23.7)	< 0.01	0.83	< 0.01	0.84	0.24
	2	80	211	144	Hay	0	13	0.73	0.66	14	1.4
	(9)	87	208	144		(85.1)	18	1.2	1.1	19	2.1
						7	6.7	1.0	0.83	7.8	1.9
						(88.3)	8.5	1.2	1.1	9.7	2.0
							7.6 (mean)			8.7 (mean)	2.0 (mean)
						14	4.2	0.47	0.26	4.7	1.4
						(86.8)	3.1	0.45	0.23	3.5	1.5
						21	2.3	0.43	0.18	2.8	0.90
						(82.7)	2.6	0.55	0.21	3.2	1.2
						27	1.7	0.37	0.14	2.1	0.85
						(59.8)	2.8	0.42	0.20	3.2	1.0
						33	1.9	0.19	0.099	2.1	0.74
						(83.1)	1.3	0.24	0.11	1.6	1.1
RV198-11DA, Kerman, California USA, 2011 (Blue Lake 274)	2	30	204	233	Forage	0	14	0.80	1.7	15	0.61
	(10)	51	204	233		(17.3)	14	0.80	1.8	15	0.55
						7	2.1	1.1	3.2	3.2	0.98
						(17.8)	2.3	0.97	3.8	3.3	0.91
										3.2 (mean)	0.95 (mean)
						14	0.54	1.1	1.4	1.7	0.62
						(18.7)	0.48	1.2	1.8	1.7	0.67
						21	0.20	1.0	0.78	1.2	0.54
						(19.1)	0.13	1.1	0.64	1.2	0.35
						28	0.069	1.2	0.25	1.3	0.32
						(18.0)	0.062	1.2	0.28	1.3	0.23
						34	0.033	1.2	0.10	1.2	0.25
						(15.8)	0.043	1.1	0.082	1.1	0.16
	2	73	205	187	Hay	0	8.7	0.42	0.69	9.1	0.26

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
	(10)	89	204	187		(40.7)	8.5	0.61	0.81	9.1	0.40
						7	2.3	0.83	1.0	3.1	0.54
						(31.4)	1.8	1.2	1.5	3.1	0.63
						14	9.1	0.30	0.59	9.4	0.57
						(66.1)	10	0.46	0.62	11	0.46
							9.8 (mean)			10 (mean)	
						21	6.3	1.4	0.99	7.7	1.4
						(68.6)	4.2	0.66	0.34	4.8	0.58
						28	0.91	0.62	0.13	1.5	0.44
						(30.8)	0.52	0.69	0.11	1.2	0.49
						35	0.82	0.73	0.10	1.5	0.82
						(33.7)	0.53	0.52	0.065	1.0	0.60
											0.71 (mean)
RV199-11DA, Rupert, Idaho USA, 2011 (Bill Z)	2	11	204	162	Forage	0	12	0.36	0.94	12	0.45
	(10)	15	203	162		(15.2)	13	0.32	0.80	14	0.43
						7	0.83	1.5	1.1	2.3	0.73
						(15.1)	0.76	1.5	1.1	2.3	0.77
											0.75 (mean)
						14	0.33	2.2	0.47	2.5	0.40
						(15.6)	0.29	1.8	0.40	2.1	0.34
						21	0.16	2.7	0.25	2.8	0.27
						(16.4)	0.18	2.6	0.20	2.8	0.24
										2.8 (mean)	
						28	0.057	3.1	0.13	3.1	0.23
						(17.3)	0.045	2.2	0.11	2.2	0.16
						35	0.021	2.3	0.034	2.3	0.12
						(15.8)	0.023	2.3	0.044	2.3	0.14
	2	78	202	148	Hay	0	8.3	1.4	0.83	9.8	2.9
	(10)	81	202	159		(88.0)	8.8	1.5	0.65	10	3.3
						7	0.66	1.3	0.20	2.0	2.7
						(88.8)	0.76	1.2	0.29	2.0	2.6
							0.71 (mean)			2.0 (mean)	
						14	0.22	0.52	0.12	0.73	2.3
						(89.7)	0.31	1.2	0.089	1.5	2.8
						21	0.21	1.2	0.088	1.4	2.7
						(89.9)	0.17	1.1	0.093	1.2	2.7
						28	0.18	1.6	0.072	1.8	2.2
						(86.6)	0.36	1.1	0.11	1.4	2.6
						35	0.19	1.1	0.082	1.3	2.4
						(88.6)	0.18	0.97	0.071	1.2	3.5
											2.9 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.) in bean forage

LOQ is 0.04 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.20 mg/kg for DFA (parent equivs.) in bean hay

Table 134 Residues in soya bean forage and hay from foliar or seed treatment applications of flupyradifurone to soya beans in the USA and Canada (Sturdivant 2012, RARVY011)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
GAP, USA, Crop Group 6	2 (10)		205			7					
RV132-10DA,	2	16	204	211	Forage	0	15.2	0.62	0.68	15.8	0.38
Seven Springs,	(10)	65	208	224		(27)	17.4	0.69	0.79	18.1	0.47
North Carolina						3	7.23	0.94	1.13	8.2	0.83
USA, 2010						(26)	9.69	1.20	1.31	10.9	0.93
(AG5605)						7	4.04	2.16	1.56	6.2	1.12
						(27)	3.46	1.63	1.16	5.1	0.79
										5.6 (mean)	0.95 (mean)
						14	1.51	2.40	1.19	3.9	0.63
						(32)	1.26	2.40	1.06	3.7	0.57
						21	0.089	2.04	0.26	2.1	0.35
						(29)	0.082	2.05	0.25	2.1	0.37
					Hay	0	31.6	1.34	1.36	32.9	1.55
						(83)	32.9	1.47	1.61	34.4	1.81
						3	24.5	4.04	3.85	28.5	3.90
						(72)	17.1	3.33	3.07	20.4	3.01
						7	11.8	4.41	3.20	16.2	2.42
						(71)	10.6	4.33	3.30	15.0	2.25
							11 (mean)			16 (mean)	2.3 (mean) c0.016
						14	1.71	2.92	1.29	4.6	0.94
						(70)	3.48	5.53	2.40	9.0	1.73
						21	0.22	6.75	0.49	7.0	1.23
						(79)	0.14	4.5	0.31	4.6	0.76
RV133-10HA,	2	51	204	218	Forage	7	1.65	1.51	0.74	3.2	1.00
Tallassee,	(10)	67	206	237		(24)	2.24	1.81	1.09	4.0	1.08
Alabama							c0.01			3.6 (mean)	1.0 (mean)
USA, 2010					Hay	7	6.39	6.85	3.09	13.2	3.92
(Stine 4782-4)						(89)	6.49	5.77	2.90	12.3	3.43
							6.4 (mean)			13 (mean)	3.7 (mean) c0.038
	1	00	32		Fora	72	0.084	1.38	0.071	1.5	0.13
	(Seed)				ge	(28)	0.061	1.27	0.053	1.3	0.11
							c0.01				c0.038
					Hay	72	0.14	2.49	0.12	2.6	0.36
						(91)	0.1	2.29	0.08	2.4	0.26
RV134-10DA,	2	15	204	187	Forage	0	13.2	0.27	0.51	13.5	0.32
Proctor,	(10)	63	205	187		(20)	13.3	0.27	0.53	13.6	0.31
Arkansas						3	8.77	0.50	0.92	9.3	0.56
USA, 2010						(19)	7.87	0.50	0.86	8.4	0.55
(Armor 47G7)						7	3.77	0.58	0.84	4.4	0.52
						(21)	4.75	0.70	1.06	5.5	0.56
										4.9 (mean)	
						14	1.40	0.69	0.81	2.1	0.51
						(19)	1.28	0.71	0.85	2.0	0.50
											0.51 (mean)
						21	0.36	0.87	0.50	1.2	0.38
						(24)	0.52	0.87	0.49	1.4	0.36
					Hay	0	34.0	0.52	1.33	34.5	0.55

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
						(55)	35.1	0.51	1.10	35.6	0.58
						3	17.0	0.80	1.39	17.8	0.87
						(46)	14.4	0.81	1.51	15.2	0.92
						7	6.59	1.37	1.85	7.96	1.21
						(46)	10.4	1.81	2.56	12.2	1.5
							8.5 (mean)			10 (mean)	1.4 (mean) c0.015
						14	3.14	1.53	1.83	4.7	1.21
						(50)	3.83	1.68	2.06	5.5	1.36
						21	0.50	1.09	0.73	1.6	0.71
						(52)	0.67	1.43	0.85	2.1	0.77
RV135-10HA,	2	63	206	189	Forage	6	0.93	1.21	0.80	2.1	0.65
Fisk,	(9)	67	205	187		(24)	1.08	1.07	0.72	2.2	0.55
Missouri										2.1 (mean)	0.60 (mean)
USA, 2010					Hay	6	3.03	6.19	2.47	9.2	3.36
(Stine 4782-4)						(84)	4.78	7.14	2.48	11.9	3.08
							3.9 (mean)			11 (mean)	3.2 (mean)
	1 (Seed)	00	51		Forage	58	0.037	2.03	0.059	2.1	0.10
						(23)	0.022	2.16	0.047	2.2	0.11
					Hay	58	0.026	1.00	0.026	1.0	0.09
						(80)	< 0.01	1.07	0.017	1.1	0.09
RV136-10HA,	2	60	203	186	Forage	7	2.43	1.5	1.89	3.9	1.37
Pollard, Arkansas	(10)	67	204	186		(20)	3.25	1.61	2.33	4.9	1.52
USA, 2010										4.4 (mean)	1.4 (mean)
(Pioneer 94M80)					Hay	7	7.22	4.51	6.70	11.7	4.84
						(84)	6.58	4.41	6.16	11.0	4.99
							6.9 (mean)			11 (mean)	4.9 (mean) c0.059
RV137-10DB,	2	65	210	310	Forage	0	11.4	0.38	0.55	11.8	0.44
Branchton,	(8)	66	196	290		(21)	10.9	0.37	0.54	11.3	0.45
Ontario						3	5.31	0.73	1.02	6.0	0.96
Canada, 2010						(22)	4.48	0.62	0.81	5.1	0.79
(Secan RCAT Matrix)						7	1.95	0.96	0.96	2.9	1.05
						(21)	2.48	1.14	1.25	3.6	1.16
										3.3 (mean)	1.1 (mean) c0.013
						14	1.37	1.42	0.79	2.8	1.05
						(27)	1.04	1.24	0.62	2.3	0.99
						21	0.42	1.41	0.29	1.8	1.04
						(28)	0.37	1.56	0.29	1.9	1.04
					Hay	0	26.2	1.35	1.96	28	2.81
						(72)	25.1	0.89	1.47	26	2.16
						3	4.51	2.18	2.05	6.7	2.54
						(55)	11.1	3.08	2.67	14.2	5.62
						7	9.58	2.88	3.41	12.5	4.97
						(67)	2.85	1.46	1.40	4.3	1.59
							6.2 (mean)			8.4 (mean)	c0.033
						14	1.17	1.85	0.46	3.0	2.96
						(50)	0.94	1.49	0.4	2.4	2.53
											2.7 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
						21	0.43	1.41	0.24	1.8	1.19
						(59)	0.38	1.81	0.29	2.2	1.56
RV138-10HA,	2	61	207	137	Fora	5	2.73	0.93	1.50	3.7	1.10
Springfield,	(9)	64	208	132	ge	(18)	3.04	0.93	1.38	4.0	0.92
Nebraska										3.8 (mean)	1.0 (mean)
USA, 2010					Hay	5	7.01	3.50	4.11	10.5	3.22
(NC+3051R)						(52)	9.09	3.28	4.56	12.4	3.12
							8.1 (mean)			11 (mean)	3.2 (mean)
	1 (Seed)	00	49		Forage	60	< 0.01	0.10	< 0.01	0.11	< 0.01
						(17)	< 0.01	0.11	< 0.01	0.12	< 0.01
					Hay	60	< 0.01	0.26	< 0.01	0.27	< 0.01
						(49)	0.016	0.37	0.017	0.39	0.068
RV148-10HA,	2	60	201	137	Forage	6	3.36	1.38	1.81	4.7	1.21
Springfield, Nebraska	(9)	63	208	139		(15)	4.44	1.52	1.97	6.0	1.50
USA, 2011										5.4 (mean)	1.4 (mean)
(S28-B4)					Hay	6	7.20	4.46	3.69	12	3.73
						(43)	5.98	4.11	3.69	10	3.11
							6.6 (mean)			11 (mean)	3.4 (mean) c0.016
RV139-10HA,	2	14	205	140	Forage	7	0.87	0.77	0.79	1.6	0.59
York, Nebraska	(10)	16	213	142		(15)	1.00	0.82	0.89	1.8	0.65
USA, 2010										1.7 (mean)	0.62 (mean)
(NC+2751R)					Hay	7	3.04	4.27	2.18	7.3	4.64
						(59)	2.08	3.92	2.03	6.0	4.12
							2.6 (mean)			6.7 (mean)	4.4 (mean)
RV150-10HA,	2	51	204	193	Forage	7	3.06	1.72	1.87	4.8	1.08
York, Nebraska	(8)	66	205	188		(18)	4.06	1.71	2.05	5.8	0.96
USA, 2011							c0.01			5.3 mean	1.0 (mean)
(16501RR)					Hay	7	11.5	7.82	5.13	19	6.43
						(82)	11.8	8.56	8.36	20	6.44
							12 (mean) c0.016			20 (mean)	6.4 (mean) c0.12
RV140-10HA,	2	63	207	119	Forage	7	3.32	1.60	1.68	4.9	1.40
Rockwood, Ontario	(10)	65	205	113		(19)	2.72	1.41	1.57	4.1	1.07
Canada, 2010							c0.034 av			4.5 (mean)	1.2 (mean) c0.018 av
(90M01)					Hay	7	8.42	4.05	1.71	12.5	5.29
						(83)	8.66	4.59	2.68	13.3	6.37
							8.5 (mean)			13 (mean)	5.8 (mean) c0.086 av
RV141-10HA,	2	60	205	187	Fora	7	2.25	1.44	1.58	3.7	1.52
Campbell,	(9)	69	206	188	ge	(17)	2.79	1.66	1.89	4.5	1.68
Minnesota										4.1 (mean)	1.6 (mean)
USA, 2010					Hay	7	6.69	4.03	4.09	10.7	4.60

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
(AG 0808)						(77)	6.43	3.75	3.63	10.2	4.46
							6.6 (mean)			10 (mean)	4.5 (mean)
RV142-10HA,	2	16	206	171	Forage	7	4.23	0.52	0.30	4.8	0.30
Clarence, Missouri	(7)	66	209	190		(21)	4.72	0.56	0.35	5.3	0.32
USA, 2010										5.0 (mean)	0.31 (mean)
(Asgrow 3803)					Hay	7	13.8	1.32	1.01	15.1	0.94
						(65)	11.4	1.20	0.85	12.6	0.82
							13 (mean) c0.013av			15 (mean)	0.88 (mean)
RV143-10HA,	2	64	211	118	Forage	7	4.47	1.07	1.01	5.5	0.94
Breslau, Ontario	(10)	66	204	119		(22)	3.66	1.0	1.11	4.7	0.95
Canada, 2010										5.1 (mean)	0.95 (mean)
(DKBOO-99)					Hay	7	7.01	2.7	1.09	9.7	4.36
						(76)	6.48	2.9	1.22	9.4	4.52
							6.7 (mean) *c0.016av.*			9.5 (mean)	4.4 (mean) c0.031 av
RV144-10HA,	2	62	207	185	Forage	7	1.18	1.47	1.46	2.7	1.10
Richland, Iowa	(10)	69	207	185		(18)	1.03	1.28	1.12	2.3	0.94
USA, 2010										2.5 (mean)	1.0 (mean)
(Pioneer 92Y80)					Hay	7	4.50	5.09	3.83	9.6	5.23
						(68)	3.88	5.24	4.13	9.1	4.85
							4.2 (mean)			9.4 (mean)	5.0 (mean)
RV145-10HA,	2	61	205	109	Forage	7	2.75	1.61	1.59	4.4	1.98
Cambridge,	(9)	65	205	110		(23)	2.81	1.56	1.60	4.4	1.98
Ontario										4.4 (mean)	2.0 (mean)
Canada, 2010					Hay	7	1.70	2.16	0.47	3.9	5.88
(90M40)						(71)	1.23	1.69	0.23	2.9	4.10
							1.5 (mean) c0.012			3.4 (mean)	5.0 (mean)
RV146-10DA,	2	11	206	189	Forage	0	22.1	0.13	0.31	22.2	0.23
Northwood,	(10)	59	202	256		(19)	19.1	0.087	0.28	19.2	0.19
North Dakota						3	12.3	0.28	0.63	12.6	0.42
USA, 2010						(19)	10.1	0.23	0.53	10.3	0.42
(Asgrow AG00901)						7	5.21	0.25	0.73	5.5	0.52
						(18)	5.39	0.26	0.67	5.7	0.44
										5.6 (mean)	0.48 (mean)
						14	1.4	0.23	0.46	1.6	0.33
						(17)	0.77	0.19	0.38	0.96	0.25
						21	0.09	0.14	0.11	0.24	0.12
						(17)	0.12	0.16	0.14	0.28	0.14
					Hay	0	38.1	0.29	0.73	38	0.48
						(77)	30.7	0.24	0.74	31	0.50
						3	34.5	0.92	1.84	35.4	1.35
						(79)	32.8	0.67	2.00	33.5	1.21
						7	13.5	0.65	1.93	14	1.25

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
						(80)	5.19	1.41	2.51	6.6	1.78
							9.3 (mean)			10 (mean)	1.5 (mean) c0.032 av
						14	7.56	0.30	0.91	7.9	0.60
						(74)	2.75	1.07	1.04	3.8	0.79
						21	0.51	0.59	0.43	1.1	0.53
						(76)	0.32	0.54	0.36	0.86	0.57
RV147-10HA,	2	63	203	187	Fora	7	5.88	1.46	2.25	7.3	0.95
Stafford, Kansas	(7)	65	205	188	ge	(20)	7.65	1.51	2.47	9.2	1.10
USA, 2011										8.2 (mean)	1.0 (mean)
(Pioneer 93Y70)					Hay	7	19.7	4.31	7.65	24	3.05
						(58)	15.01	3.95	6.67	19	3.00
							17 (mean)			21 (mean)	3.0 (mean)
RV149-10HA,	2	61	207	145	Forage	7	3.81	1.27	1.70	5.1	0.69
Lenexa, Kansas	(10)	64	209	147		(23)	4.34	1.23	1.51	5.6	0.63
USA, 2011										5.3 (mean)	0.66 (mean)
(Willcross RR2428N)					Hay	7	14.1	3.05	4.48	17.2	2.18
						(81)	15.5	2.99	4.94	18.5	2.44
							15 (mean)			18 (mean)	2.3 (mean) c0.057
RV151-10HA,	2	14	210	219	Fora	7	5.26	0.86	1.31	6.1	0.77
Kimballton, Iowa	(10)	59	201	312	ge	(17)	4.70	0.89	1.54	5.6	0.79
USA, 2011										5.9 (mean)	0.78 (mean)
(Stine 2862-4)					Hay	7	7.48	1.39	1.67	8.9	1.68
						(54)	9.13	1.52	1.57	11	1.81
							8.3 (mean)			9.8 (mean)	1.7 (mean) c0.037

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.) for both forage and hay

Table 135 Residues in barley straw and hay from foliar or seed treatment application of flupyradifurone to barley in the USA and Canada (Hoag 2012a, RARVY001)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
GAP, USA, Crop Group 15	2 (7)		205			Hay 7, Straw 21					
RV001-10HA,	2	51	204	173	Hay	7 (63)	13	1.6	0.99	14	1.7
Germansville, Pennsylvania	(7)	59	206	174			12	1.6	0.91	13	1.5
USA, 2010							12 (mean)			14 (mean)	1.6 (mean)
(AC Minoa)	2	77	207	175	Straw	16 (89)	3.8	0.38	< 0.05	4.2	0.25
	(6)	85	207	175			2.5	0.38	< 0.05	2.8	0.23
							3.1 (mean)			3.5	0.24

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
										(mean)	(mean)
RV002-10HA,	2	65	208	136	Hay	6 (60)	1.5	0.58	0.19	2.0	0.28
Springfield, Nebraska	(5)	69	209	135			1.3	0.56	0.22	1.8	0.23
USA, 2010							1.4 (mean)			1.9 (mean)	0.25 (mean)
(Robust)	2	71	208	137	Straw	19 (54)	0.41	0.54	< 0.05	0.95	0.64
	(5)	83	206	134			0.42	0.64	< 0.05	1.1	0.71
							0.42 (mean)			1.0 (mean)	0.67 (mean)
	Seed	00	123		Hay	73 (59)	< 0.05	0.32	< 0.05	0.37	< 0.01
							< 0.05	0.31	< 0.05	0.36	< 0.01
					Straw	92 (47)	< 0.05	0.21	< 0.05	0.26	0.04
							< 0.05	0.12	< 0.05	0.17	0.05
RV003-10DA,	2	47	210	296	Hay	0 (52)	19	0.39	0.52	20	0.29
Carlyle, Illinois	(8)	52	209	343			16	0.39	0.54	16	0.26
USA, 2010						3 (52)	6.0	0.34	0.27	6.3	0.21
(–)							6.3	0.33	0.25	6.6	0.18
						7 (45)	0.79	0.31	0.086	1.1	0.13
							0.66	0.24	0.070	0.9	0.13
							0.73 (mean)			1.0 (mean)	0.13 (mean)
						14 (70)	0.84	0.57	0.171	1.4	0.11
							0.84	0.57	0.152	1.41	0.12
						21 (56)	0.29	0.56	0.073	0.85	0.12
							0.25	0.50	0.062	0.75	0.10
	2	75	209	255	Straw	10 (73)	9.7	0.30	0.20	10	0.70
	(7)	83	208	248			11	0.29	0.23	11	0.64
						15 (74)	2.1	0.13	< 0.05	2.3	0.077
							2.2	0.11	< 0.05	2.3	0.11
						20 (77)	1.2	0.11	< 0.05	1.3	0.070
							1.4	0.11	< 0.05	1.6	< 0.05
							1.3 (mean)			1.4 (mean)	0.060 (mean)
						28 (85)	0.96	0.079	< 0.05	1.0	< 0.05
							1.0	0.080	< 0.05	1.1	< 0.05
						35 (83)	0.74	0.14	< 0.05	0.88	< 0.05
							0.79	0.11	< 0.05	0.90	< 0.05
RV004-10HA,	2	58	256	119	Hay	7 (66)	0.30	0.33	< 0.05	0.63	0.21
Rockwood, Ontario	(7)	65	206	116			0.37	0.33	< 0.05	0.70	0.22
Canada, 2010							0.33 (mean)			0.66 (mean)	0.22 (mean)
(Dignity)	2	83	202	109	Straw	21 (70)	3.9	0.25	< 0.05	4.1	0.15
	(7)	85	206	114			4.1	0.25	< 0.05	4.3	0.16
							4.0 (mean)			4.2 (mean)	0.16 (mean)
RV005-10HA,	2	58	207	191	Hay	7 (74)	1.7	0.73	0.14	2.4	0.18
Grand Island, Nebraska	(7)	71	207	189			1.4	0.72	0.11	2.1	0.18
USA, 2010							1.5 (mean) c0.10			2.3 (mean)	0.18 (mean)
(Robust)	2	75	207	190	Straw	22 (70)	1.0	0.20	< 0.05	1.2	0.23
	(7)	85	205	190			1.0	0.26	< 0.05	1.3	0.24
							1.0 (mean)			1.3 (mean)	0.23 (mean)
	Seed	0	202		Hay	74 (62)	< 0.05	0.74	< 0.05	0.79	0.02
							< 0.05	0.88	< 0.05	0.93	< 0.05
					Straw	110 (60)	< 0.05	0.16	< 0.05	0.206	0.07
							< 0.05	0.13	< 0.05	0.179	0.06

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
RV006-10HA,	2	54	205	187	Hay	0 (62)	11	0.55	0.54	12	0.54
Carrington, North Dakota	(6)	73	206	189			11	0.53	0.50	12	0.51
USA, 2010						3 (55)	9.9	0.53	0.53	10	0.49
(Pinneacle)							7.4	0.52	0.54	7.9	0.43
						5 (79)	7.3	0.86	0.57	8.2	1.2
							7.2	0.75	0.53	8.0	1.1
						14 (71)	1.3	0.46	0.077	1.8	0.43
							1.8	0.59	0.12	2.4	0.57
						22 (87)	1.4	0.71	0.087	2.1	0.72
							1.3	0.67	0.073	2.0	0.69
	2	77	204	187	Straw	10 (40)	0.94	0.13	< 0.05	1.1	0.15
	(5)	83	204	186			1.3	0.24	0.073	1.5	0.26
						15 (44)	1.9	0.27	0.075	2.2	0.39
							2.4	0.281	0.082	2.7	0.44
						21 (35)	0.30	0.16	< 0.05	0.46	0.078
							0.36	0.16	< 0.05	0.51	0.093
							0.33 (mean)			0.49 (mean)	
						29 (63)	0.67	0.27	< 0.05	0.94	0.21
							0.86	0.29	< 0.05	1.2	0.24
											0.22 (mean)
						35 (56)	0.59	0.21	< 0.05	0.80	0.12
							0.43	0.19	< 0.05	0.62	0.092
RV007-10HA,	2	54	203	184	Hay	5 (64)	4.2	0.38	0.18	4.6	0.62
Oberon, North Dakota	(8)	75	205	188			5.2	0.35	0.15	5.5	0.60
USA, 2010	2	83	203	187	Straw	21 (41)	0.44	0.16	< 0.05	0.60	0.082
(Pinneacle)	(5)	85	209	190			0.40	0.16	< 0.05	0.56	0.073
							0.42 (mean)			0.58 (mean)	0.077 (mean)
RV008-10HA,	2	58	207	187	Hay	7 (76)	1.7	0.39	0.15	2.1	0.17
Jamestown, North Dakota	(5)	69	204	141			1.9	0.37	0.14	2.2	0.18
USA, 2010							1.8 (mean)			2.2 (mean)	0.18 (mean)
(Tradition)	2	83	208	189	Straw	19 (81)	1.3	0.12	< 0.05	1.46	0.11
	(5)	85	208	191			1.4	0.10	< 0.05	1.5	0.13
							1.4 (mean)			1.5 (mean)	0.12 (mean)
RV009-10HA,	2	43	206	193	Hay	7 (66)	8.1	0.37	0.45	8.5	0.082
Jerome, Idaho	(7)	52	205	179			6.2	0.38	0.40	6.6	0.10
USA, 2010							7.2 (mean)			7.5 (mean)	0.092 (mean)
(Harrington)	2	83	207	188	Straw	20 (84)	5.4	< 0.05	0.11	5.4	< 0.05
	(7)	87	210	192			5.8	< 0.05	0.10	5.9	< 0.05
							5.6 (mean)			5.6 (mean)	< 0.05 (mean)
RV010-10HA,	2	75	211	291	Hay	7 (70)	4.4	0.46	0.26	4.8	0.23
Sanger, California	(5)	77	208	299			2.3	0.37	0.19	2.7	0.17
USA, 2010							3.3 (mean)			3.7 (mean)	0.20 (mean)
(UC937)	2	85	204	289	Straw	21 (43)	3.0	0.081	0.076	3.1	0.23
	(7)	87	207	300			1.9	0.070	0.055	2.0	0.12
							2.5 (mean)			2.5 (mean)	0.18 (mean)
RV011-10HA,	2	71	205	280	Hay	7 (92)	5.7	0.33	0.40	6.0	0.056
Ephrata, Washington	(7)	77	207	283			4.7	0.25	0.29	5.0	< 0.05

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
USA, 2010							5.2 (mean)			5.5 (mean)	0.053 (mean)
(AC Metcalfe)	2	85	205	280	Straw	21 (78)	3.3	0.10	0.13	3.4	0.19
	(7)	87	207	282			4.4	0.13	0.16	4.5	0.15
							3.8 (mean)			3.9 (mean)	0.17 (mean)
RV012-10HA,	2	69	209	238	Hay	7 (90)	6.43	0.66	0.76	1.31	0.11
Payette, Idaho	(7)	73	204	232			5.13	0.66	0.70	5.79	0.10
USA, 2010							2.9 (mean)			3.5 (mean)	0.11 (mean)
(Champion)	2	73	205	233	Straw	21 (94)	0.69	0.39	0.074	1.1	0.074
	(7)	77	209	238			0.86	0.35	0.095	1.2	0.088
							0.77 (mean)			1.1 (mean)	0.081 (mean)
	Seed	0	195		Hay	80 (87)	< 0.05	0.11	< 0.05	0.16	0.02
							< 0.05	0.13	0.056	0.18	0.02
					Straw	101 (94)	< 0.05	0.061	< 0.05	0.11	0.04
							< 0.05	0.058	< 0.05	0.11	< 0.05
RV013-10DA,	2	52	207	101	Hay	0 (41)	5.9	0.80	0.62	6.7	1.2
Josephburg, Alberta	(7)	59	207	101			4.6	0.62	0.40	5.2	0.85
Canada, 2010						3 (45)	2.2	0.51	0.31	2.7	0.81
(Coalition)							2.7	0.53	0.34	3.2	0.81
						8 (37)	0.43	0.43	0.061	0.86	0.47
							0.42	0.49	0.049	0.91	0.40
							0.42 (mean)			0.89 (mean)	0.44 (mean)
						13 (85)	0.50	0.54	0.067	1.1	0.44
							0.54	0.45	0.067	0.99	0.34
						21 (63)	0.23	0.41	< 0.05	0.64	0.44
							0.28	0.40	< 0.05	0.69	0.46
	2	73	206	101	Straw	10 (28)	3.0	0.25	0.12	3.3	0.30
	(8)	85	206	99			4.0	0.27	0.124	4.3	0.33
						13 (33)	1.9	0.19	0.082	2.1	0.23
							1.9	0.21	0.068	2.1	0.30
						19 (32)	2.2	0.28	0.090	2.4	0.50
							2.3	0.21	0.077	2.5	0.45
							2.2 (mean)			2.5 (mean)	0.47 (mean)
						27 (28)	1.3	0.21	< 0.05	1.6	0.31
							1.4	0.21	< 0.05	1.6	0.29
						34 (76)	2.1	0.35	0.068	2.4	0.57
							1.1	0.26	< 0.05	1.3	0.38
RV014-10DA,	2	52	203	200	Hay	0 (87)	45	0.70	0.79	46	0.41
Alvena, Saskatchewan	(5)	61	211	207			35	0.62	0.71	36	0.35
Canada, 2010						3 (82)	34	0.96	0.83	35	0.72
(Metcalf)							32	0.72	0.71	33	0.59
						6 (87)	22	0.78	0.70	23	0.51
							26	1.1	0.85	27	0.90
							24 (mean)			25 (mean)	0.70 (mean)
						13 (84)	2.2	0.54	0.058	2.8	0.19
							3.0	0.72	0.083	3.7	0.30
						20 (84)	1.6	0.54	< 0.05	2.1	0.42
							1.8	0.70	< 0.05	2.5	0.64
	2	65	211	207	Straw	9 (46)	3.4	0.27	0.078	3.7	0.26
	(5)	72	195	192			3.1	0.28	0.076	3.4	0.29
						15 (55)	3.3	0.32	0.065	3.7	0.36

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
							3.9	0.35	0.063	4.3	0.39
						20 (52)	5.1	0.26	0.064	5.4	0.34
							2.5	0.31	0.071	2.8	0.43
							3.8 (mean)			4.1 (mean)	0.38 (mean)
						28 (62)	0.92	0.21	< 0.05	1.1	0.096
							1.0	0.24	< 0.05	1.2	0.069
						34 (31)	0.48	0.19	< 0.05	0.67	0.039
							0.46	0.19	< 0.05	0.65	0.043
RV015-10HA,	2	58	209	205	Hay	8 (86)	3.7	0.65	0.16	4.3	0.21
Wakaw, Saskatchewan	(6)	64	212	208			3.9	0.69	0.17	4.5	0.28
Canada, 2010							3.8 (mean)			4.4 (mean)	0.25 (mean)
(Ranger)	2	58	201	198	Straw	21 (33)	0.84	0.24	< 0.05	1.1	0.30
	(6)	64	201	197			1.0	0.24	< 0.05	1.3	0.35
							0.92 (mean)			1.2 (mean)	0.32 (mean)
RV016-10HA,	2	49	204	158	Hay	7 (80)	19	0.87	1.0	20	0.33
Minto, Manitoba	(7)	69	204	158			16	0.78	0.88	17	0.27
Canada, 2010							17 (mean)			18 (mean)	0.30 (mean)
(CDC Copeland)	2	83	204	158	Straw	21 (57)	0.55	0.22	< 0.05	0.77	0.17
	(7)	87	204	158			0.66	0.23	< 0.05	0.89	0.20
							0.61 (mean)			0.83 (mean)	0.19 (mean)
RV017-10HA,	2	41	210	162	Hay	7 (64)	2.8	0.40	0.24	3.2	0.13
Boissevain, Manitoba	(6)	59	206	160			1.3	0.33	0.15	1.6	0.10
Canada, 2010							2.0 (mean)			2.4 (mean)	0.12 (mean)
(Metcalfe)	2	77	204	159	Straw	21 (44)	0.52	0.25	< 0.05	0.77	0.46
	(7)	83	209	162			0.53	0.34	< 0.05	0.87	0.57
							0.52 (mean)			0.82 (mean)	0.51 (mean)
RV018-10HA,	2	61	203	197	Hay	5 (73)	10	0.85	0.43	11	1.3
Wellwood, Manitoba	(7)	69	195	191			7.3	0.69	0.39	8.0	1.1
Canada, 2010							8.8 (mean)			9.6 (mean)	1.2 (mean)
(Tradition)	2	65	203	198	Straw	21 (28)	0.31	0.090	< 0.05	0.40	< 0.05
	(7)	71	198	193			0.32	0.091	< 0.05	0.41	< 0.05
							0.31 (mean)			0.41 (mean)	< 0.05 (mean)
RV019-10HA,	2	54	207	102	Hay	7 (55)	4.5	0.60	0.36	5.1	1.0
Fort Saskatchewan, Alberta	(7)	59	208	104			6.3	0.68	0.46	7.0	1.3
Canada, 2010							5.4 (mean)			6.1 (mean)	1.2 (mean)
(Coalition)	2	77	204	100	Straw	19 (44)	5.0	0.19	0.10	5.2	0.35
	(8)	85	199	98			5.1	0.18	0.10	5.3	0.41
							5.1 (mean)			5.2 (mean)	0.38 (mean)
RV020-10DA,	2	55	207	201	Hay	7 (55)	1.8	0.43	0.070	2.2	0.27
Waldheim, Saskatchewan	(7)	59	201	195			1.4	0.45	0.063	1.9	0.31
Canada, 2010							1.6 (mean)			2.0 (mean)	0.29 (mean)
(Metcalf)	2	59	207	201	Straw	20 (45)	1.2	0.12	< 0.05	1.3	0.080
	(7)	77	210	204			1.4	0.13	0.017	1.6	0.084
							1.3 (mean)			1.4	0.082

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
										(mean)	(mean)

LOQ is 0.05 mg/kg for each of parent flupyradifurone and the metabolites DFEAF, DFA and 6-CNA (parent equivs.)

Table 136 Residues in wheat forage, hay and straw from foliar or seed treatment application of flupyradifurone to wheat in the USA and Canada (Fischer and Niczyporowicz 2012, RARVY003)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
GAP, USA, Crop Group 15	2 (7)		205			Forage 7, Hay 7, Straw 21					
RV054-10HA, Seven Springs, North Carolina USA, 2010 (Pioneer 26R15)	2	23	200	200	Forage	7	0.929	0.215	0.11	1.1	0.054
	(6)	28	203	190		(22.8)	0.879	0.209	0.11	1.1	0.057
										1.1 (mean)	0.055 (mean)
	2	75	209	290	Hay	5	20.9	0.223	0.205	21	0.48
	(6)	83	206	290		(85.6)	24	0.105	0.161	24	0.32
							22 (mean)			23 (mean)	0.40 (mean)
					Straw	20	19	0.204	0.14	19	0.32
						(83.8)	19.5	0.19	0.116	20	0.34
							19 (mean)			19 (mean)	0.33 (mean)
RV055-10HA, Cheneyville, Louisiana USA, 2010 (Terral Brand LA821)	2	26	211	170	Forage	7	2.07	0.503	0.219	2.6	0.056
	(7)	28	204	170		(17.2)	1.58	0.53	0.177	2.1	0.049
										2.3 (mean)	0.053
	2	77	207	180	Hay	7	7.56 c 0.0154	0.227	0.052	7.8	0.20
	(7)	85	206	180		(79.9)	7.38	0.148	0.043	7.5	0.17
							7.5 (mean)			7.7 (mean)	0.19 (mean)
					Straw	21	7.86	0.128	0.046	8.0	0.33
						(74.8)	6.04	0.082	0.035	6.1	0.29
							7.0 (mean)			7.1 (mean)	0.31 (mean)
RV056-10HA, Stafford, Kansas USA, 2010 (Found Juniper)	2	26	204	190	Forage	7	2.52	0.217	0.102	2.7	0.041
	(7)	26	208	190		(22.3)	2.59	0.23	0.106	2.8	0.039
										2.8 (mean)	0.040 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
	2	69	209	190	Hay	7	13.6	0.362	0.377	14	0.21
	(6)	75	209	190		(81.7)	19.1	0.409	0.568	20	0.30
							16 (mean)			17 (mean)	0.25 (mean)
					Straw	21	12.2	0.285	0.335	12	0.52
						(85.2)	9.77	0.23	0.183	10	0.34
							11 (mean)			11 (mean)	0.43 (mean)
	1 (Seed)	0	128		Forage	171	0.064	0.081	< 0.01	0.15	0.02
						(21.0)	0.069	0.106	< 0.01	0.18	0.01
					Hay	238	0.113	0.524	< 0.01	0.64	0.06
						(81.4)	< 0.01	0.361	< 0.01	0.37	0.03
					Straw	252	0.118	0.133	< 0.01	0.25	0.06
						(84.7)	0.073	0.116	< 0.01	0.19	0.06
RV057-10HA, Gardner, Kansas USA, 2010 (Winter Hawk)	2	27	201	130	Forage	5	2.2	0.225	0.108	2.4	0.057
	(7)	29	201	130		(20.6)	2.61	0.222	0.111	2.8	0.060
	2	77	201	140	Hay	7	5.59	0.12	< 0.01	5.7	0.15
	(7)	83	206	150		(85.2)	5.77	0.262	< 0.01	6.0	0.14
							5.7 (mean)			5.9 (mean)	0.14 (mean)
					Straw	21 (85.3)	4.34	0.192	0.192	4.5	0.23
							3.15	0.147	0.147	3.3	0.20
							3.7 (mean)			3.9 (mean)	0.22 (mean)
RV058-10HA, Rockwood, Ontario Canada, 2010 (Glenn (Hard Red, Spring))	2	22	208	110	Forage	7	0.474	0.206	0.04	0.68	0.041
	(7)	24	204	100		(13.4)	0.523	0.244	0.058	0.77	0.044
										0.72 (mean)	0.042 (mean)
	2	83	197	110	Hay	7	18.3 c 0.043	0.335	0.011	19	0.43
	(7)	85	205	110		(79.8)	7.78	0.126	0.083	7.9	0.24
							13 (mean)			13 (mean)	0.34 (mean)
					Straw	21	4.01 c 0.011	0.188	0.023	4.2	0.14
						(65.6)	3.61	0.397	0.026	4.0	0.13
							3.8 (mean)			4.1 (mean)	0.14 (mean)
RV059-10HA, Campbell, Minnesota USA, 2010 (RB07)	2	15	205	190	Forage	7	0.186	0.090	0.028	0.28	0.036
	(7)	30	205	190		(15.8)	0.196	0.101	0.026	0.30	0.038
										0.29 (mean)	0.037 (mean)
	2	77	205	190	Hay	7	3.07	1.38	0.141	4.5	0.47
	(7)	83	205	190		(81.1)	2.13	0.511	0.071	2.6	0.28

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
							2.6 (mean)			3.5 (mean)	0.37 (mean)
					Straw	21	1.34 c 0.014	0.555	0.022	1.9	0.65
						(62.2)	0.711	0.498	0.013	1.2	0.56
							1.0 (mean)			1.6 (mean)	0.60 (mean)
RV060-10DA, Fisk, Missouri USA, 2010 (Beretta)	2	26	205	190	Forage	0	20.7	0.051	0.213	21	0.47
	(7)	27	205	190		(19.8)	22.1	< 0.05	0.183	22	0.34
						3	7.76	0.056	0.263	7.8	0.25
						(19.6)	7.41	0.087	0.293	7.5	0.18
						6	1.91	0.118	0.078	2.0	0.029
						(17.0)	2.21	0.237	0.149	2.4	0.14
										2.2 (mean)	
						13	0.317	0.212	0.018	0.53	0.093
						(17.5)	0.307	0.227	0.023	0.53	0.097
											0.095 (mean)
						20	0.067	0.269	0.011	0.34	0.045
						(16.5)	0.098	0.276	0.013	0.37	0.033
	2	77	206	190	Hay	0	7.85	0.756	0.212	8.6	0.18
	(6)	83	206	190		(52.9)	8.34	0.634	0.183	9.0	0.19
						3	6.28	1.08	0.185	7.4	0.42
						(73.2)	5.94	1.13	0.20	7.1	0.37
						7	8.28	1.7	0.337	10	0.76
						(86.1)	8.42	1.41	0.256	9.8	0.75
							8.4 (mean)			9.9 (mean)	
						14	6.13	2.12	0.182	8.3	2.17
						(89.5)	8.4	2.11	0.159	11	2.49
											2.3 (mean)
						21	5.7	2.27	0.108	8.0	2.15
						(83.8)	5.8	1.84	0.094	7.6	2.05
					Straw	10	4.55	0.797	0.123	5.3	1.52
						(52.4)	4.43	0.70	0.119	5.1	1.22
						15	3.38	0.805	0.059	4.2	1.50
						(61.4)	3.19	0.847	0.066	4.0	1.44
						21	7.19 c 0.016	1.38	0.042	8.6	2.49
						(81.9)	4.17	0.733	0.048	4.9	1.35
							5.7 (mean)			6.7 (mean)	1.9 (mean)
						28	2.69	0.89	0.025	3.6	0.43
						(87.3)	2.39	0.69	0.017	3.1	0.43
						35	2.58	0.666	0.026	3.2	0.50
						(89.4)	2.6	0.912	0.032	3.5	0.69
RV061-10HA, East Bernard, Texas	2	34	206	260	Forage	7	5.59	0.497	0.578	6.1	0.14
	(5)	41	205	270		(30.2)	6.28	0.367	0.67	6.6	0.17

Trial No., Location, Year (Variety)	Application				Sample	DALA (% DM)	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	Parent + DFA	6-CNA
USA, 2010 (Fannin)										6.4 (mean)	0.16 (mean)
	2	34	207	270	Hay	7	18.5	0.132	0.333	19	0.12
	(7)	41	203	270		(93.6)	16.5	0.145	0.251	17	0.10
							18 (mean)			18 (mean)	0.11 (mean)
					Straw	21	12.8	0.146	0.139	13	0.17
						(86.7)	12.6	0.102	0.145	13	0.16
							13 (mean)			13 (mean)	0.16 (mean)
RV062-10HA, Grand Island, Nebraska USA, 2010 (Traverse)	2	23	206	190	Forage	7	0.051	< 0.05	< 0.01	0.10	< 0.01
	(7)	23	206	190		(17.3)	0.087	< 0.05	< 0.01	0.14	< 0.01
										0.12 (mean)	< 0.01 (mean)
	2	71	206	190	Hay	7	6.38	1.44	0.44	7.8	0.68
	(7)	73	207	190		(75.22)	6.47	1.37	0.376	7.8	0.72
							6.4 (mean)			7.8 (mean)	0.70 (mean)
					Straw	22	1.43	0.845	0.036	2.3	0.75
						(68.1)	0.853	0.862	0.016	1.7	0.79
							1.1 (mean)			2.0 (mean)	0.77 (mean)
	1 (Seed)	0	113		Forage	45 (17.6)	0.14	< 0.05	0.014	0.19	0.02
							0.121	< 0.05	0.016	0.17	0.01
					Hay	74	0.086	0.212	< 0.01	0.30	0.02
						(75.1)	0.042	0.16	< 0.01	0.20	0.01
					Straw	103	0.026	0.125	< 0.01	0.15	0.04
						(66.3)	< 0.01	0.082	< 0.01	0.09	0.03
RV063-10HA, Carrington, North Dakota USA, 2010 (Faller)	2	13	207	95	Forage	7	0.147	< 0.05	< 0.01	0.20	0.011
	(6)	21	210	96		(12.7)	0.154	0.08	< 0.01	0.23	0.017
										0.22 (mean)	0.014 (mean)
	2	73	204	190	Hay	7	2.46	1.08	0.023	3.5	0.71
	(5)	77	205	190		(76.7)	2.61	1.02	0.115	3.6	0.67
							2.5 (mean)			3.6 (mean)	0.69 (mean)
					Straw	21	0.373	0.393	0.01	0.77	0.40
						(42.8)	0.508	0.633	0.011	1.1	0.49
							0.44 (mean)			0.95 (mean)	0.45 (mean)
RV064-10HA, Jamestown, North Dakota USA, 2010	2	14	207	190	Forage	7	0.449	0.136	0.072	0.59	0.020
	(6)	24	202	180		(17.1)	0.463	0.126	0.041	0.59	0.026
										0.59	0.023

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
(Oklee)										(mean)	(mean)
	2	77	205	190	Hay	6	6.33	0.616	0.152	6.9	0.53
	(6)	83	207	180		(89.1)	7.08	0.68	0.177	7.8	0.64
							6.7 (mean)			7.4 (mean)	0.59 (mean)
					Straw	20	2.22	0.665	0.018	2.9	0.63
						(71.1)	1.9	0.622	0.029	2.5	0.46
							2.1 (mean)			2.7 (mean)	0.55 (mean)
RV065-10HA, Oberon, North Dakota USA, 2010 (Faller)	2	13	203	93	Forage	7	0.251	< 0.05	0.012	0.30	0.029
	(6)	21	206	94		(12.7)	0.146	0.096	< 0.01	0.24	0.019
										0.27 (mean)	0.024 (mean)
	2	73	197	180	Hay	7	3.33 c 0.035	0.385	0.022	3.7	0.34
	(5)	77	206	190		(82.9)	3.69	0.379	0.036	4.1	0.55
							3.5 (mean)			3.9 (mean)	0.44 (mean)
					Straw	21	3.03	0.551	0.023	3.6	0.86
						(64.3)	2.0	0.363	0.014	2.4	0.55
							2.5 (mean)			3.0 (mean)	0.70 (mean)
RV066-10DA, Grand Island, Nebraska USA, 2010 (Overland HRW)	2	24	210	190	Forage	0	10.7	0.128	0.094	11	0.12
	(7)	25	207	190		(22.2)	15.3	< 0.05	0.165	15	0.05
						3	1.29	< 0.05	0.066	1.3	0.03
						(26.9)	1.37	< 0.05	0.06	1.4	0.02
						7	0.562	< 0.05	0.048	0.61	0.02
						(26.3)	0.578	< 0.05	0.05	0.63	0.02
										0.62 (mean)	0.019 (mean)
						14 (22.4)	0.099	< 0.05	< 0.01	0.15	0.01
							0.112	0.054	0.011	0.17	0.01
						19	0.019	< 0.05	< 0.01	0.069	0.01
						(24.9)	0.027	< 0.05	< 0.01	0.077	0.01
	2	71	205	190	Hay	0	19.9	0.236	0.252	20	0.13
	(7)	83	205	180		(78.3)	16.4	0.255	0.276	17	0.10
						3	12.7	0.142	0.209	13	0.15
						(74.1)	12.3	0.171	0.208	12	0.14
						7	7.25	0.437	0.209	7.7	0.27
						(75.1)	5.88	0.506	0.177	6.4	0.20
							6.6 (mean)			7.1 (mean)	0.23 (mean)
						14	0.07	< 0.05	< 0.01	0.12	< 0.01
						(83.1)	0.123	0.056	0.01	0.18	< 0.01
						20	2.56	0.287	0.04	2.8	0.20
						(79.8)	3.1	0.259	0.04	3.4	0.19
					Straw	9	0.111	< 0.05	< 0.01	0.16	0.013

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
						(55.9)	0.14	< 0.05	< 0.01	0.19	0.012
						14	0.098	< 0.05	< 0.01	0.15	0.018
						(74.3)	0.152	< 0.05	< 0.01	0.2	< 0.01
						20	3.89	0.221	0.04	4.1	0.33
						(86.9)	6.15	0.141	0.043	6.3	0.24
							5.0 (mean)			5.2 (mean)	
						27	3.55	0.188	0.046	3.7	0.38
						(82.7)	3.21	0.243	0.045	3.5	0.47
											0.43 (mean)
						35	2	0.105	0.017	2.1	0.22
						(88.1)	1.47	0.111	0.012	1.6	0.16
RV067-10HA, Taber, Alberta Canada, 2010 (Supurb)	2	23	205	170	Forage	7	0.453	< 0.05	0.053	0.50	0.025
	(7)	32	205	140		(18.2)	0.517	0.051	0.066	0.57	0.036
										0.54 (mean)	0.031 (mean)
	2	83	204	140	Hay	8	5.49	0.394	0.112	5.9	0.21
	(7)	87	197	130		(82.9)	5.52	0.288	0.146	5.8	0.20
							5.5 (mean)			5.8 (mean)	0.20 (mean)
					Straw	30 (51.9)	1.16	0.146	0.011	1.3	0.15
							1.16	0.153	0.012	1.3	0.17
							1.2 (mean)			1.3 (mean)	0.16 (mean)
RV068-10HA, Meadow, Texas USA, 2010 (Hatcher)	2	29	208	190	Forage	6	6.38	0.327	0.416	6.7	0.078
	(8)	29	210	190		(25.0)	4.77	0.306	0.358	5.1	0.070
										5.9 (mean)	0.074 (mean)
	2	69	208	190	Hay	7	10.4	0.239	0.238	11	0.15
	(6)	77	208	190		(95.5)	11.3	0.082	0.336	11	0.075
							11 (mean)			11 (mean)	0.11 (mean)
					Straw	21	8.48 c 0.019	< 0.05	0.391	8.5	0.16
						(94.8)	7.67	< 0.05	0.319	7.7	0.13
							8.1 (mean)			8.1 (mean)	0.15 (mean)
RV069-10HA, Hinton, Oklahoma USA, 2010 (Jagger)	2	22	203	200	Forage	7	5.1	0.254	0.396	5.4	0.045
	(6)	30	206	200		(23.2)	5.74	0.304	0.314	6	0.035
										5.7 (mean)	0.040 (mean)
	2	83	201	180	Hay	7	11.5	0.182	0.249	12	0.16
	(7)	83	207	180		(83.5)	12.5	0.21	0.289	13	0.16
							12 (mean)			12 (mean	0.16 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
							c0.01			)	
					Straw	21	4.65	< 0.05	0.059	4.7	0.22
						(74.2)	4.6	< 0.05	0.046	4.7	0.19
							4.6 (mean)			4.7 (mean)	0.21 (mean)
RV070-10HA, Levelland, Texas USA, 2010 (TAM 111)	2	29	207	190	Forage	7	8.84	0.295	0.399	9.1	0.021
	(7)	29	206	190		(23.5)	9.66	0.257	0.461	9.9	0.014
										9.5 (mean)	0.017 (mean)
	2	71	205	190	Hay	5	7.13	< 0.05	0.269	7.2	0.096
	(8)	83	206	190		(89.8)	7.3	< 0.05	0.288	7.4	0.064
							7.2 (mean)			7.3 (mean)	0.080 (mean)
					Straw	21	6.22	< 0.05	0.267	6.3	0.12
						(93.8)	5.96	< 0.05	0.205	6	0.10
							6.1 (mean)			6.1 (mean)	0.11 (mean)
	1 (Seed)	0	102		Forage	135	0.11	< 0.05	< 0.01	0.16	< 0.01
						(24.5)	0.047	< 0.05	< 0.01	0.1	< 0.01
					Hay	189	0.023	< 0.05	< 0.01	0.073	< 0.01
						(91.4)	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
					Straw	205	0.076	< 0.05	< 0.01	0.013	< 0.01
						(88.3)	0.017	< 0.05	< 0.01	0.07	0.02
RV071-10HA, Wall, Texas USA, 2010 (Coronado)	2	25	200	140	Forage	7	11.3	0.281	0.58	12	0.02
	(7)	25	204	140		(19.5)	14.7	0.296	0.684	15	0.02
										13 (mean)	0.021 (mean)
	2	85	205	180	Hay	7	8.76 c 0.016	< 0.05	0.116	8.8	0.040
	(7)	87	201	170		(94.0)	8.09	0.07	0.118	8.2	0.013
							8.4 (mean)			8.5 (mean)	0.027 (mean)
					Straw	21	7.57	< 0.05	0.175	7.6	0.073
						(88)	6.39	< 0.05	0.124	6.4	0.042
							7.0 (mean)			7.0 (mean)	0.058 (mean)
RV072-10HA, Uvalde, Texas USA, 2010 (TAM 203)	2	29	212	150	Forage	7	2.05	0.293	0.195	2.3	0.055
	(7)	29	199	150		(16.6)	2.26	0.366	0.231	2.6	0.057
										2.5 (mean)	0.056 (mean)
	2	77	201	190	Hay	7	9.56	0.131	0.22	9.7	0.13
	(7)	87	201	180		(85.4)	10.3	0.198	0.181	10.5	0.10
							9.9			10	0.11

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
							(mean)			(mean)	(mean)
					Straw	20 (83.6)	7.91	0.142	0.119	8.1	0.23
							7.53	0.123	0.096	7.7	0.25
							7.7 (mean)			7.9 (mean)	0.24 (mean)
RV073-10DA, Wall, Texas USA, 2010 (Doans)	2	28	202	180	Forage	0	29.6	0.052	0.369	30	0.11
	(4)	29	202	180		(26.3)	30.1	0.052	0.374	30	0.091
						3	16.6	0.073	0.366	17	0.063
						(27.3)	19.6	0.069	0.446	20	0.072
						7	15.3	0.083	0.542	15	0.88
						(29.0)	13.9	0.078	0.427	14	0.094
										15 (mean)	0.091 (mean)
						14	9.04	0.182	0.441	9.2	0.071
						(26.4)	8.72	0.186	0.396	8.9	0.038
						20	6.77	0.294	0.26	7.1	0.051
						(19.8)	6.05	0.284	0.292	6.3	0.040
	2	85	201	180	Hay	0	14.7	< 0.05	0.097	15	< 0.01
	(7)	85	201	180		(92.7)	13.8	< 0.05	0.112	14	0.15
						3	11.6	< 0.05	0.176	12	0.036
						(88.8)	9.72	< 0.05	0.137	9.8	0.063
						6	7.71	< 0.05	0.133	7.8	0.045
						(89.9)	8.54	< 0.05	0.151	8.6	0.034
							8.1 (mean)			8.2 (mean)	
						14	6.98	< 0.05	0.137	7	0.049
						(91.1)	7.3	< 0.05	0.14	7.4	0.042
						21	6.25	< 0.05	0.135	6.3	0.059
						(86.4)	6.81	< 0.05	0.14	6.9	0.056
											0.057 (mean)
					Straw	10	6.74	< 0.05	0.11	6.8	0.048
						(75.3)	6.84	< 0.05	0.152	6.9	0.057
						15	7.4	0.063	0.15	7.5	0.059
						(81.5)	0.592	< 0.05	0.015	0.64	< 0.01
						21	7.15	0.055	0.11	7.2	0.062
						(77.6)	6.59	0.09	0.127	6.7	0.062
							6.9 (mean)			6.9 (mean)	
						28	6.6	< 0.05	0.155	6.7	0.088
						(77.1)	6.1	< 0.05	0.141	6.2	0.093
											0.090 (mean)
						34	4.45	< 0.05	0.101	4.5	0.089
						(82.6)	5.62	< 0.05	0.149	5.7	0.073
RV074-10HA, Parkdale, Oregon USA, 2010 (Penawara)	2	23	204	190	Forage	7	0.52	< 0.05	0.034	0.57	0.022
	(6)	30	204	190		(14.7)	0.485	< 0.05	0.041	0.54	0.017
										0.55 (mean	0.019 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
	2	77	209	200	Hay	7	4.01	0.466	0.146	4.5	0.61
	(6)	77	205	190		(67.5)	3.2	0.491	0.219	3.7	0.34
							3.6 (mean)			4.1 (mean)	0.48 (mean)
					Straw	20	4.37	0.712	0.268	5.1	0.59
						(89.5)	3.59	0.608	0.145	4.2	0.47
							4.0 (mean)			4.6 (mean)	0.53 (mean)
RV075-10HA, Alvena, Saskatchewan Canada, 2010 (Infinity)	2	14	204	200	Forage	7	0.295	0.073	0.022	0.37	0.016
	(7)	21	202	200		(15.5)	0.359	0.084	0.029	0.44	0.015
										0.41 (mean)	0.016 (mean)
	2	51	204	200	Hay	7	17.2 c 0.013	1.44	1.02	19	0.42
	(5)	59	212	210		(86.2)	15.2	1.68	0.764	17	0.63
							16 (mean)			18 (mean)	0.52 (mean)
					Straw	21	1.93	1.49	0.052	3.4	0.39
						(46.6)	2.0	1.81	0.07	3.8	0.49
							2.0 (mean)			3.6 (mean)	0.44 (mean)
RV076-10HA, Minto, Manitoba Canada, 2010 (Infinity)	2	12	206	160	Forage	7	0.058	0.066	< 0.01	0.12	0.013
	(7)	22	209	160		(13.8)	0.047	0.063	< 0.01	0.11	0.017
										0.12 (mean)	0.015 (mean)
	2	83	205	160	Hay	7	5.91 c 0.039	0.435	0.12	6.3	0.26
	(7)	85	200	160		(86.6)	5.47	0.557	0.135	6	0.32
							5.7 (mean)			6.2 (mean)	0.29 (mean)
					Straw	21	2.03 c 0.014	0.254	0.026	2.3	0.64
						(81.3)	1.99	0.235	0.05	2.2	0.63
							2.0 (mean)			2.3 (mean)	0.64 (mean)
RV077-10HA, Boissevain, Manitoba Canada, 2010 (Glenn)	2	12	211	160	Forage	7	0.068	< 0.05	< 0.01	0.12	0.035
	(7)	12	212	170		(11.2)	0.021	< 0.05	< 0.01	0.071	0.018
										0.095 (mean)	0.027 (mean)
	2	75	208	160	Hay	7	4.16	1.24	0.139	5.4	0.21
	(6)	83	206	160		(87.4)	4.46	1.4	0.13	5.9	0.26
							4.3 (mean)			5.6 (mean)	0.23 (mean)
					Straw	21	0.216	0.346	< 0.01	0.56	0.30

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
						(35.6)	0.254	0.39	< 0.01	0.64	0.32
							0.24 (mean)			0.60 (mean)	0.31 (mean)
RV078-10HA, Wellwood, Manitoba Canada, 2010 (Glenn)	2	14	196	190	Forage	7	0.091	< 0.05	0.012	0.14	0.022
	(7)	21	200	200		(13.3)	0.105	< 0.05	< 0.01	0.16	0.024
										0.15 (mean)	0.023 (mean)
	2	71	199	190	Hay	5	7.77	1.59	0.22	9.4	1.26
	(5)	73	201	200		(75.8)	7.75	1.74	0.244	9.5	1.10
							7.8 (mean)			9.4 (mean)	1.2 (mean)
					Straw	21	0.667	0.785	0.014	1.5	0.28
						(49.2)	0.647	0.852	0.013	1.5	0.32
							0.66 (mean)			1.5 (mean)	0.30 (mean)
RV079-10HA, Wakaw, Saskatchewan Canada, 2010 (Harvest)	2	12	205	200	Forage	7	0.172	< 0.05	0.012	0.22	0.023
	(7)	21	202	200		(16.8)	0.155	0.053	0.02	0.21	0.024
										0.22 (mean)	0.023 (mean)
	2	57	206	200	Hay	7	10.9	1.53	0.296	12	0.52
	(5)	64	211	210		(84.2)	11	1.65	0.353	13	0.63
							11 (mean)			13 (mean)	0.58 (mean)
					Straw	21	0.56	0.934	0.024	1.5	0.24
						(38.3)	0.603	0.913	0.025	1.5	0.25
							0.58 (mean)			1.5 (mean)	0.24 (mean)
RV080-10HA, Waldheim, Saskatchewan Canada, 2010 (Infinity)	2	13	205	200	Forage	7	0.088	< 0.05	< 0.01	0.14	< 0.01
	(7)	21	204	200		(16.8)	0.111	< 0.05	< 0.01	0.16	0.011
										0.15 (mean)	0.011 (mean)
	2	65	206	200	Hay	7	7.46	0.9	0.373	8.4	0.22
	(7)	73	210	200		(86.1)	8.49	0.91	0.386	9.4	0.25
							8.0 (mean)			8.9 (mean)	0.24 (mean)
					Straw	20	0.896	0.477	0.02	1.4	0.20
						(38.7)	0.965	0.475	0.022	1.4	0.18
							0.93 (mean)			1.4 (mean)	0.19 (mean)
RV081-10HA, Fort Saskatchewan, Alberta Canada, 2010 (Superb)	2	13	203	99	Forage	7	0.164	0.057	0.018	0.22	0.019
	(7)	22	205	100		(17.3)	0.074	< 0.05	< 0.01	0.12	0.015
										0.17 (mean)	0.017 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
	2	75	201	99	Hay	6	6.06	0.446	0.188	6.5	0.18
	(7)	85	209	100		(89.0)	5.75	0.486	0.149	6.2	0.18
							5.9 (mean)			6.4 (mean)	0.18 (mean)
					Straw	21	3.34	0.271	0.073	3.6	0.41
						(41.2)	3.69	0.319	0.061	4.0	0.45
							3.5 (mean)			3.8 (mean)	0.43 (mean)
RV082-10DA, Josephburg, Alberta Canada, 2010 (Superb)	2	13	207	100	Forage	0	35.9	0.082	0.25	36	< 0.01
	(7)	22	204	100		(17.7)	41.8	0.056	0.286	42	0.019
						3	4.14	0.062	0.227	4.2	0.063
						(17.8)	3.74	0.078	0.214	3.8	0.054
						7	0.119	< 0.05	0.01	0.17	0.019
						(16.7)	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
										0.11 (mean)	0.015 (mean)
						13	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
						(14.6)	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
						21	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
						(11.9)	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
	2	83	213	100	Hay	0	12	0.293	0.084	12	0.18
	(7)	83	211	100		(80.7)	13.8	0.131	0.085	14	0.12
						3	11	0.437	0.062	11	0.19
						(81.1)	8.9	0.482	0.149	9.4	0.27
						7	7.30 c 0.011	0.64	0.06	7.9	0.70
						(83.7)	7.92	0.471	0.103	8.4	0.20
						13	6.85	0.482	0.076	7.3	0.17
						(82.3)	11.5	0.982	0.114	12	0.54
							9.2 (mean)			9.9 (mean)	
						21	8.69	0.841	0.081	9.5	0.66
						(77.4)	7.48	0.636	0.092	8.1	0.72
											0.69 (mean)
					Straw	10	3.84	0.355	0.057	4.2	0.20
						(34.5)	3.84	0.541	0.091	4.4	0.28
						14	2.99	0.206	0.028	3.2	0.27
						(33.2)	2.97	0.157	0.046	3.1	0.23
						21	4.9	0.732	0.087	5.6	0.65
						(43.4)	4.29	0.595	0.088	4.9	0.47
							4.6 (mean)			5.3 (mean)	0.56 (mean)
						28	4.87	0.664	0.106	5.5	0.68
						(50.4)	2.46	0.349	0.032	2.8	0.29
						35	2.7	0.332	0.028	3	0.26

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
						(60.3)	1.98	0.37	0.019	2.4	0.18

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 137 Residues in field corn forage and stover from foliar or seed treatment application of flupyradifurone to field corn in the USA and Canada (Fischer 2012, RARVY002)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
GAP, USA, Crop Group 15	2 (7)		205			Forage 7, Stover 21					
RV021-10HA, Alton, New York USA, 2010 (Hyland Seeds HL 2093)	2	79	206	280	Forage	7	1.95	0.112	< 0.05	2.1	< 0.05
	(5)	83	205	280		(38)	2.37	0.104	< 0.05	2.5	< 0.05
										2.3 (mean)	< 0.05 (mean)
	2	79	205	280	Stover	20	1.14	0.105	< 0.05	1.3	< 0.05
	(6)	83	204	280		(24)	1.28	0.12	< 0.05	1.4	< 0.05
							1.2 (mean)			1.3 (mean)	< 0.05 (mean)
RV022-10HA, Blackville, South Carolina USA, 2010 (DKC69-72)	2	75	205	140	Forage	7	1.57	0.161	< 0.05	1.7	< 0.05
	(6)	83	205	140		(39)	1.79	0.188	< 0.05	2.0	< 0.05
										1.9 (mean)	< 0.05 (mean)
	2	89	203	130	Stover	21	1.83	0.199	< 0.05	2.0	0.242
	(6)	89	205	130		(63)	1.94	0.201	< 0.05	2.1	0.134
							1.9 (mean)			2.1 (mean)	0.19 (mean)
RV023-10HA, Richland, Iowa USA, 2010 (09HYBK110HOER)	2	85	205	150	Forage	7	1.62	0.099	< 0.05	1.7	< 0.05
	(6)	85	205	150		(37)	1.48	0.102	< 0.05	1.6	< 0.05
										1.7 (mean)	< 0.05 (mean)
	2	87	207	160	Stover	21	1.34	0.13	< 0.05	1.5	< 0.05
	(7)	87	205	170		(84)	1.36	0.121	< 0.05	1.5	< 0.05
							1.4 (mean)			1.5 (mean)	< 0.05 (mean)
	1	0	119		Forage	98	< 0.05	0.058	< 0.05	0.11	< 0.05
	(Seed)					(28)	< 0.05	0.081	< 0.05	0.13	< 0.05
					Stover	133	< 0.05	0.056	< 0.05	0.11	< 0.05

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
							< 0.05	0.062	< 0.05	0.11	< 0.05
RV024-10HA, Gardner, Kansas USA, 2010 (09HYBK110HOER)	2	83	208	140	Forage	7	2.45	0.095	< 0.05	2.6	< 0.05
	(7)	83	204	150		(36)	2.33	0.101	< 0.05	2.4	< 0.05
										2.5 (mean)	< 0.05 (mean)
	2	85	204	150	Stover	21	1.21	0.121	< 0.05	1.3	0.063
	(5)	85	201	150		(52)	1.5	0.105	< 0.05	1.6	0.0802
							1.4 (mean)			1.5 (mean)	0.072 (mean)
	1	0	54		Forage	89	< 0.05	< 0.05	< 0.05	< 0.10	< 0.05
	(Seed)					(34)	< 0.05	< 0.05	< 0.05	< 0.10	< 0.05
					Stover	119	< 0.05	< 0.05	< 0.05	< 0.10	< 0.05
							< 0.05	0.052	< 0.05	0.1	< 0.05
RV033-10HA,	2	83	208	140	Forage	7	1.84	0.093	< 0.05	1.9	< 0.05
Gardner, Kansas	(7)	83	204	150		(40)	2.0	0.118	< 0.05	2.1	< 0.05
USA, 2010										2.0 (mean)	< 0.05 (mean)
(83R38-3000GT)	2	85	206	150	Stover	21	1.04	0.096	< 0.05	1.1	0.0615
	(5)	85	207	150		(49)	1.36	0.128	< 0.05	1.5	0.0764
							1.2 (mean)			1.3 (mean)	0.069 (mean)
RV025-10HA, Northwood, North Dakota USA, 2010 (Decalb DKC35-19)	2	79	208	190	Forage	6	1.55	0.074	< 0.05	1.6	< 0.05
	(7)	83	205	190		(31)	1.43	0.064	< 0.05	1.5	< 0.05
										1.6 (mean)	< 0.05 (mean)
	2	85	206	190	Stover	20	0.649	< 0.05	< 0.05	0.70	< 0.05
	(6)	85	206	190		(40)	1.16	< 0.05	< 0.05	1.2	< 0.05
							0.90 (mean)			0.95 (mean)	< 0.05 (mean)
RV026-10HA, Brant, Ontario Canada, 2010 (Decalb 3832 Non-BT)	2	75	205	300	Forage	7	2.15	0.075	< 0.05	2.2	< 0.05
	(5)	83	205	300		(34)	1.48	0.063	< 0.05	1.5	< 0.05
										1.9 (mean)	< 0.05 (mean)
	2	85	207	310	Stover	22	1.39	< 0.05	< 0.05	1.4	< 0.05
	(7)	85	209	310		(31)	1.81	< 0.05	< 0.05	1.9	< 0.05
							1.6 (mean)			1.7 (mean)	< 0.05 (mean)
RV029-10HA,	2	75	209	300	Forage	7	2.45	0.063	< 0.05	2.5	< 0.05
Brant, Ontario	(5)	83	205	300		(45)	2.15	0.057	< 0.05	2.2	< 0.05
Canada, 2010										2.4 (mean)	< 0.05 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
(Dekalb 4660)	2	87	205	310	Stover	22	1.27	< 0.05	< 0.05	1.3	< 0.05
	(7)	87	206	300		(30)	1.2	< 0.05	< 0.05	1.3	< 0.05
							1.2 (mean)			1.3 (mean)	< 0.05 (mean)
RV027-10HA, Stafford, Kansas USA, 2010 (A:09HYBK105 HOER)	2	73	207	190	Forage	6	2.18	0.101	< 0.05	2.3	< 0.05
	(7)	75	202	180		(28)	2.02	0.116	< 0.05	2.1	< 0.05
										2.2 (mean)	< 0.05 (mean)
	2	85	205	190	Stover	21	6.1	0.089	< 0.05	6.2	0.15
	(7)	87	199	190		(48)	5.7	0.089	< 0.05	5.8	0.15
							5.9 (mean)			5.9 (mean)	0.15 (mean)
	1	0	118		Forage	86	< 0.05	0.055	< 0.05	0.11	< 0.05
	(Seed)					(28)	< 0.05	0.056	< 0.05	0.11	< 0.05
					Stover	131	< 0.05	< 0.05	< 0.05	< 0.100	< 0.05
							< 0.05	< 0.05	< 0.05	< 0.100	< 0.05
RV035-10HA,	2	73	210	190	Forage	5	2.71	0.058	< 0.05	2.8	< 0.05
Stafford, Kansas	(5)	75	200	180		(28)	2.87	0.065	< 0.05	2.9	< 0.05
USA, 2010										2.9 (mean)	< 0.05 (mean)
(Pioneer 32B34)	2	85	207	190	Stover	21	3.31	0.204	< 0.05	3.5	0.116
	(7)	87	209	190		(52)	1.84	0.069	< 0.05	1.9	0.060
							2.6 (mean)			2.7 (mean)	0.088 (mean)
RV028-10HA, Clarence, Missouri USA, 2010 (MFA Trophy)	2	79	199	190	Forage	7	3.11	0.098	< 0.05	3.2	< 0.05
	(7)	85	210	190		(31)	2.97	0.059	< 0.05	3.0	< 0.05
										3.1 (mean)	< 0.05 (mean)
	2	85	202	180	Stover	21	4.96	0.106	< 0.05	5.1	0.203
	(7)	87	209	190		(43)	4.21	0.099	< 0.05	4.3	0.206
							4.6 (mean)			4.7 (mean)	0.20 (mean)
RV030-10HA, York, Nebraska USA, 2010 (Channel 207-03VT)	2	75	203	180	Forage	7	2.7	0.062	< 0.05	2.76	< 0.05
	(7)	83	210	180		(38)	2.41	0.069	< 0.05	2.48	< 0.05
										2.6 (mean)	< 0.05 (mean)
	2	87	204	190	Stover	21	3.4	< 0.05	< 0.05	3.5	0.0571
	(7)	87	204	180		(64)	2.56	0.064	< 0.05	2.6	0.0596
							3.0 (mean)			3.0 (mean)	0.058 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
RV031-10HA, Perry, Iowa USA, 2010 (P1162XR)	2	71	206	170	Forage	6	1.62	0.091	< 0.05	1.7	< 0.05
	(8)	79	206	180		(44)	3.31	0.134	< 0.05	3.4	< 0.05
										2.6 (mean)	< 0.05 (mean)
	2	79	201	190	Stover	21	2.65	0.055	< 0.05	2.7	0.0992
	(7)	87	205	190		(68)	1.69	< 0.05	< 0.05	1.7	< 0.05
							2.2 (mean)			2.2 (mean)	0.075 (mean)
RV032-10HA, Rockwood, Ontario Canada, 2010 (25T87)	2	83	205	120	Forage	7	4.0	0.06	< 0.05	4.1	< 0.05
	(6)	85	205	130		(39)	2.46	0.057	< 0.05	2.5	< 0.05
										3.3 (mean)	< 0.05 (mean)
	2	85	206	130	Stover	21	3.21	0.051	< 0.05	3.3	< 0.05
	(7)	87	207	140		(49)	3.23	0.061	< 0.05	3.3	< 0.05
							3.2 (mean)			3.3 (mean)	< 0.05 (mean)
RV034-10HA,	2	83	212	150	Forage	7	3.48	< 0.05	< 0.05	3.5	< 0.05
Breslau, Ontario	(6)	85	210	150		(45)	4.11	< 0.05	< 0.05	4.2	< 0.05
Canada, 2010										3.8 (mean)	< 0.05 (mean)
(20T16)	2	87	202	200	Stover	21	1.47	< 0.05	< 0.05	1.5	< 0.05
	(7)	87	204	200		(46)	1.47	< 0.05	< 0.05	1.5	< 0.05
							1.5 (mean)			1.5 (mean)	< 0.05 (mean)
RV036-10HA, Atlantic, Iowa USA, 2010 (Cgarst 85R08-3000GT)	2	85	206	290	Forage	0	4.36	< 0.05	< 0.05	4.4	< 0.05
	(7)	85	208	300		(41)	5.14	< 0.05	< 0.05	5.2	< 0.05
						3	2.24	< 0.05	< 0.05	2.3	< 0.05
						(38)	1.71	< 0.05	< 0.05	1.8	< 0.05
						7	0.777	< 0.05	< 0.05	0.83	< 0.05
						(38)	0.594	< 0.05	< 0.05	0.64	< 0.05
						14 (14)	0.673	< 0.05	< 0.05	0.72	< 0.05
							0.805	< 0.05	< 0.05	0.86	< 0.05
										0.79 (mean)	< 0.05 (mean)
						22 (22)	0.713	< 0.05	< 0.05	0.76	< 0.05
							0.916	< 0.05	< 0.05	0.97	< 0.05
	2	85	211	300	Stover	10	1.37	< 0.05	< 0.05	1.4	< 0.05
	(7)	85	210	300		(29)	1.42	< 0.05	< 0.05	1.5	< 0.05
						14	1.25	< 0.05	< 0.05	1.3	< 0.05
						(30)	1.24	< 0.05	< 0.05	1.3	< 0.05
						22	1.58	0.064	< 0.05	1.6	< 0.05
						(42)	1.2	< 0.05	< 0.05	1.3	< 0.05
						28	1.51	< 0.05	< 0.05	1.6	< 0.05
						(41)	1.92	< 0.05	< 0.05	2.0	< 0.05

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
							1.7 (mean)			1.8 (mean)	< 0.05 (mean)
						34	1.28	0.051	< 0.05	1.3	< 0.05
						(50)	1.67	0.068	< 0.05	1.7	< 0.05
RV037-10DA,	2	83	201	290	Forage	0	3.64	< 0.05	< 0.05	3.7	< 0.05
Branchton, Ontario	(6)	83	224	320		(38)	4.23	< 0.05	< 0.05	4.3	< 0.05
Canada, 2010						3	3.63	< 0.05	< 0.05	3.7	< 0.05
(Maizex)						(40)	3.18	< 0.05	< 0.05	3.2	< 0.05
						7	1.61	< 0.05	< 0.05	1.7	< 0.05
						(46)	1.87	< 0.05	< 0.05	1.9	< 0.05
											< 0.05 (mean)
						14	1.67	< 0.05	< 0.05	1.7	< 0.05
						(51)	2.02	< 0.05	< 0.05	2.1	< 0.05
										1.9 (mean)	
						20	1.31	< 0.05	< 0.05	1.4	< 0.05
						(53)	1.53	< 0.05	< 0.05	1.6	< 0.05
	2	85	203	280	Stover	10	3.84	< 0.05	< 0.05	3.9	< 0.05
	(7)	85	199	300		(32)	3.79	< 0.05	< 0.05	3.8	< 0.05
						14	2.46	0.057	< 0.05	2.5	< 0.05
						(46)	1.7	< 0.05	< 0.05	1.8	< 0.05
						19	1.74	< 0.05	< 0.05	1.8	< 0.05
						(28)	1.68	< 0.05	< 0.05	1.7	< 0.05
							1.7 (mean)			1.8 (mean)	< 0.05 (mean)
						27	0.949	< 0.05	< 0.05	1	< 0.05
						(36)	1.34	0.053	< 0.05	1.4	< 0.05
						33	1.4	0.054	< 0.05	1.5	< 0.05
						(37)	1.34	0.069	< 0.05	1.4	< 0.05
RV038-10DA, Springfield, Nebraska USA, 2010 (N38B4)	2	83	203	220	Forage	0	2.08	< 0.05	< 0.05	2.1	< 0.05
	(6)	85	206	190		(29)	1.69	< 0.05	< 0.05	1.7	< 0.05
						3	1.73	0.063	< 0.05	1.8	< 0.05
						(33)	2.2	0.09	< 0.05	2.3	< 0.05
						7	1.55	0.106	< 0.05	1.7	< 0.05
						(35)	3.78	0.156	< 0.05	3.9	< 0.05
										2.8 (mean)	
						14	0.775	0.11	< 0.05	0.89	< 0.05
						(42)	0.973	0.143	< 0.05	1.1	< 0.05
						19	0.732	0.132	< 0.05	0.86	0.053
						(48)	0.92	0.18	< 0.05	1.1	< 0.05
											0.052 (mean)
	2	87	204	180	Stover	10	3.92	0.069	< 0.05	4	< 0.05
	(6)	87	202	170		(47)	2.23	0.062	< 0.05	2.3	< 0.05
						13	2.29	0.085	< 0.05	2.4	0.060
						(47)	3.24	0.102	< 0.05	3.3	0.062
						19	2.73	0.112	< 0.05	2.8	0.063

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
						(40)	1.54	0.081	< 0.05	1.6	< 0.05
							2.1 (mean)			2.2 (mean)	0.056 (mean)
						26	1.14	0.077	< 0.05	1.2	< 0.05
						(41)	1.47	0.091	< 0.05	1.6	< 0.05
						33	0.845	0.109	< 0.05	0.95	< 0.05
						(55)	0.693	0.085	< 0.05	0.78	< 0.05
RV039-10DA, Campbell, Minnesota USA, 2010 (Cekalb 38-89)	2	83	206	190	Forage	0	2.24	< 0.05	< 0.05	2.3	< 0.05
	(7)	85	205	190		(32)	2.59	< 0.05	< 0.05	2.6	< 0.05
						3	1.63	< 0.05	< 0.05	1.7	< 0.05
						(33)	1.37	< 0.05	< 0.05	1.4	< 0.05
						7	1.83	0.074	< 0.05	1.9	< 0.05
						(42)	1.7	0.064	< 0.05	1.8	< 0.05
										1.8 (mean)	< 0.05 (mean)
						14	0.741	0.076	< 0.05	0.82	< 0.05
						(44)	0.588	0.072	< 0.05	0.66	< 0.05
						21	0.311	0.088	< 0.05	0.4	< 0.05
						(48)	0.33	0.078	< 0.05	0.41	< 0.05
	2	85	205	190	Stover	10	1.3	< 0.05	< 0.05	1.4	< 0.05
	(7)	85	205	190		(40)	1.37	< 0.05	< 0.05	1.4	< 0.05
						15	1.62	< 0.05	< 0.05	1.7	< 0.05
						(44)	1.36	< 0.05	< 0.05	1.4	< 0.05
						21	0.943	< 0.05	< 0.05	0.99	< 0.05
						(49)	1.18	< 0.05	< 0.05	1.2	< 0.05
											< 0.05 (mean)
						28	1.13	< 0.05	< 0.05	1.2	< 0.05
						(49)	0.997	< 0.05	< 0.05	1.1	< 0.05
						35	1.21	< 0.05	< 0.05	1.3	< 0.05
						(78)	1.62	< 0.05	< 0.05	1.7	< 0.05
							1.4 (mean)			1.5 (mean)	
RV040-10HA, Raymondville, Texas USA, 2010 (Dyna-Gro; H6284162)	2	75	207	94	Forage	7	1.63	0.121	< 0.05	1.8	< 0.05
	(6)	79	208	95		(26)	1.78	0.12	< 0.05	1.9	< 0.05
										1.8 (mean)	< 0.05 (mean)
	2	87	211	96	Stover	20	3.14	0.182	0.056	3.3	0.23
	(7)	87	212	96		(50)	3.05	0.194	0.064	3.2	0.25
							3.1 (mean)			3.3 (mean)	0.24 (mean)

LOQ is 0.05 mg/kg for parent flupyradifurone and the metabolites DFEAF, DFA and 6-CNA (parent equivs.) in forage and stover

Table 138 Residues in sweet corn forage and stover from foliar or seed treatment application of flupyradifurone to sweet corn in the USA and Canada (Fischer 2012b, RARVY002)

Trial No.,	Application	Sample	DALA	Residues as parent (mg/kg)

Location, Year (Variety)	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	Parent + DFA	6-CNA
GAP, USA, Crop Group 15	2 (7)		205			Forage 7, Stover 21					
RV041-10HA, Germansville, Pennsylvania, USA, 2010 (Extra Tender)	2	71	208	260	Forage	6	10.2	0.165	0.16	10.4	0.071
	(6)	73	208	260		(16)	8.18	0.163	0.147	8.3	0.074
										9.4 (mean)	0.073 (mean)
	2	85	206	310	Stover	20	7.1	0.288	0.073	7.4	0.266
	(6)	87	210	310		(30)	9.22	0.357	0.109	9.6	0.385
							8.2 (mean)			8.5 (mean)	0.33 (mean)
	1	0	115		Forage	72	< 0.05	< 0.05	< 0.05	< 0.10	< 0.05
	(Seed)					(16)	< 0.05	< 0.05	< 0.05	< 0.10	< 0.05
					Stover	110	< 0.05	0.165	< 0.05	0.22	< 0.05
						(33)	< 0.05	0.169	< 0.05	0.22	< 0.05
RV042-10DA, North Rose, New York, USA, 2010 (Serendipity)	2	71	207	330	Forag	0	3.68	0.068	< 0.05	3.8	< 0.05
	(7)	75	207	330	e	(17)	5.41	0.065	< 0.05	5.5	< 0.05
						3	2.24	0.086	< 0.05	2.3	< 0.05
						(17)	1.95	0.089	< 0.05	2	< 0.05
						7	1.58	0.14	< 0.05	1.7	< 0.05
						(19)	1.14	0.095	< 0.05	1.2	< 0.05
										1.5 (mean)	< 0.05 (mean)
						14	1.47	0.133	< 0.05	1.6	< 0.05
						(21)	0.547	0.081	< 0.05	0.63	< 0.05
						21	0.607	0.148	< 0.05	0.76	< 0.05
						(22)	0.637	0.145	< 0.05	0.78	< 0.05
	2	71	204	330	Stover	10	2.42	0.199	< 0.05	2.6	0.138
	(7)	75	204	330		(21)	3.15	0.244	< 0.05	3.4	0.186
						15	1.51	0.169	< 0.05	1.7	0.106
						(21)	2.14	0.208	< 0.05	2.4	0.149
						21	0.601	0.141	< 0.05	0.74	< 0.05
						(23)	0.454	0.148	< 0.05	0.60	< 0.05
							0.53 (mean)			0.67 (mean)	< 0.05 (mean)
						28	0.50	0.171	< 0.05	0.67	< 0.05
						(27)	0.426	0.157	< 0.05	0.58	< 0.05
						35	0.261	0.12	< 0.05	0.38	< 0.05
						(28)	0.265	0.129	< 0.05	0.39	< 0.05
RV043-10HA, Sycamore, Georgia, USA, 2010 (Bi-Color)	2	73	208	190	Forage	7	2.36	0.117	< 0.05	2.5	< 0.05
	(7)	75	207	190		(22)	3.87	0.156	< 0.05	4.0	< 0.05
										3.3 (mean)	< 0.05 (mean)
	2	85	201	200	Stover	21	4.69	0.115	< 0.05	4.8	< 0.05
	(7)	85	210	240		(43)	5.43	0.181	< 0.05	5.6	0.0715
							5.1 (mean)			5.2 (mean)	0.061 (mean)
RV044-10HA,	2 (7)	65	201	240	Forage	7 (21)	0.796	0.117	< 0.05	0.91	< 0.05
High Springs, Florida		73	204	240			0.994	0.123	< 0.05	1.1	< 0.05
USA, 2010										1.0 (mean)	< 0.05 (mean)
(Obsession)	2 (7)	87	208	240	Stover	20 (36)	3.86	0.248	0.062	4.1	0.383
		89	201	230			3.29	0.213	< 0.05	3.5	0.338
							3.6 (mean)			3.8 (mean)	0.36 (mean)
RV045-10HA,	2 (5)	71	207	180	Forage	7 (15)	< 0.05	< 0.05	< 0.05	< 0.100	< 0.05
Richland, Iowa,		71	205	160			3.26	0.114	< 0.05	3.4	< 0.05
USA, 2010										1.7	< 0.05

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	Parent + DFA	6-CNA
										(mean)	(mean)
(Augusta)	2 (7)	75	207	160	Stover	20 (46)	1.54	0.267	< 0.05	1.8	0.101
		83	208	150			1.37	0.23	< 0.05	1.6	0.0742
							1.5 (mean)			1.7 (mean)	0.087 (mean)
	1 (Seed)	0	93		Forage	76 (17)	4.21†	0.138	0.075	4.4	< 0.05
							0.097	< 0.05	< 0.05	0.15	< 0.05
					Stover	106 (38)	< 0.05	0.103	< 0.00	0.15	< 0.05
							< 0.05	0.1	< 0.05	0.15	< 0.05
RV046-10HA,	2 (7)	67	206	310	Forage	7 (17)	1.84	0.128	< 0.05	2.0	< 0.05
Stewardson, Illinois,		71	201	320			1.8	0.141	< 0.05	1.9	0.052
USA, 2010										2.0 (mean)	0.051 (mean)
(XTRA-tender 274A)	2 (7)	75	210	330	Stover	19 (60)	3.59	0.192	< 0.05	3.8	0.119
		85	209	330			3.21	0.216	< 0.05	3.4	0.155
							3.4 (mean)			3.6 (mean)	0.14 (mean)
RV047-10HA,	2 (7)	79	201	110	Forage	7 (19)	1.94	0.082	< 0.05	2.0	< 0.05
Rockwood, Ontario,		79	205	110			1.97	0.089	< 0.05	2.1	< 0.05
Canada, 2010										2.0 (mean)	< 0.05 (mean)
(Brocade TSW)	2 (7)	83	206	130	Stover	20 (40)	1.05	< 0.05	< 0.05	1.1	< 0.05
		85	207	130			1.23	< 0.05	< 0.05	1.3	< 0.05
							1.1 (mean)			1.2 (mean)	< 0.05 (mean)
RV048-10HA,	2 (6)	63	207	190	Forage	7 (16)	1.28	0.081	< 0.05	1.4	< 0.05
Osceola, Nebraska,		69	206	190			1.49	0.082	< 0.05	1.6	< 0.05
USA, 2010										1.5 (mean)	< 0.05 (mean)
(Augusta)	2 (7)	85	202	180	Stover	21 (57)	3.14	0.249	< 0.05	3.4	0.134
		85	209	190			3.98	0.328	< 0.05	4.3	0.241
							3.6 (mean)			3.8 (mean)	0.19 (mean)
	1 (Seed)	0	85		Forage	73 (15)	0.112	0.112	< 0.05	0.22	< 0.05
							0.072	0.068	< 0.05	0.14	< 0.05
					Stover	118 (54)	0.07	0.237	< 0.05	0.31	< 0.05
							0.093	0.292	< 0.05	0.39	< 0.05
RV049-10DA,	2	63	207	190	Forage	0	3.32	< 0.05	< 0.05	3.4	< 0.05
York, Nebraska,	(7)	71	205	190		(13)	3.74	< 0.05	< 0.05	3.8	< 0.05
USA, 2010						3	3.51	0.107	< 0.05	3.6	< 0.05
(Xtra-Tender 278A)						(16)	3.39	0.1	< 0.05	3.5	< 0.05
						7	2.32	0.12	< 0.05	2.4	< 0.05
						(18)	2.48	0.112	< 0.05	2.6	< 0.05
										2.5 (mean)	< 0.05 (mean)
						14	1.29	0.182	< 0.05	1.5	< 0.05
						(21)	1.19	0.179	< 0.05	1.4	< 0.05
						21	0.958	0.203	< 0.05	1.2	< 0.05
						(23)	1.04	0.221	< 0.05	1.3	< 0.05
	2	85	210	190	Stover	10	3.09	0.124	< 0.05	3.2	0.0666
	(7)	87	206	190		(52)	2.49	0.157	< 0.05	2.7	0.0676
						15	0.853	0.114	< 0.05	0.97	< 0.05
						(54)	1.16	0.109	< 0.05	1.3	< 0.05
						21	1.54	0.15	< 0.05	1.7	0.0545
						(64)	0.825	0.129	< 0.05	0.95	< 0.05
							1.2 (mean)			1.3 (mean)	0.052 (mean)
						28	0.599	0.1	< 0.05	0.7	< 0.05

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	Parent + DFA	6-CNA
						(48)	0.682	0.105	< 0.05	0.79	< 0.05
						35	0.827	0.123	< 0.05	0.95	< 0.05
						(66)	0.852	0.112	< 0.05	0.96	< 0.05
RV050-10HA, Outlook, Saskatchewan, Canada, 2010 (Jackpot)	2	71	205	200	Forage	7	0.646	< 0.05	< 0.05	0.70	< 0.05
	(7)	71	207	200		(15)	1.0	< 0.05	< 0.05	1.1	< 0.05
										0.87 (mean)	< 0.05 (mean)
	2	71	205	200	Stover	20	1.34	< 0.05	< 0.05	1.4	< 0.05
	(7)	71	208	200		(36)	1.5	< 0.05	< 0.05	1.6	< 0.05
							1.4 (mean)			1.5 (mean)	< 0.05 (mean)
RV051-10HA, Sanger, California, USA, 2010 (Golden Queen)	2	71	206	360	Forage	5	2.2	< 0.05	< 0.05	2.3	< 0.05
	(6)	75	209	270		(27)	3.47	< 0.05	< 0.05	3.5	< 0.05
										2.9 (mean)	< 0.05 (mean)
	2	85	205	380	Stover	21	1.36	< 0.05	< 0.05	1.4	< 0.05
	(7)	85	206	350		(31)	2.27	< 0.05	< 0.05	2.3	0.0714
							1.8 (mean)			1.9 (mean)	0.061 (mean)
RV052-10HA, Jerome, Idaho, Canada, 2010 (Jackpot)	2	71	208	190	Forage	7	1.69	0.056	< 0.05	1.7	< 0.05
	(7)	75	207	180		(18)	0.948	< 0.05	< 0.05	1.0	< 0.05
										1.4 (mean)	< 0.05 (mean)
	2	83	206	200	Stover	21	4.43	0.147	0.052	4.6	0.0596
	(7)	85	202	200		(37)	2.47	0.134	< 0.05	2.6	< 0.05
							3.5 (mean)			3.6 (mean)	0.055 (mean)
RV053-10HA, Portland, Oregon, USA, 2010 (Serendipity)	2	73	211	200	Forage	7	3.03	< 0.05	< 0.05	3.1	< 0.05
	(7)	75	208	190		(16)	4.84	0.071	< 0.05	4.9	< 0.05
										4.0 (mean)	< 0.05 (mean)
	2	73	205	200	Stover	21	3.18	0.233	< 0.05	3.4	0.0531
	(7)	75	205	180		(31)	3.14	0.212	< 0.05	3.4	< 0.05
							3.2 (mean)			3.4 (mean)	0.052 (mean)

LOQ is 0.05 mg/kg for parent flupyradifurone and the metabolites DFEAF, DFA and 6-CNA (parent equivs.) in forage and stover

Table 139 Residues in sorghum forage and stover from foliar or seed treatment application of flupyradifurone to sorghum in the USA (Krolski and Dallstream 2012, RARVY004)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
GAP, USA, Crop Group 15	2 (7)		205			Forage 7,					
						Stover 21					
RV083-10HA, Proctor, Arkansas USA, 2010 (Pioneer 85Y40)	2	55	206	188	Forage	7	3.7	0.14	0.059	3.8	0.026
	(7)	65	205	188		(28)	2.8	0.12	0.041	2.9	0.019
										3.4 (mean)	0.022 (mean)
	2	85	208	96	Stover	21	1.2	0.11	0.011	1.3	< 0.01
	(7)	85	206	95			0.77	0.085	0.011	0.86	< 0.01
							0.97			1.1	< 0.01

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume		(%DM)	Parent	DFA	DFEAF	Parent +	6-CNA
				(L/ha)						DFA	
							(mean)			(mean)	(mean)
RV084-10HA, Gardner, Kansas USA, 2010 (B-7B47)	2	80	209	146	Forage	7	3.4	0.14	0.072	3.5	0.037
	(7)	81	208	148		(34)	4.7	0.16	0.072	4.9	0.047
										4.2 (mean)	0.042 (mean)
	2	85	207	149	Stover	21	1.3	0.099	< 0.01	1.4	0.021
	(5)	85	208	151			1.2	0.11	< 0.01	1.3	0.021
							1.2 (mean)			1.3 (mean)	0.021 (mean)
	1	00	26		Forage	86	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
	(Seed)					(36)	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
					Stover	116	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
							< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
RV085-10HA, Dudley, Missouri USA, 2010 (DKS54-00)	2	75	208	190	Forage	7	2.4	0.081	0.069	2.5	0.030
	(7)	85	206	188		(43)	3.0	0.069	0.069	3.1	0.029
										2.8 (mean)	0.029 (mean)
	2	75	205	188	Stover	21	1.2	0.05	< 0.01	1.2	0.026
	(7)	85	202	184			1.4	0.071	< 0.01	1.5	0.040
							1.3 (mean)			1.4 (mean)	0.033 (mean)
RV086-10DA, Springfield, Nebraska USA, 2010 (NC+371)	2	79	206	185	Forage	0	3.9	< 0.05	0.014	3.9	< 0.01
	(5)	85	206	157		(25)	4.1	< 0.05	0.017	4.1	0.011
						3	2.2	0.052	0.022	2.3	0.016
						(28)	1.8	< 0.05	0.018	1.9	0.013
						6	2.1	0.095	0.026	2.2	0.016
						(34)	2.3	0.10	0.026	2.4	0.027
										2.3 (mean)	0.021 (mean)
						13	1.2	0.15	0.012	1.4	0.016
						(40)	1.5	0.12	0.018	1.6	0.022
						19	0.94	0.13	0.012	1.1	< 0.01
						(38)	1.1	0.14	0.016	1.3	0.016
	2	87	204	184	Stover	10	2.2	0.11	0.015	2.3	< 0.01
	(6)	87	203	174			1.8	0.10	< 0.01	1.9	0.015
						13	2.2	0.13	0.024	2.4	0.018
							2.4	0.10	< 0.01	2.5	0.028
						19	2.8	0.20	0.020	3.0	0.049
							2.1	0.14	0.013	2.2	0.035
							2.4 (mean)			2.6 (mean)	0.042 (mean)
						26	1.2	0.12	< 0.01	1.4	0.019
							1.5	0.16	< 0.01	1.6	< 0.01
						33	1.0	0.19	< 0.01	1.2	< 0.01
							0.77	0.18	< 0.01	0.95	< 0.01
RV087-10HA, Uvalde,	2	65	204	186	Forage	6	2.7	0.051	0.067	2.7	< 0.01
	(7)	85	203	152		(24)	2.3	0.056	0.054	2.3	< 0.01

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
Texas USA, 2010 (Asgrow A571)										2.5 (mean)	< 0.01 (mean)
	2	85	204	186	Stover	21	2.6	0.26	0.029	2.8	0.058
	(7)	85	205	160			2.8	0.23	0.031	3.0	0.076
							2.7 (mean)			2.9 (mean)	0.067 (mean)
	1 (Seed)	00	14		Forage	86	0.013	< 0.05	< 0.01	0.063	< 0.01
						(24)	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
					Stover	119	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
							< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
RV088-10HA, Raymondville, Texas USA, 2010 (Dekalb; DKS 707)	2	75	216	99	Forage	7	2.3	0.11	0.096	2.4	0.030
	(6)	80	212	96		(35)	2.1	0.12	0.098	2.2	0.027
										2.3 (mean)	0.028 (mean)
	2	85	211	96	Stover	20	2.9	0.37	0.019	3.2	0.12
	(7)	85	209	95			1.9	0.37	0.012	2.3	0.093
							2.4 (mean)			2.7 (mean)	0.11 (mean)
RV089-10HA, Grand Island, Nebraska USA, 2010 (7B47)	2	85	205	188	Forage	7	3.9	0.075	0.085	4.0	0.024
	(7)	85	206	188		(40)	4.6	0.072	0.10	4.6	0.031
										4.3 (mean)	0.028 (mean)
	2	85	206	188	Stover	21	1.7	< 0.05	0.020	1.7	0.024
	(7)	85	206	191			1.8	0.054	0.027	1.8	0.027
							1.7 (mean)			1.8 (mean)	0.025 (mean)
RV090-10HA, Levelland, Texas USA, 2010 (F-270E)	2	75	209	188	Forage	7	3.2	< 0.05	0.034	3.2	< 0.01
	(7)	85	210	190		(43)	2.4	< 0.05	0.039	2.4	< 0.01
										2.8 (mean)	< 0.01 (mean)
	2	85	211	190	Stover	21	1.3	0.079	0.015	1.4	< 0.01
	(6)	85	204	184			3.3	0.12	0.039	3.4	< 0.01
							2.3 (mean)			2.4 (mean)	< 0.01 (mean)
	1	00	20		Forage	104	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
	(Seed)					(44)	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
					Stover	124	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
							< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
RV091-10HA, Wall, Texas USA, 2010 (Garst: 5515)	2	75	207	177	Forage	6	2.7	0.10	0.062	2.8	0.023
	(7)	81	208	187		(33)	3.5	0.11	0.084	3.6	0.034
										3.2 (mean)	0.029 (mean)
	2	85	203	181	Stover	21	4.2	0.22	0.074	4.4	0.027

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume		(%DM)	Parent	DFA	DFEAF	Parent +	6-CNA
				(L/ha)						DFA	
	(7)	87	203	179			5.8	0.29	0.084	6.1	0.034
							5.0 (mean)			5.3 (mean)	0.031 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Thirteen supervised trials were carried out on alfalfa (Table 140) in the USA during the 2010 growing season to determine residues in forage and hay after two foliar applications of BYI 02960 200 SL (Dacus and Lenz 2012, RARVY017). All applications were made using ground-based equipment and included a non-ionic surfactant adjuvant at a rate of 0.25% v/v.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in alfalfa forage and hay were determined using LC/MS/MS Method 01304. Acceptable concurrent recovery data (mean recoveries, RSDs) were obtained for all analytes.

Table 140 Residues in alfalfa forage and hay from the foliar application of flupyradifurone to alfalfa in the USA (Dacus and Lenz 2012, RARVY017)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
GAP, USA, Alfalfa	2 (10)		205			Forage and Hay 7					
RV214-10HA, Germansville, Pennsylvania USA, 2010 (Ameristand 403T)	2	47	211	288	Forage	6	2.18	1.48	0.524	3.7	0.423
	(9)	55	205	281		(22)	3.32	1.91	0.585	5.2	0.455
										4.4 (mean)	0.44 (mean)
						29	< 0.01	0.434	< 0.01	0.44	< 0.01
						(30)	< 0.01	0.494	< 0.01	0.50	0.011
						55	< 0.01	0.198	< 0.01	0.21	< 0.01
						(15)	< 0.01	0.206	< 0.01	0.22	< 0.01
					Hay	6	6.88	4.40	1.21	11	1.18
						(68)	5.40	4.92	1.28	10	1.22
							6.1 (mean)			11 (mean)	1.2 (mean)
						29	0.021	1.68	0.014	1.7	0.044
						(77)	0.01	1.23	0.01	1.2	0.037
						55	< 0.01	0.880 *c0.065*	0.013	0.89	0.025
						(72)	< 0.01	1.07	0.014	1.1	0.027
RV215-10HA, Athens, Georgia USA, 2010 (Certified 505)	2	51	199	259	Forage	7	1.15	1.83	0.447	3.0	0.459
	(9)	61	203	290		(24)	1.11	1.75	0.365	2.9	0.378
										2.9 (mean)	0.42 (mean)
						41	< 0.01	0.452	< 0.01	0.46	< 0.01
						(28)	< 0.01	0.467	< 0.01	0.48	< 0.01
						81	< 0.01	0.121	< 0.01	0.13	< 0.01
						(21)	< 0.01	0.145	< 0.01	0.16	< 0.01
					Hay	7	2.53	5.46	0.796	8.0	4.10

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
								c0.067			
						(90)	2.82	5.54	0.701	8.4	3.52
							2.7 (mean)			8.2 (mean)	3.8 (mean)
						41	0.016	1.01 c0.092	< 0.01	1.0	0.034
						(74)	0.013	1.00	< 0.01	1.0	0.021
						81	0.067 c0.029	0.379	< 0.01	0.45	0.026
						(63)	0.052	0.429	< 0.01	0.48	0.025
RV216-10HA, Atlantic, Iowa USA, 2010 (Triple A Gold Blend)	2	55	206	297	Forage	7	0.427	0.973	0.156	1.4	0.239
	(9)	61	214	318		(18)	0.457	1.00	0.182	1.5	0.298
										1.4 (mean)	0.27 (mean)
						62	< 0.01	0.182	< 0.01	0.19	< 0.01
						(25)	0.019	0.66	< 0.01	0.68	0.018
						131	< 0.01	0.093	< 0.01	0.10	< 0.01
						(25)	< 0.01	0.080	< 0.01	0.090	< 0.01
					Hay	7	2.68 c0.011	5.02 c0.081	0.753	7.7	1.57
						(79)	2.41	4.70	0.871	7.1	1.41
							2.5 (mean)			7.4 (mean)	1.49 (mean)
						62	0.030 c0.037	0.165 c0.096	< 0.01	0.20	< 0.01
						(63)	0.049	0.270	< 0.01	0.32	< 0.01
						131 (69)	0.031 *c0.016*	0.270 c0.178	< 0.01	0.30	< 0.01
							0.021	0.362	< 0.01	0.38	< 0.01
RV217-10HA, Northwood, North Dakota USA, 2010 (-)	2	51	204	187	Forage	7	3.00	1.85	0.847	4.8	0.376
	(7)	59	205	187		(23)	3.16	2.03	0.677	5.2	0.477
										5.0 (mean)	0.43 (mean)
						41	< 0.01 *c 0.018*	0.171	< 0.01	0.18	< 0.01
						(22)	< 0.01	0.192	< 0.01	0.20	< 0.01
						82	< 0.01	0.208 c0.063	< 0.01	0.22	< 0.01
						(37)	< 0.01	0.255	< 0.01	0.27	0.012
					Hay	7	6.67	5.57	1.02	12	3.74
						(73)	8.18	6.75	1.22	15	5.20
							7.4 (mean)			14 (mean)	4.5 (mean)
						41	0.024 c 0.045	0.403 c 0.074	< 0.01	0.43	0.076
						(73)	0.037	0.478	< 0.01	0.52	0.075
						82	0.119 c0.017	0.465 c0.05	0.12	0.58	0.233
						(70)	0.098	0.297	0.052	0.39	0.130
RV218-10HA, Geneva, Minnesota USA, 2010 (Viking 3000)	2	51	205	158	Forage	7	4.76	1.23	0.668	6.0	0.240
	(10)	59	208	164		(20)	3.53	1.10	0.558	4.6	0.202
										5.3 (mean)	0.22 (mean)
						47	< 0.01	0.452 c0.05	< 0.01	0.46	< 0.01
						(22)	< 0.01	0.463	< 0.01	0.47	< 0.01

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
						95	< 0.01	0.371 c0.055	< 0.01	0.38	< 0.01
						(23)	< 0.01	0.400	< 0.01	0.41	< 0.01
					Hay	7	4.87	1.88 c0.058	0.99	6.7	0.441
						(60)	6.12	2.44	1.00	8.6	0.575
							5.5 (mean)			7.7 (mean)	0.51 (mean)
						47	0.014 *c0.013*	1.32 c0.060	< 0.01	1.3	0.033
						(53)	< 0.01	1.06	< 0.01	1.1	0.024
						95	< 0.01	1.30 c0.053	< 0.01	1.3	0.017
						(80)	< 0.01	1.13	< 0.01	1.1	0.020
RV219-10HA, Springfield, Nebraska USA, 2010 (Nebraska Certified)	2	51	207	140	Forage	7	7.05	0.959	0.918	8.0	0.327
	(9)	55	208	138		(19)	8.69	0.961	1.00	9.7	0.270
										8.8 (mean)	0.30 (mean)
					Hay	7	7.61	1.87 c0.096	1.17	9.5	0.513
						(55)	9.28	1.81	1.20	11	0.530
							8.4 (mean)			10 (mean)	0.52 (mean)
RV219-10HB, Springfield, Nebraska USA, 2011 (Nebraska Certified)	2	55	207	132	Forage	5	2.96	1.21	0.569	4.2	0.043
	(8)	59	207	132		(21)	2.32	1.14	0.480	3.5	0.047
										3.8 (mean)	0.045 (mean)
						37	0.018	0.666 c0.101	< 0.01	0.68	0.011
						(24)	0.011	0.730	< 0.01	0.74	0.01
						63	< 0.01	0.947 c0.275	< 0.01	0.96	0.017
						(28)	< 0.01	0.795	< 0.01	0.81	0.019
					Hay	5	4.79 c0.015	2.97	1.13	7.8	0.779
						(58)	3.68	3.30	0.738	7.0	0.839
							4.2 (mean)			7.4 (mean)	0.81 (mean)
						37	0.030 c0.017	2.07 c0.062	0.013	2.1	0.037
						(66)	0.031	1.80	< 0.01	1.8	0.038
						63	0.016	1.72 c0.108	< 0.01	1.7	0.038
						(70)	0.015	1.59	< 0.01	1.6	0.030
RV220-10HA, Stafford, Kansas USA, 2010 (Pioneer 5010)	2	45	204	187	Forage	6	2.55	1.33 c0.051	0.528	3.9	0.265
	(8)	51	206	188		(28)	3.57	1.33	0.537	4.9	0.272
										4.4 (mean)	0.27 (mean)
						35	< 0.01	0.552	< 0.01	0.56	0.024
						(25)	< 0.01	0.541	< 0.01	0.55	0.018
						61	< 0.01	0.196 c0.078	< 0.01	0.21	< 0.01
						(25)	< 0.01	0.289	< 0.01	0.30	0.01
					Hay	6	5.62	4.75	1.35	10	2.18
						(75)	6.16	4.40	1.22	11	2.00
							5.9 (mean)			10 (mean)	2.1 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
						35	< 0.01 *c0.021*	1.68 c0.075	0.027	1.7	0.087
						(75)	0.022	1.92	0.018	1.9	0.092
						61	< 0.01	0.756 c0.052	< 0.01	0.77	0.076
						(74)	0.013	0.739	< 0.01	0.75	0.074
RV221-10DA,	2	49	208	268	Forag	0	7.19	1.06	0.345	8.2	0.294
Carlyle,	(9)	59	208	263	e	(18)	17.7	1.86	0.900	20	0.534
Illinois						3	7.94	2.74	1.23	11	0.739
USA, 2010						(19)	9.85	3.28	1.35	13	0.910
(NK 919)						7	3.63	3.07	0.733	6.7	0.744
						(20)	3.74	3.27	0.792	7.0	0.959
										6.9 (mean)	0.85 (mean)
						14	0.609	3.37	0.151	4.0	0.536
						(21)	0.748	2.83	0.152	3.6	0.398
						21	0.174	2.95	0.015	3.1	0.864
						(26)	0.180	2.93	0.027	3.1	0.703
						60	< 0.01	0.804	< 0.01	0.81	< 0.01
						(25)	< 0.01	0.469	< 0.01	0.48	0.013
						118	< 0.01	0.139	< 0.01	0.15	< 0.01
						(26)	< 0.01	0.187	< 0.01	0.20	< 0.01
					Hay	0	35.0	7.61	2.54	43	2.34
						(75)	72.6	11.9	4.02	85	3.10
						3	22.1	11.7	3.97	34	2.98
						(62)	26.2	10.4	4.40	37	3.06
						7	11.2	17.9 c0.062	3.09	29	4.67
						(70)	7.65	13.6	1.87	21	5.08
							9.4 (mean)			25 (mean)	4.9 (mean)
						14	2.20	12.1	0.497	14	3.46
						(75)	2.17	13.9	0.513	16	3.25
						21	0.357	10.8	0.056	11	1.74
						(83)	0.378	10.6	0.062	11	2.05
						60	0.027	1.59 c0.116	< 0.01	1.6	0.044
						(71)	0.016	1.80	< 0.01	1.8	0.040
						118 (61)	0.023 c0.033	0.443 c0.120	< 0.01	0.47	0.01
							0.019	0.525	< 0.01	0.54	0.011
RV222-10DA,	2	51	205	189	Forag	0	5.78	0.828	0.411	6.6	0.220
York,	(8)	60	205	188	e	(19)	4.94	0.824	0.344	5.8	0.273
Nebraska						3	4.25	1.82	0.722	6.1	0.482
USA, 2010						(22)	3.98	1.40	0.619	5.4	0.474
(WL 319 HQ)						7	3.03	1.88	0.700	4.9	0.594
						(22)	4.24	2.11	0.784	6.4	0.496
										5.6 (mean)	0.54 (mean)
						14	0.964	2.06	0.309	3.0	0.428
						(24)	1.29	2.39	0.338	3.7	0.652
						21	0.270	1.97	0.089	2.2	0.630
						(25)	0.312	1.77	0.095	2.1	0.516
						51	0.013	0.842	< 0.01	0.85	< 0.01
						(24)	< 0.01	0.758	< 0.01	0.77	< 0.01
						79	< 0.01	0.471	< 0.01	0.48	< 0.01
						(22)	< 0.01	0.496	< 0.01	0.51	< 0.01

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
					Hay	0	23.3	4.07	2.11	27	1.11
						(89)	22.3	3.66	2.11	26	1.02
						3	11.6	5.69	2.20	17	1.21
						(82)	9.67	4.19	1.87	14	0.966
						7	9.62	5.44 c0.072	2.24	15	1.28
						(84)	8.98	6.03	2.07	15	1.25
							9.3 (mean)			15 (mean)	
						14	2.23	5.08	0.770	7.3	1.47
						(85)	2.53	4.93	0.590	7.5	1.86
											1.7 (mean)
						21	0.519	4.98	2.05	5.5	1.10
						(87)	1.23	6.69	0.336	7.9	1.53
						51	< 0.01 *c0.019*	2.33 c0.069	< 0.01	2.3	0.027
						(83)	< 0.01	2.04	< 0.01	2.1	0.158
						79	0.017	1.52 c0.123	< 0.01	1.5	0.023
						(81)	< 0.01	1.62	< 0.01	1.6	0.012
RV223-10HA, Berthoud, Colorado USA, 2010 (Pioneer)	2 (9)	47 51	208 204	117 118	Forage	7	5.06	1.04	0.513	6.1	0.232
						(26)	3.01	0.834	0.314	3.8	0.185
										5.0 (mean)	0.21 (mean)
						36	0.049 c0.019	0.440 c0.093	< 0.01	0.49	< 0.01
						(26)	0.025	0.505	< 0.01	0.53	< 0.01
						67	< 0.01	0.283 c0.145	< 0.01	0.29	< 0.01
						(25)	< 0.01	0.249	< 0.01	0.26	< 0.01
					Hay	7	6.52	1.88	0.920	8.4	0.563
						(67)	5.92	2.25	0.892	8.2	0.585
							6.2 (mean)			8.3 (mean)	0.57 (mean)
						36	0.066	0.932	0.011	1	0.051
						(75)	0.084	0.782	0.014	0.87	0.057
						67	< 0.01	0.320	< 0.01	0.33	0.012
						(61)	< 0.01	0.285	< 0.01	0.3	0.020
RV224-10HA, Sanger, California USA, 2010 (WL 530 HQ)	2 (9)	55 62	204 206	280 270	Forage	7	1.19	0.549	0.206	1.7	0.055
						(32)	0.940	0.429	0.147	1.4	0.051
										1.6 (mean)	0.053 (mean)
						56	0.014	0.514	0.014	0.53	< 0.01
						(25)	0.014	0.916	0.015	0.93	< 0.01
						111	< 0.01	0.228	< 0.01	0.24	< 0.01
						(28)	< 0.01	0.277	< 0.01	0.29	< 0.01
					Hay	7	5.47	2.68	0.972	8.1	0.464
						(93)	4.08	1.94	0.666	6.0	0.389
							4.8 (mean)			7.1 (mean)	0.43 (mean)
						56	0.045 c0.031	2.09 c0.085	0.055	2.1	0.099
						(90)	0.068	2.61	0.062	2.7	0.122
						111 (84)	0.030 c0.013	0.966 c0.076	0.036	1.0	0.049
							0.020	0.983	0.034	1.0	0.042
RV225-10HA,	2	38	211	159	Forage	7	3.61	0.663	0.427	4.3	0.175

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(%DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
Rupert, Idaho USA, 2010 (Western Choice II)	(9)	51	205	155	e	(16)	3.13	0.641	0.390	3.8	0.154
										4.0 (mean)	0.16 (mean)
						50	0.012	0.546	< 0.01	0.56	< 0.01
						(29)	< 0.01	0.379	< 0.01	0.390	< 0.01
						86	< 0.01	0.217	< 0.01	0.23	< 0.01
						(24)	< 0.01	0.217	< 0.01	0.23	< 0.01
					Hay	7	9.02	2.80 c0.053	1.38	12	1.26
						(71)	9.93	3.59	1.56	14	2.01
							9.5 (mean)			13 (mean)	1.6 (mean)
						50	0.015 c0.019	1.33 c0.064	< 0.01	1.3	0.028
						(81)	0.012	1.43	< 0.01	1.4	0.027
						86	< 0.01	0.795	< 0.01	0.81	0.037
						(85)	< 0.01	0.763	< 0.01	0.77	0.026

LOQ is 0.01 mg/kg for parent flupyradifurone and the metabolites DFEAF and 6-CNA (parent equivs.) and 0.05 mg/kg for DFA (parent equivs.) in forage and stover

Four supervised trials were carried out on clover (Table 141) in the USA during the 2011 growing season to determine residues after two foliar applications of BYI 02960 200 SL (Dorschner 2012, RARVY017). All applications were made using ground-based equipment and included a non-ionic surfactant adjuvant (Washington trial) or a crop oil concentrate (Oregon trials) at a rate of 0.25% v/v.

Residues of flupyradifurone, difluoroacetic acid (DFA), difluoroethylaminofuranone (DFEAF) and 6-chloronicotinic acid (6-CNA) in clover forage and hay were determined using LC/MS/MS Method 01304. Acceptable concurrent recovery data (mean recoveries, RSDs) were obtained for all analytes.

Table 141 Residues in clover forage and hay from the foliar application of flupyradifurone to clover in the USA (Dorschner 2012, RARVY017)

Trial No., Location, Year (Variety)	Application			Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	Parent + DFA	6-CNA
GAP, USA, Clover	2 (10)	205			Forage 7 Hay 14					
OR32,	2	204	233	Forage	6	6.20	0.151	0.398	6.35	0.035
Aurora,	(9)	202	231			5.82	0.148	0.339	5.97	0.035
Oregon									6.2 (mean)	0.035 (mean)
USA, 2011				Hay	12	11.6	0.919	1.06	12.5	0.13
(Medium Red)						10.7	0.905	1.02	11.6	0.15
	2	103	230	Forage	6	3.75	0.088	0.198	3.84	0.024
	(9)	104	233			3.24	0.084	0.203	3.32	0.019
				Hay	12	5.43	0.652	0.554	6.08	0.097
						5.51	0.544	0.556	6.05	0.078
OR33, Aurora, Oregon USA, 2011 (Medium Red)	2	208	361	Forage	6	5.20	0.134	0.282	5.34	0.042
	(9)	209	361			4.37	0.112	0.220	4.48	0.048
									4.9 (mean)	0.045 (mean)
				Hay	14	9.08	0.875	0.209	9.95	0.57
						7.50	0.762	0.351	8.26	0.11

Trial No., Location, Year (Variety)	Application			Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	Parent + DFA	6-CNA
	2	102	355	Forage	6	2.71	0.090	0.152	2.79	0.026
	(9)	103	360			2.37	0.066	0.101	2.44	0.022
				Hay	14	3.70	0.522	0.231	4.23	0.089
						3.34	0.436	0.459	3.77	0.15
OR34,	2	206	839	Forage	7	5.37	0.122	0.166	5.49	0.033
Sherwood,	(12)	203	830			3.82	0.108	0.135	3.93	0.032
Oregon									4.7 (mean)	0.032 (mean)
USA, 2011				Hay	11	10.2	0.851	0.577	11.0	0.31
(Medium Red)						9.96	0.751	0.509	10.7	0.29
	2	98	810	Forage	7	1.65	0.052	0.082	1.70	0.015
	(12)	100	814			1.82	0.055	0.080	1.88	0.016
				Hay	11	3.49	0.382	0.217	3.88	0.12
						4.07	0.425	0.286	4.49	0.18
WA32, Prosser, Washington USA, 2011 (Medium Red)	2	206	248	Forage	7	6.37	0.153	0.283	6.53	0.028
	(10)	204	256			5.16	0.151	0.257	5.31	0.028
									5.9 (mean)	0.028 (mean)
				Hay	17	9.55	1.98	0.687	11.5	0.15
						9.45 c0.054	1.81	0.675	11.3	0.11
	2	103	248	Forage	7	2.68	0.114	0.133	2.80	0.01
	(10)	102	255			2.79	0.161	0.157	2.95	0.01
				Hay	17	4.49	1.54	0.335	6.03	0.05
						5.11 c0.054	1.54	0.345	6.65	0.060

LOQ is 0.01 mg/kg for parent flupyradifurone and the metabolites DFEAF and 6-CNA (parent equivs.) and 0.05 mg/kg for DFA (parent equivs.) in forage

LOQ is 0.02 mg/kg for parent flupyradifurone and the metabolites DFEAF and 6-CNA (parent equivs.) and 0.10 mg/kg for DFA (parent equivs.) in hay

Table 142 Residues in almond hulls from the foliar application of flupyradifurone to almonds in the USA (Niczyporowicz and Netzband 2012, RARVY016)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% dry matter)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
RV204-10DA,	2	78	204	423	Hull	0	1.4	0.10	0.05	1.5	0.080
Madera,	(14)	79	205	425		(22)	2.6	0.09	0.02	2.7	0.030
California,						3	2.5	0.12	0.02	2.6	0.035
USA, 2010						(25)	2.6	0.17	0.02	2.7	0.046
(Non-Pareil)						7	1.7	0.16	< 0.01	1.9	0.030
						(23)	1.9	0.19	0.01	2.1	0.043
							1.8 (mean)			2.0 (mean)	0.036 (mean)
						14	2.3	0.22	0.02	2.5	0.032
						(39)	3.2	0.43	0.03	3.6	0.032
							2.7 (mean)			3.1 (mean)	0.032 (mean)
						21	2.6	0.40	0.03	3.0	0.037
						(79)	2.2	0.37	0.03	2.6	0.024
							2.4 (mean)			2.8 (mean)	0.030 (mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% dry matter)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
	2	78	210	2131	Hull	7	2.0	0.11	0.01	2.10	0.037
	(14)	79	211	2147		(23)	1.4	0.10	0.02	1.50	0.024
							1.7 (mean)			1.8 (mean)	0.03 (mean)
RV205-10DA,	2	78	204	94	Hull	0	0.57	< 0.05	< 0.01	0.62	< 0.01
Orland,	(14)	89	206	94		(26)	0.68	< 0.05	< 0.01	0.73	< 0.01
California,						3	0.25	< 0.05	< 0.01	0.30	< 0.01
USA, 2010						(32)	0.29	< 0.05	< 0.01	0.34	< 0.01
(Non-Pareil)						7	0.043	< 0.05	< 0.01	0.090	< 0.01
						(48)	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
							0.03 (mean)			0.08 (mean)	
						14	0.028	< 0.05	< 0.01	0.080	< 0.01
						(74)	0.032	< 0.05	< 0.01	0.080	< 0.01
							0.03 (mean)			0.08 (mean)	
						21	0.03	< 0.05	< 0.01	0.08	< 0.01
						(93)	< 0.01	< 0.05	< 0.01	< 0.060	< 0.01
							0.02 (mean)			0.07 (mean)	
	2	78	204	1869	Hull	7	0.20	< 0.05	< 0.01	0.25	< 0.01
	(14)	89	204	1871		(48)	0.20	< 0.05	< 0.01	0.25	< 0.01
							0.20 (mean)			0.25 (mean)	
RV206-10HA,	2	85	203	379	Hull	7	2.5	0.053	< 0.01	2.6	< 0.01
Hickman,	(14)	89	205	382		(86)	3.1	0.053	0.024	3.1	< 0.01
Florida, USA, 2011							2.8 (mean)			2.9 (mean)	
(Sonora)	2	85	201	2217	Hull	7	3.8	< 0.05	0.024	3.8	< 0.01
	(14)	89	205	2185		(87)	4.0	< 0.05	0.028	4.1	< 0.01
							3.9 (mean)			3.9 (mean)	
RV207-10HA,	2	85	205	358	Hull	7	0.23	< 0.05	< 0.01	0.28	< 0.01
Lost Hills,	(14)	85	205	353		(22)	0.18	0.061	< 0.01	0.24	< 0.01
California,							0.20 (mean)			0.26 (mean)	
USA, 2011	2	85	206	2296	Hull	7	0.54	0.054	< 0.01	0.59	< 0.01
(Monterey)	(14)	85	206	2216		(25)	0.46	< 0.05	< 0.01	0.51	< 0.01
							0.50 (mean)			0.55 (mean)	
RV208-10HA,	2	79	201	417	Hull	7	4.6	< 0.05	0.020	4.7	0.031
Kerman,	(14)	85	202	420		(79)	5.2	< 0.05	0.035	5.2	0.027
California,							4.9 (mean)			4.9 (mean)	0.03 (mean)
USA, 2010	2	79	206	2364	Hull	7	4.5	0.079	0.014	4.5	0.024
(Padre)	(14)	85	209	2391		(67)	6.2	0.076	0.018	6.2	0.016
							5.3 (mean)			5.4 (mean)	0.02 (mean)

LOQ is 0.01 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 0.05 mg/kg for DFA (parent equivs.)

Table 143 Residues in cotton gin by-products from the foliar application of flupyradifurone to cotton in the USA (Timberlake and Harbin 2012, RARVY009)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
GAP, USA, Cotton	2 (10)		205			14					
RV111-10HA, Suffolk, Virginia, USA, 2010 (PHY375WRF)	2	87	205	186	Gin,	19	3.0	< 1.00	< 0.20	4.0	0.22
	(9)	88	206	188	by-product	(89)	2.7	< 1.00	0.22	3.7	0.24
							2.9 (mean)			3.9 (mean)	
							c 1.12				
RV112-10HA, Wall, Texas, USA, 2010 (FM 1740 B2F)	2	87	206	143	Gin,	13	13	< 1.00	0.25	14	0.32
	(8)	88	207	142	by-product	(91)	13	< 1.00	0.27	14	0.30
							12.5 (mean)			14 (mean)	0.31 (mean)
							c 0.220				
RV116-10DA, Greenville, Mississippi, USA, 2010 (ST 5458BIIRF)	2	88	206	112	Gin,	0	57	< 1.00	0.51	58	0.39
	(8)	89	206	111	by-product	6	37	< 1.00	0.63	38	0.66
						14	19	< 1.00	0.40	20	0.38
						(73)	20	< 1.00	0.54	21	0.50
							20 (mean)			21 (mean)	0.44 (mean)
							c 1.74				
						21	10	< 1.00	< 0.200	11	0.23
						27	8.3	< 1.00	< 0.200	9.3	0.46
RV117-10DA, Proctor, Arkansas, USA, 2010 (DynaGro2400 RF)	2	88	205	95	Gin,	0	26.3	< 1.00	0.26	27	0.40
	(10)	89	205	95	by-product	7	13	< 1.00	0.23	14	0.37
						13	22	< 1.00	0.30	23	0.58
						(91)	15	< 1.00	0.27	16	0.42
							18 (mean)			19 (mean)	0.50 (mean)
							c 1.85				
						21	8.0	< 1.00	< 0.200	9.0	0.28
						28	3.2	< 1.00	< 0.200	4.2	0.24
RV118-10DA, Uvalde, Texas, USA, 2010 (Stoneville 5458)	2	88	202	170	Gin,	0	9.7	< 1.00	< 0.200	11	0.17
	(9)	89	202	184	by-product	6	9.7	< 1.00	< 0.200	11	0.20
						14	8.0	< 1.00	< 0.200	9.0	0.37
						(82)	6.6	< 1.00	< 0.200	7.6	0.33
							7.3 (mean)			8.3 (mean)	0.35 (mean)
						19	7.5	< 1.00	< 0.200	8.5	0.49
						28	7.3	< 1.00	< 0.200	8.3	0.57
RV119-10DA,	2	89	204	283	Gin,	0	11	< 1.00	0.203	12	0.37
Hinton, Oklahoma,	(9)	89–99	207	286	by-product	7	6.1	< 1.00	< 0.200	7.1	0.37
USA, 2010						13	5.93	< 1.00	< 0.200	6.9	0.34
(FM9063B2F)						(74)	6.33	< 1.00	< 0.200	7.3	0.31
							6.1 (mean)			7.1 (mean)	0.32 (mean)
							c 0.201			c 1.2	
						28	4.13	< 1.00	< 0.200	5.1	0.32

LOQ is 0.20 mg/kg for each of parent flupyradifurone and the metabolites DFEAF and 6-CNA and 1.0 mg/kg for DFA (parent equivs.)

Table 144 Residues in peanut hay from the foliar application of flupyradifurone to peanuts in the USA (Krolski and Harbin 2012a, RARVY010)

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
GAP, USA, Peanuts	2 (10)		205			7					
RV120-10HA,	2	83	205	130	Hay	6	3.28	1.70	0.077	5.0	0.098
Elko, South Carolina,	(11)	86	206	140		(90)	4.04	1.73	0.080	5.8	0.11
USA, 2010							3.7			5.4	0.11
(Gregory)							(mean)			(mean)	(mean)
RV121-10HA,	2	79	206	270	Hay	7	1.90	1.52	0.060	3.4	< 0.05
Athens, Georgia,	(10)	79	204	280		(37)	2.18	1.92	0.065	4.1	0.051
USA, 2010							2.0			3.8	0.051
(Georgia-06G)							(mean)			(mean)	(mean)
RV122-10HA,	2	87	210	120	Hay	7	2.15	2.22	< 0.05	4.4	0.44
Suffolk, Virginia,	(10)	88	211	120		(47)	3.31	3.27	< 0.05	6.6	0.69
USA, 2010							2.7			5.5	0.57
(Champs)							(mean)			(mean)	(mean)
RV123-10HA,	2	89	204	230	Hay	7	8.84	3.49	0.292	12.33	0.22
Tallassee, Alabama,	(10)	89	206	190		(89)	9.29	3.65	0.277	12.9	0.21
USA, 2010							9.1			13	0.22
(Georgia Greener)							(mean)			(mean)	(mean)
RV124-10HA,	2	85	201	190	Hay	7	6.34	2.70	0.178	9.0	0.078
Seven Springs, North Carolina,	(10)	87	205	200		(52)	5.04	2.34	0.172	7.4	0.082
USA, 2010							5.7			8.2	0.080
(Perry)							(mean)			(mean)	(mean)
RV128-10DA,	2	88	208	200	Hay	0	16.5	1.32	0.206	18	0.053
Seven Springs, North Carolina,	(10)	89	205	200		(47)	11.7	0.981	0.138	13	< 0.05
USA, 2010						3	10.7	1.43	0.169	12	0.055
(Champs)						(56)	9.36	1.35	0.180	11	0.066
						7	8.52	1.42	0.224	9.9	0.16
						(58)	9.80	1.49	0.211	11	0.16
						14	9.87	2.06	0.217	12	0.88
						(66)	11.0	2.09	0.249	13	1.1
							10 (mean)			13 (mean)	
						21	7.99	1.67	0.156	9.7	1.4
						(62)	6.76	1.79	0.154	8.6	1.4
											1.4 (mean)
RV125-10HA,	2	88	205	220	Hay	8	4.50	1.65	0.199	6.2	0.10
Cuthbert, Georgia,	(10)	88	205	230		(83)	4.48	1.36	0.164	5.8	0.081
USA, 2010							4.5			6.0	0.093
(Georgia Green)							(mean)			(mean)	(mean)
RV126-10HA,	2	87	208	190	Hay	3	8.61	2.35	0.365	11	0.24
Greenville, Florida,	(7)	89	205	190		(90)	6.99	2.06	0.281	9.1	0.25
USA, 2010											0.25
(GA-06)											(mean)

Trial No., Location, Year (Variety)	Application				Sample	DALA (% DM)	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)			Parent	DFA	DFEAF	Parent + DFA	6-CNA
RV127-10HA,	2	84	198	180	Hay	6	4.01	0.971	0.155	5.0	0.066
Hinton, Oklahoma,	(10)	85	199	200		(78)	5.95	1.15	0.176	7.1	0.079
USA, 2010							5.0			6.0	0.072
(Tamnut 0L06)							(mean)			(mean)	(mean)
RV129-10DA,	2	88	206	230	Hay	0	9.76	1.64	0.477	11	0.33
Tifton, Georgia,	(10)	88	205	230		(82)	9.60	1.98	0.378	12	0.36
USA, 2010						3	1.37	1.20	< 0.05	2.6	0.88
(Georgia 06G)						(78)	1.15	0.767	< 0.05	1.9	0.44
						8	1.35	1.94	0.055	3.3	0.17
						(77)	1.62	1.84	0.074	3.5	0.097
						14	1.65	3.64	0.063	5.3	0.26
						(84)	1.68	3.79	0.057	5.5	0.31
							1.7 (mean)			5.4 (mean)	
						21	0.561	1.78	< 0.05	2.3	0.25
						(80)	1.05	3.20	< 0.05	4.3	0.43
											0.34 (mean)
RV130-10DA,	2	84	205	180	Hay	0	10.8	0.580	0.259	11	< 0.05
Madill, Oklahoma,	(10)	89	206	180		(65)	14.0	0.658	0.309	15	< 0.05
USA, 2010						3	9.82	1.13	0.479	11	0.072
(Tamrun)						(83)	12.3	1.14	0.57	13	0.081
						7	16.9	1.91	0.676	18	0.13
						(88)	5.49	1.10	0.274	6.6	0.063
							11 (mean)			13 (mean)	
						14	14.2	1.67	0.647	16	0.11
						(85)	6.16	1.20	0.359	7.4	0.077
											0.096 (mean)
						21	11.7	1.44	0.471	13	0.094
						(85)	7.84	1.24	0.328	9.1	0.076
RV131-10DA,	2	85	207	180	Hay	0	12.0	0.598	0.239	13	0.052
Wellington, Texas,	(10)	89	208	180		(71)	12.0	0.596	0.246	13	< 0.05
USA, 2010						3	10.3	0.978	0.466	11	0.062
(Florida 07)						(87)	10.0	1.27	0.531	11	0.073
						7	11.0	1.66	0.564	13	0.10
						(87)	10.5	1.56	0.503	12	0.093
							11 (mean)			12 (mean)	0.098 (mean)
						14	2.81	0.829	0.159	3.6	< 0.05
						(84)	3.57	0.928	0.238	4.5	< 0.05
						21	3.05	0.859	0.202	3.9	< 0.05
						(84)	1.72	0.56	0.110	2.3	< 0.05

Trial No., Location, Year (Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage	Rate (g ai/ha)	Volume (L/ha)		(% DM)	Parent	DFA	DFEAF	Parent + DFA	6-CNA
								5			

LOQ is 0.05 mg/kg for each of parent flupyradifurone and the metabolites DFEAF, DFA and 6-CNA (parent equivs.)

FATE OF RESIDUES IN STORAGE AND PROCESSING

Residues after processing

The fate of flupyradifurone residues during processing of raw agricultural commodities was investigated in citrus fruit (orange), pome fruit (apples), grapes, strawberries, Brassica vegetables (broccoli), fruiting vegetables (summer squash, tomato, cucumber), leafy vegetables (Indian mustard, spinach), pulses (soya bean), root and tuber vegetables (carrot, potato), cereals (barley, maize, wheat), oilseed crops (cotton, peanut), coffee and hops.

As a measure for the transfer of residues into processed products, a transfer factor (TF) was used, this is defined as:

$$TF = \frac{\text{Residue in processed products (mg/kg)}}{\text{Residue in raw agricultural commodity (mg/kg)}}$$

The high temperature hydrolysis of residues of flupyradifurone under varying conditions was studied (Weber 2011a, MEF-10/856). An acetonitrile solution of [pyridinylmethyl-^{14}C]-flupyradifurone was added to aqueous buffer solutions and subjected to hydrolysis at pH 4, 5 and 6 at high temperature at concentrations of 0.92, 0.77 and 1.04 µg/ml respectively. The conditions were selected to simulate hydrolysis under processing conditions and included:

The effect of pasteurisation (pH 4 and pasteurised at 90 °C for 20 minutes)

The effect of baking, brewing and boiling (pH 5 and baked at 100 °C for 60 minutes)

The effect of sterilisation (pH 6 and autoclaving at 120 °C for 20 minutes)

The pH ranged from 3.97 (beginning of the trial)–3.98 (end of the trial) for the pH 4 samples, 4.97 (beginning of the trial)–4.98 (end of the trial) for the pH 5 samples and from 5.95 (beginning of the trial)–6.12 (end of the trial) for the pH 6 samples.

No major loss of radioactivity material occurred; pH 4 at 90 °C experiment—101.7% of total applied radioactivity remained after test, pH 5 at 100 °C experiment—104.9% of total applied radioactivity remained after test, pH 6 at 120 °C experiment—97.3% of total applied radioactivity remained after test. The amount of radioactive material in the samples at the end of the test corresponded to parent compound only.

In summary, the data show that flupyradifurone was not degraded during the simulation of pasteurisation (pH 4, 90 °C, 20 minutes), during the simulation of baking, boiling and brewing (pH 5, 100 °C, 60 minutes) and during sterilisation (pH 6, 120 °C, 20 minutes). No hydrolysis products of flupyradifurone were detected above an estimated LOD of 0.5%.

Oranges

The effect of processing (laboratory scale) on residues of flupyradifurone in oranges (Table 145) was investigated in two trials carried out in the 2010 growing season in Spain (Schulte and Teubner 2012b, 10-3405). Oranges with incurred residues were obtained where trees were sprayed with one foliar application at 375 g ai/ha. The application was made approximately 30 days prior to harvest of mature oranges at beginning of ripening (BBCH 81). Orange bulk RAC samples were harvested 29 days after treatment.

Bulk orange samples (one control and one treated for each trial) were processed into processed orange commodity samples (juice, marmalade and oil) per simulated commercial procedures. Before the start of processing into marmalade, juice and oil specimens of fruit, stored were taken. Orange fruits were washed manually in tap water for three minutes giving specimens of whole fruit, washed and washings. Washed orange fruits were peeled by hand using a knife. Peel and peeled oranges were cut into smaller pieces. Sugar, glucose syrup and citric acid were added to the mixture of approximately 85% peeled fruit (pulp) and 15% peel. After heating and cooling, a 5% pectin solution was added and the intermediate was further cooked until a dry matter content of 60–62% was reached (pH 2.5–2.6). After cooling, specimens of marmalade were taken.

Washed orange fruits were cut in halves and squeezed with a citrus fruit squeezer which separated the fruit into raw juice, remaining pulp (without peel) and orange peel by a rotating cone and an integrated sieve. The raw juice was pasteurised for 2 minutes at 80–92 °C. Specimens of raw juice were taken before pasteurisation and specimens of juice after pasteurisation. The remaining orange peel was cut into small pieces to produce (together with the remaining pulp) pomace, wet. Specimens of pomace, wet were taken. The wet pomace was dried in drying cupboards at 63–65 °C until the moisture content was 10% (pomace, dried).

The field sample was washed manually in tap water for three minutes. Some unwashed orange fruits were separated into peel and pulp for the preparation of the specimen peel (unwashed).

Washed orange fruits were separated into peel and pulp. Specimens of peel, washed and pulp were taken. Subsequently the separated peel was used only for the oil production in a steam distillation plant.

The washed peel was put in a special vessel with an inside stirrer. A direct steam jet was transferred through the bulk goods. The process resulted in a steam mixture which was cooled down and condensed in a cooler and sampled in a collecting vessel. After self-separation of the oil containing phase and the water containing phase, the water was removed. The oil containing phase was concentrated in a centrifuge. Orange oil and peel without oil were sampled.

Residues of flupyradifurone and metabolites in orange RAC and processed commodities were quantitated by LC/MS/MS using Method 01304. Acceptable concurrent recovery data for orange commodities were obtained for each analyte.

Table 145 Residues in orange processed fractions from the foliar application of flupyradifurone to oranges in Spain (Schulte and Teubner 2012, 10-3405)

Trial No.,	Sample	DAT	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Crop, Variety)							+ 6-CNA		+ 6-CNA
GAP, USA, Citrus Foliar		1							
10-3405-01,	Fruit (RAC)	29	0.21	0.03	< 0.01	< 0.01	0.24	NA	NA
Los Palacios,	Pulp	29	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.048	0.13
Spain, 2010	Fruit, stored	29	0.17	0.04	< 0.01	< 0.01	0.21	NA	NA
(Orange, Navelina)	Whole fruit, washed	29	0.17	0.03	< 0.01	< 0.01	0.20	0.81	0.83
	Washings	29	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.048	< 0.13
1 application	Pomace, wet	29	0.29	0.04	< 0.01	< 0.01	0.33	1.4	1.4
Growth Stage 81	Pomace, dried	29	0.89	0.11	< 0.01	0.018	1.0	4.2	4.2
375 g ai/ha	Raw juice	29	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.048	< 0.13
1000 L/ha	Juice	29	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.048	< 0.13
	Fruit, stored	29	0.12	0.03	< 0.01	< 0.01	0.15	NA	NA
	Marmalade	29	0.02	< 0.02	< 0.01	< 0.01	0.04	0.095	0.17

Trial No.,	Sample	DAT	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Crop, Variety)							+ 6-CNA		+ 6-CNA
	Fruit, stored	29	0.12	0.03	< 0.01	< 0.01	0.15	NA	NA
	Pulp	29	0.02	0.03	< 0.01	< 0.01	0.05	0.095	0.21
	Peel	29	0.46	0.04	< 0.01	< 0.01	0.50	2.2	2.1
	Peel, washed	29	0.62	0.05	< 0.01	< 0.01	0.67	3.0	2.8
	Peel without oil	29	0.65	0.06	< 0.01	0.016	0.73	3.1	3.0
	Oil	29	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.048	< 0.13
10-3405-02,	Fruit (RAC)	29	0.12	0.02	< 0.01	< 0.01	0.14	NA	NA
Olivares,	Pulp	29	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.083	< 0.21
Spain, 2010	Fruit, stored	29	0.04	< 0.02	< 0.01	< 0.01	0.06	NA	NA
(Orange, Navelina)	Whole fruit, washed	29	0.19	0.02	< 0.01	< 0.01	0.21	1.6	1.5
	Washings	29	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.083	< 0.21
1 application	Pomace, wet	29	0.15	0.03	< 0.01	< 0.01	0.18	1.3	1.3
Growth Stage 83	Pomace, dried	29	0.63	0.09	< 0.01	0.013	0.72	5.3	5.2
375 g ai/ha	Raw juice	29	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.083	< 0.21
1000 L/ha	Juice	29	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.083	< 0.21
	Fruit, stored	29	0.11	0.03	< 0.01	< 0.01	0.14	NA	NA
	Marmalade	29	0.01	< 0.02	< 0.01	< 0.01	0.03	0.083	0.21
	Fruit, stored	29	0.08	< 0.02	< 0.01	< 0.01	0.10	NA	NA
	Pulp	29	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.083	< 0.21
	Peel	29	0.20	0.03	< 0.01	< 0.01	0.23	1.7	1.6
	Peel, washed	29	0.27	0.03	< 0.01	< 0.01	0.30	2.3	2.1
	Peel without oil	29	0.51	0.05	< 0.01	< 0.01	0.56	4.3	4.0
	Oil	29	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.083	< 0.21

LOQ of parent, DFEAF and 6-CNA 0.01, DFA 0.02 mg/kg (parent equivs.) for all processed commodities

The effect of processing (laboratory scale) on residues of flupyradifurone in oranges (Table 146) was also investigated in two trials carried out in the 2010 growing season in the USA (Lenz 2012d, RARVY035). Oranges with incurred residues were obtained where trees were sprayed with two foliar applications at 1 kg ai/ha. All applications were made using ground-based equipment. A non-ionic surfactant was used in all applications. Single composite samples were taken from treated and control plots at 1 day after the last application.

Bulk orange samples were processed into processed orange commodity samples (juice, marmalade and oil) per simulated commercial procedures.

A representative unwashed sample was taken to generate unwashed sample fractions. A representative sample of unwashed peel was taken.

Oranges were batch washed. Representative samples of washed fruit were taken. An aliquot of washed fruit was peeled to obtain a washed peel sample. A representative sample of wash water was also obtained.

An aliquot of washed fruit was put aside for marmalade. The rind of the oranges for marmalade processing was removed with a vegetable peeler. Rind was chipped in a food processor and cooked for 20 minutes and set aside. The remaining rind on the oranges was removed and the seeds discarded. The orange pulp was chopped in the food processor and cooked on the stove for 45 minutes with 20% water to fruit by weight. The rind and fruit were combined with lemon juice and sugar. The mixture was boiled, pectin added and the mixture boiled again. After standing the marmalade was packed in jars.

Remaining washed oranges were transferred to a Hobart peeler for scarifying. An aliquot of scarified fruit was set aside for the peel without oil fraction. The collected oil-water emulsion was screened to separate any flavedo fragments from the oil-water emulsion. The scarified flavedo was set aside for later addition to the shredded peel. The cream separator was used to separate the oil. Free oil was removed. The residual emulsion was frozen, thawed, centrifuged and further oil removed.

Juice was recovered from the scarified oranges. Juice was transferred to the pulper finisher and screened to give the required juice fraction. Remaining fresh juice was pasteurised.

Peel from the juice extractor was shredded using the food processor. Shredded peel was combined with the scarified flavedo to generate wet peel. A representative sample of the wet pulp was removed.

Lime was added to the remaining wet peel and mixed. The limed peel was pressed. A representative sample of wet pomace was removed. Remaining wet peel pulp was dried to below 10% moisture. The dried pulp was milled using the hammermill.

An additional aliquot of scarified oranges was weighed and transferred to the juice extractor to recover juice from the peel for strainings. The juice and peel recovered were transferred to the pulper finisher and screened to remove vesicular membranes, seeds, segment membranes and peel fragments from the juice. A representative sample of the strainings was taken.

Residues of flupyradifurone and metabolites in orange RAC and processed commodities were quantitated by using LC/MS/MS Method 01304. Acceptable concurrent recovery data for orange commodities were obtained for each analyte.

Table 146 Residues in orange processed fractions from the foliar application of flupyradifurone to oranges in the USA (Lenz 2012d, RARVY035)

Trial No.,	Sample	DALA	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Crop, Variety)							+ 6-CNA		+ 6-CNA
GAP, USA, Citrus Foliar		1							
RV200-11PA,	Fruit (RAC)	1	0.393	< 0.05	< 0.01	< 0.01	0.44	NA	NA
Arroyo Grande,	Peel ripe, unwashed	1	1.034	< 0.05	< 0.01	< 0.01	1.1	2.6	2.5
California,	Fruit, washed	1	0.429	< 0.05	< 0.01	< 0.01	0.48	1.1	1.1
USA, 2011	Washings	1	0.019	< 0.05	< 0.01	< 0.01	0.069	0.048	0.16
(Orange, Olinda)	Peel, washed	1	1.81	< 0.05	< 0.01	< 0.01	1.9	4.6	4.3
	Peel without oil	1	0.917	< 0.05	< 0.01	< 0.01	0.97	2.3	2.2
2 applications	Raw juice	1	0.015	< 0.05	< 0.01	< 0.01	0.065	0.038	0.15
10 day RTI	Juice	1	0.011	< 0.05	< 0.01	< 0.01	0.061	0.028	0.14
Growth Stage 83 (×2)	Oil	1	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.025	< 0.14
1000 and 999 g ai/ha	Strain rest	1	0.013	< 0.05	< 0.01	< 0.01	0.063	0.033	0.14
2515 (×2) L/ha	Pulp	1	0.455	< 0.05	< 0.01	< 0.01	0.51	1.2	1.2
	Pomace, wet	1	0.51	< 0.05	< 0.01	< 0.01	0.56	1.3	1.3
	Pomace, dried	1	2.05	< 0.05	< 0.01	< 0.01	2.1	5.2	4.8
	Marmalade	1	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.025	< 0.14
RV201-11PA,	Fruit (RAC)	1	0.517	< 0.05	< 0.01	< 0.01	0.57	NA	NA
Sanger,	Peel ripe, unwashed	1	0.853	< 0.05	< 0.01	< 0.01	0.90	1.6	1.6
California,	Fruit, washed	1	0.413	< 0.05	< 0.01	< 0.01	0.46	0.80	0.81
USA, 2011	Washings	1	0.025	< 0.05	< 0.01	< 0.01	0.075	0.048	0.13
(Orange, Valencia)	Peel, washed	1	1.56	0.051	< 0.01	< 0.01	1.6	3.0	2.8
	Peel without oil	1	0.986	< 0.05	< 0.01	< 0.01	1.04	1.9	1.8
2 applications	Raw juice	1	0.011	< 0.05	< 0.01	< 0.01	0.061	0.021	0.11
10 day RTI	Juice	1	0.013	< 0.05	< 0.01	< 0.01	0.063	0.025	0.11
Growth Stage 89 (×2)	Oil	1	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.019	< 0.11
1000 (×2) g ai/ha	Strain rest	1	0.015	< 0.05	< 0.01	< 0.01	0.065	0.029	0.11
2291 and 2337 L/ha	Pulp	1	0.461	< 0.05	< 0.01	< 0.01	0.51	0.89	0.89
	Pomace, wet	1	0.545	< 0.05	< 0.01	< 0.01	0.60	1.1	1.1
	Pomace, dried	1	2.03	0.103	0.011	0.0278	2.1	3.9	3.9
	Marmalade	1	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.019	< 0.11

LOQ of parent, DFEAF and 6-CNA 0.01, DFA 0.02 mg/kg (parent equivs.) for all processed commodities

Apple

The effect of processing (laboratory scale) on residues of flupyradifurone in apples (Table 147) was investigated in four trials carried out in the 2010 growing season in Germany and Belgium (Schulte and Bauer 2012c, 10-3171) and Italy and Spain (Schulte and Ballmann 2012a, 10-3172).

Apples with incurred residues were obtained where trees were sprayed with two foliar sprays at 150–188 g ai/ha with a 13 or 14-day retreatment interval. Apple bulk RAC samples were harvested 14 days after the last application. Bulk apple samples (one control and one treated for each trial) were processed into apple commodity samples. The washing and peeling of apple fruits was done using household practice. The preparation of juice, sauce and dried fruit simulated industrial practice at a laboratory scale.

For processing into juice, fruit was washed in lukewarm water. After washing, the fruits were drained in a sieve to create the laboratory samples whole fruit, washed and washings. The washed fruits were crushed into mash in a cutter. The apple mash was pressed in a high-pressure press to obtain the laboratory samples raw juice and pomace, wet. The remining part of the sample material wet pomace was dried in a fan-assisted oven at about 100 °C until the water content was approximately 10% to obtain the laboratory sample pomace, dried. The remaining raw juice was enzymated by heating the juice in a cooking pan for about 5 minutes at 90 °C and a further 5 minutes at 45 °C. The juice was stored overnight at room temperature, then was fined coarsely. The coarsely fined juice was filtered in a laboratory ultra-filtration set-up into the clear juice. The obtained retentate was taken as laboratory sample retentate. The filtered juice was pasteurised in a plate heat-exchanger. After pasteurisation, the juice was collected in fractions. The first fractions had lower Brix values than the filtrate because they were mixed with water still present in the heat exchanger. These fractions were rejected. Only fractions with a similar Brix value compared to the filtrate were collected in a bigger container and constitute the sample pasteurised juice. The laboratory sample juice was taken.

For processing into sauce, fruits were washed in lukewarm water. After washing, the fruits were left to drain in a sieve to create the laboratory samples whole fruit, washed and washings. The remaining part of the washed whole fruit was cut in small pieces using a knife. For steaming, the apple pieces were heated to 98–100 °C after addition of water to prevent enzymatic reactions as well as changes of colour and taste. After steaming for about 20 minutes, the pulp was passed through a strainer to obtain the laboratory samples raw sauce and strain rest. The remaining raw sauce was mixed with sugar and filled into preserving cans. The canned sauce was pasteurised in an autoclave. After pasteurisation, the preserving cans were emptied in a larger vessel and the sauce was mixed thoroughly to obtain the laboratory sample apple sauce.

For processing into dried fruit, lightly defrosted apples were peeled with a knife. An aliquot of the obtained laboratory sample of fruit, peeled and the whole sample of peel were taken. From the remaining sample of peeled fruit the apple cores were removed. The prepared fruits were deep frozen for further processing to prevent the loss of fruit water. Subsequently the deep-frozen fruits were cut into 5–7 mm slices. To prevent enzymatic reactions, oxidation and to maintain the vitamins, a treatment with sulphite solution and citric acid solution followed. The treated apple slices were thoroughly washed in standing lukewarm water. The washed apple slices were dried a fan-assisted oven at 80 °C until a water content of 10–30% was reached to obtain the laboratory sample fruit, dried.

Residues of flupyradifurone and metabolites in apple RAC and processed commodities were quantitated using LC/MS/MS Method 01304. Acceptable concurrent recovery data for apple commodities were obtained for each analyte.

Table 147 Residues in apple processed fractions from the foliar application of flupyradifurone to apples in Europe (Schulte and Bauer 2012c, 10-3171 and Schulte and Ballmann 2012a, 10-3172)

Trial No.,	Sample	DALA	Residues as parent (mg/kg)	Processing Factor

Location, Year (Crop, Variety)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6CNA	Parent	Parent + DFA + 6-CNA
GAP, USA, Pome fruits		14							
10-2171-03 (field/RAC),	Fruit (RAC)	14	0.07	< 0.02	< 0.01	< 0.01	0.09	NA	NA
10-3171-03 (processing),	Whole fruit, washed	14	0.08	< 0.02	< 0.01	< 0.01	0.10	1.1	1.1
Zaschwitz,	Washings	14	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.14	< 0.33
Germany, 2010	Raw sauce	14	0.07	< 0.02	< 0.01	< 0.01	0.09	1.0	1.0
(Apple, Jonagold)	Strain rest	14	0.10	< 0.02	< 0.01	< 0.01	0.12	1.4	1.3
	Sauce	14	0.05	< 0.02	< 0.01	< 0.01	0.07	0.71	0.78
2 applications	Pomace, wet	14	0.12	< 0.02	< 0.01	< 0.01	0.14	1.7	1.6
14 day RTI	Pomace, dried	14	0.34	< 0.02	< 0.01	< 0.01	0.36	4.9	4.0
Growth Stage 79 and 81	Raw juice	14	0.04	< 0.02	< 0.01	< 0.01	0.06	0.57	0.67
150 (×2) g ai/ha	Retentate	14	0.03	< 0.02	< 0.01	< 0.01	0.05	0.43	0.56
1000 (×2) L/ha	Juice	14	0.03	< 0.02	< 0.01	< 0.01	0.05	0.43	0.56
	Peel	14	0.38	0.02	< 0.01	< 0.01	0.40	5.4	4.4
	Fruit, peeled	14	0.03	< 0.02	< 0.01	< 0.01	0.05	0.43	0.56
	Fruit, dried	14	0.14	0.03	< 0.01	< 0.01	0.17	2.0	1.9
10-2171-06 (field/RAC),	Fruit (RAC)	14	0.09	< 0.02	< 0.01	< 0.01	0.11	NA	NA
10-3171-06 (processing),	Raw sauce	14	0.09	< 0.02	< 0.01	< 0.01	0.11	1.0	1.0
Fleurs,	Strain rest	14	0.16	< 0.02	< 0.01	< 0.01	0.18	1.8	1.6
Belgium, 2010	Sauce	14	0.07	< 0.02	< 0.01	< 0.01	0.09	0.78	0.82
(Apple, Elstar)	Whole fruit, washed	14	0.13	< 0.02	< 0.01	< 0.01	0.15	1.4	1.4
	Washings	14	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.11	< 0.27
2 applications	Pomace, wet	14	0.13	< 0.02	< 0.01	< 0.01	0.15	1.4	1.4
13 day RTI	Pomace, dried	14	0.41	0.02	< 0.01	< 0.01	0.43	4.6	3.9
Growth Stage 81 and 85	Raw juice	14	0.09	< 0.02	< 0.01	< 0.01	0.11	1.0	1.0
169 (×2) g ai/ha	Retentate	14	0.07	< 0.02	< 0.01	< 0.01	0.09	0.78	0.82
1120 (×2) L/ha	Juice	14	0.06	< 0.02	< 0.01	< 0.01	0.08	0.67	0.73
10-2172-03 (field/RAC),	Fruit (RAC)	14	0.04	< 0.02	< 0.01	< 0.01	0.06	NA	NA
10-3172-03 (processing),	Whole fruit, washed	14	0.03	0.02	< 0.01	< 0.01	0.05	0.75	0.83
San Martino,	Washings	14	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.25	< 0.50
Italy, 2010	Raw sauce	14	0.04	0.02	< 0.01	< 0.01	0.06	1.0	1.0
(Apple, Fuji)	Strain rest	14	0.05	0.02	< 0.01	< 0.01	0.07	1.3	1.2
	Sauce	14	0.03	< 0.02	< 0.01	< 0.01	0.05	0.75	0.83
2 applications	Pomace, wet	14	0.06	< 0.02	< 0.01	< 0.01	0.08	1.5	1.3
14 day RTI	Pomace, dried	14	0.20	0.03	< 0.01	< 0.01	0.23	5.0	3.8
Growth Stage 81 and 85	Raw juice	14	0.02	< 0.02	< 0.01	< 0.01	0.04	0.50	0.67
150 (×2) g ai/ha	Retentate	14	0.02	< 0.02	< 0.01	< 0.01	0.04	0.50	0.67
1000 (×2) L/ha	Juice	14	0.01	< 0.02	< 0.01	< 0.01	0.03	0.25	0.50
10-2172-06 (field/RAC),	Fruit (RAC)	14	0.10	0.04	< 0.01	< 0.01	0.14	NA	NA

Trial No.,	Sample	DALA	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Crop, Variety)							+ 6CNA		+ 6-CNA
10-3172-06 (processing),	Whole fruit, washed	14	0.11	0.04	< 0.01	< 0.01	0.15	1.1	1.1
Caldes de Malavella,	Washings	14	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.10	< 0.21
Spain, 2010	Raw sauce	14	0.09	0.04	< 0.01	< 0.01	0.13	0.90	0.93
(Apple, Galaxia)	Strain rest	14	0.17	0.03	< 0.01	< 0.01	0.20	1.7	1.4
	Sauce	14	0.07	0.03	< 0.01	< 0.01	0.10	0.70	0.71
2 applications	Pomace, wet	14	0.13	0.03	< 0.01	< 0.01	0.16	1.3	1.1
13 day RTI	Pomace, dried	14	0.49	0.09	< 0.01	< 0.01	0.58	4.9	4.1
Growth Stage 78 and 81	Raw juice	14	0.08	0.04	< 0.01	< 0.01	0.12	0.80	0.86
188 (×2) g ai/ha	Retentate	14	0.07	0.04	< 0.01	< 0.01	0.11	0.70	0.79
1250 (×2) L/ha	Juice	14	0.06	0.03	< 0.01	< 0.01	0.09	0.60	0.64

LOQ of parent, DFEAF and 6-CNA 0.01, DFA 0.02 mg/kg (parent equivs.) for all processed commodities

Grapes

The effect of processing (laboratory scale) on residues of flupyradifurone in grapes (Table 148) was investigated in four trials carried out in Germany in the 2010 growing season (Noss, Bauer and Ruhl 2011, 10-3406).

Grapes with incurred residues were obtained after one spray at approximately 300 g ai/ha. Grape bulk RAC samples were harvested 14 days after the last application from each of the control and treated plots. Grape processing was designed to simulate industrial procedures for the processing of bunches of grapes into juice and wine as well as jelly and raisins.

For juice preparation, unwashed grapes were crushed in a grape mill (crusher) and pressed to extract the liquid. A specimen of pomace, grape was taken. After clarification, a specimen of must was taken. Subsequently the must (raw juice) was pasteurised (at 83–87 °C for approximately 2 minutes). A specimen of juice, pasteurised was taken.

For vinification, pure culture yeast and nutrient salt were added to the raw juice to start the fermentation. After termination of the fermentation and clarification, $K_2S_2O_5$ (SO_2) was added and the intermediate was transferred into new vessels, leaving the sediment in the original vessel. After the first transfer (racking), bentonite was added to absorb the proteins. Upon completion of the clarification, the second transfer of wine was carried out and $K_2S_2O_5$ (SO_2) was added. The young wine was filtered through filter pads after a resting time (clarification). Wine at bottling (young wine) was sampled. A portion of the remaining young wine was bottled for maturation and taken as an intermediate sample for wine at first taste test (white wine). The intermediate was stored at 5–12 °C for approximately 6.5 months. After maturation time, wine at first taste test (white wine) was sampled for analysis.

To produce jelly, raw juice and gelling sugar were mixed and heated. After approximately 4 minutes cooking time, lemon juice was added and mixed with the cooked product. After cooling, specimens of jelly were taken.

For raisin production bunches of grapes were manually washed in tap water for three minutes. The ratio of water to fruit was approximately 1:1. The water covered the fruit. A specimen of washings was taken. Washed grape bunches were briefly put in boiling water and afterwards were manually washed (3 minutes) with cold tap water in a vessel. The ratio of water to fruit was again approximately 1:1. The water covered the bunches of grapes. The bunches were then dried in an oven (approximately 66–74 °C) for approximately 24 hours, until a moisture content of 10–14% was achieved. After drying, raisins were manually removed from the stalks. Specimens of <u>raisin</u> and <u>raisin waste</u> (stalks) were sampled.

Residues of flupyradifurone and metabolites in grape RAC and processed commodities were quantitated by LC/MS/MS using Method 01304. Acceptable recovery data for grape commodities were obtained for each analyte.

Table 148 Residues in grape processed fractions from the foliar application of flupyradifurone to grapes in Germany (Noss, Bauer and Ruhl 2011, 10-3406)

Trial No.,	Sample	DAT	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6CNA		+ 6CNA
GAP, USA, Grape		0							
10-3406-01,	Bunch of grapes (RAC)	14	0.36	0.03	< 0.01	0.01	0.40	NA	NA
Traustadt,	Berry	14	0.34	0.02	0.01	< 0.01	0.36	0.94	0.90
Germany, 2010	Pomace, grape	14	0.69	< 0.02	< 0.01	0.018	0.73	1.9	1.8
(Red-Blauer Spaetburgunder)	Must	14	0.31	0.03	< 0.01	0.011	0.35	0.86	0.88
1 application	Juice, pasteurised	14	0.31	0.03	< 0.01	0.011	0.35	0.86	0.88
Growth Stage 85	Wine at bottling	14	0.24	0.03	< 0.01	0.01	0.28	0.67	0.70
300 g ai/ha	Wine at first taste test	14	0.18	0.02	< 0.01	< 0.01	0.20	0.50	0.50
800 L/ha	Jelly	14	0.13	< 0.02	< 0.01	< 0.01	0.15	0.36	0.38
10-3406-02,	Bunch of grapes (RAC)	14	0.38	< 0.02	< 0.01	< 0.01	0.40	NA	NA
Windischenbach,	Berry	14	0.45	< 0.02	< 0.01	< 0.01	0.47	1.2	1.2
Germany, 2010	Pomace, grape	14	0.68	< 0.02	< 0.01	< 0.01	0.70	1.8	1.8
(Red-Blauer Spaetburgunder)	Must	14	0.32	< 0.02	< 0.01	< 0.01	0.34	0.84	0.85
1 application, Growth Stage 85	Wine at bottling	14	0.29	< 0.02	< 0.01	< 0.01	0.31	0.76	0.78
300 g ai/ha, 800 L/ha	Wine at first taste test	14	0.20	< 0.02	< 0.01	< 0.01	0.22	0.53	0.55
10-3406-03,	Bunch of grapes (RAC)	14	0.63	0.03	< 0.01	< 0.01	0.66	NA	NA
Radebeul,	Berry	14	0.52	0.03	< 0.01	< 0.01	0.55	0.83	0.83
Germany, 2010	Pomace, grape	14	1.0	0.02	< 0.01	0.013	1.0	1.6	1.5
(White-Riesling)	Must	14	0.32	0.04	< 0.01	< 0.01	0.36	0.51	0.55
	Juice, pasteurised	14	0.29	0.04	< 0.01	< 0.01	0.33	0.46	0.50
1 application	Wine at bottling	14	0.17	0.03	< 0.01	< 0.01	0.20	0.27	0.30
Growth Stage 81	Wine at first taste test	14	0.12	0.03	< 0.01	< 0.01	0.15	0.19	0.23
300 g ai/ha	Jelly	14	0.12	< 0.02	< 0.01	< 0.01	0.14	0.19	0.21
800 L/ha	Washings	14	0.03	< 0.02	< 0.01	< 0.01	0.05	0.048	0.076
	Raisin waste	14	1.5	0.07	< 0.01	0.037	1.6	2.4	2.4
	Raisin	14	1.3	0.07	< 0.01	0.037	1.4	2.1	2.2
10-3406-04,	Bunch of grapes (RAC)	14	0.45	< 0.02	< 0.01	< 0.01	0.47	NA	NA
Hoehnstedt,	Berry	14	0.20	< 0.02	< 0.01	< 0.01	0.22	0.44	0.47
Germany, 2010	Pomace, grape	14	0.78	< 0.02	< 0.01	< 0.01	0.80	1.7	1.7

Trial No.,	Sample	DAT	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6CNA		+ 6CNA
(White-Mueller-Thurgau)	Must	14	0.19	< 0.02	< 0.01	< 0.01	0.21	0.42	0.45
	Wine at bottling	14	0.19	< 0.02	< 0.01	< 0.01	0.21	0.42	0.45
1 application	Wine at first taste test	14	0.13	< 0.02	< 0.01	< 0.01	0.15	0.29	0.33
Growth Stage 83	Washings	14	0.03	< 0.02	< 0.01	< 0.01	0.05	0.067	0.11
300 g ai/ha	Raisin waste	14	1.2	0.05	< 0.01	0.015	1.3	2.7	2.8
800 L/ha	Raisin	14	1.3	0.03	< 0.01	< 0.01	1.3	2.9	2.8

LOQ of parent, DFEAF and 6-CNA 0.01, DFA 0.02 mg/kg (parent equivs.) for all processed commodities

Cucumber

The effect of processing (laboratory scale) on residues of flupyradifurone in cucumbers (Table 149) was investigated in two trials conducted in France during the 2010 growing season (Schulte and Ruhl 2012a, 10-3184).

Cucumbers with incurred residues were obtained where plants were sprayed with two foliar sprays at 125 g ai/ha. Cucumber bulk RAC samples were harvested 2 or 3 days after the last application. Bulk cucumber samples were processed into processed cucumber commodity samples per simulated commercial procedures.

A portion of the cucumber fruit was soaked and washed by brushing to give whole fruit, washed and washings. Cucumber fruits were scratched to allow better absorption of brine (vinegar, salt, sugar and spices) and to avoid bursting the fruit. Pasteurisation and homogenisation in a blender gave preserve.

Another portion of fruit was used to make fermented cucumber. As described above, a portion of the cucumber fruit was soaked and washed by brushing to give whole fruit, washed and washings. Brine was filled into the pickling jar followed by a layer of herbs. Cucumbers were filled alternating with herbs into the pickling jar. More brine was added and the jar closed. The jar was stored at 21–23 °C for 27–30 days with control of pH monitored. Fermentation was finalised when a constant acid value was reached and the cucumbers became glassy inside. Brine and fruit, fermented were obtained.

Residues of flupyradifurone and metabolites in cucumber RAC and processed commodities were quantitated by LC/MS/MS using Method 01304. Acceptable concurrent recovery data for cucumber commodities were obtained for each analyte.

Table 149 Residues in cucumber processed fractions from the foliar application of flupyradifurone to cucumber in France (Schulte and Ruhl 2012a, 10-3184)

Trial No.,	Sample	DALA	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6-CNA		+ 6-CNA
GAP, USA, Cucumber		1							
10-2184-01 (field/RAC),	Fruit (RAC)	3	0.06	0.09	< 0.01	< 0.01	0.15	NA	NA
10-3184-01	Washings	3	< 0.0	< 0.0	< 0.01	< 0.01	< 0.03	< 0.17	< 0.2

Trial No.,	Sample	DAL A	Residues as parent (mg/kg)					Processing Factor	
Location,			Paren t	DFA	DFEA F	6- CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6- CNA		+ 6- CNA
(processing),			1	2					
Toulouse,	Whole fruit, washed	3	0.09	0.14	< 0.01	0.013	0.23	1.5	1.5
France, 2010	Preserve	3	0.03	0.07	< 0.01	< 0.01	0.10	0.50	0.67
(Miranda-Gherkin)	Fruit, fermented	3	0.02	0.05	< 0.01	< 0.01	0.07	0.33	0.47
2 applications	Brine	3	0.02	0.04	< 0.01	< 0.01	0.06	0.33	0.40
14 day RTI									
Growth Stages 76 and 88									
125 (×2) g ai/ha									
500 (×2) L/ha									
10-2184-04 (field/RAC),	Fruit (RAC)	2	0.03	0.06	< 0.01	< 0.01	0.09	NA	NA
10-3184-04 (processing),	Washings	2	< 0.0 1	< 0.0 2	< 0.01	< 0.01	< 0.03	< 0.33	< 0.33
Chazay d'Azergues	Whole fruit, washed	2	0.02	0.09	< 0.01	< 0.01	0.11	0.67	1.2
France, 2010	Preserve	2	0.02	0.06	< 0.01	< 0.01	0.08	0.67	0.89
(Vert petit de Paris)	Fruit, fermented	2	0.01	0.04	< 0.01	< 0.01	0.05	0.33	0.56
2 applications	Brine	2	0.01	0.03	< 0.01	< 0.01	0.04	0.33	0.44
14 day RTI									
Growth Stages 72 and 79									
125 (×2) g ai/ha									
550 (×2) L/ha									

LOQ of parent, DFEAF and 6-CNA 0.01, DFA 0.02 mg/kg (parent equivs.) for all processed commodities

Tomato

The effect of processing (laboratory scale) on residues of flupyradifurone in tomatoes (Table 148) was investigated in four trials carried out during the 2010 growing season in France, Italy, Spain and Portugal (Schulte and Bauer 2012b, 10-3186).

Tomatoes with incurred residues were obtained where plants were sprayed with two foliar sprays of BYI 02960 SL 200 at approximately 125 g ai/ha. Tomato bulk RAC samples were harvested 3 days after the last application. The washing and peeling of tomatoes was done using household practices. The preparation of juice, preserve, puree, paste and dried fruit simulated industrial practice at a laboratory scale.

The process was started in the deep-frozen state. The tomatoes were washed in lukewarm standing water and drained. An aliquot of each laboratory sample of washings and whole fruit, washed was taken. The remaining washed tomatoes were cut into small pieces. The cut tomatoes were heated, after addition of water, to 80–100 °C to prevent enzymatic reactions. The tomato pulp was then passed through a strainer to separate raw juce and strain rest. Sodium chloride [0.5–0.7% (w/w)] was added to the raw juice. An aliquot of raw juice was taken. One part of the remaining raw juice was used for processing into preserves. Another portion was filled into preserving cans and pasteurised. After pasteurisation, an aliquot of the laboratory sample juice was taken.

An aliquot of the raw juice was concentrated while stirring to obtain tomato raw puree (water content 12–20%). The remaining raw puree was filled into preserving cans and pasteurised. After pasteurisation, tomato puree was obtained. For the preparation of raw paste an aliquot of the raw juice was concentrated while stirring to obtain tomato raw puree (water content 24–29%). The tomato raw paste was filled into preserving cans and pasteurised. After pasteurisation, the obtained laboratory sample of tomato paste was taken.

Deep-frozen tomatoes were washed in warm standing water. After a few minutes the peel could be removed with a knife. Aliquots of the laboratory samples peel, peeling water and fruit, peeled were taken. After addition of raw juice a part of the remaining peeled tomatoes was filled into preserving cans and pasteurised. After pasteurisation, the tomato preserves were minced with a hand mixer. An aliquot of the obtained laboratory sample of tomato preserve was taken.

Deep-frozen tomatoes were washed in luke-warm standing water. After washing the fruits were drained in a sieve. The washed tomatoes were cut with a knife in half. The cut tomatoes were put with the cut surface face up onto a baking tray, sprinkled with salt and dried in a fan-assisted oven at 90–95 °C for 9 hours giving fruit, dried.

Residues of flupyradifurone and metabolites in tomato RAC and processed commodities were quantitated by LC/MS/MS using Method 01304. Acceptable concurrent recovery data for tomato commodities were obtained for each analyte.

Table 150 Residues in tomato processed fractions from the foliar application of flupyradifurone to tomatoes in Europe (Schulte and Bauer 2012, 10-3186)

Trial No.,	Sample	DAT	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6-CNA		+ 6-CNA
GAP, USA, Tomato		1							
10-2186-01 (field/RAC),	Fruit (RAC)	3	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	NA	NA
10-3186-01 (processing),	Whole fruit, washed	3	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	NC	NC
Boé,	Washings	3	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	NC	NC
France, 2010	Raw juice	3	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	NC	NC
(Perfect Peel—hybrid variety)	Juice	3	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	NC	NC
	Strain rest	3	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	NC	NC
2 applications	Raw puree	3	0.01	0.03	< 0.01	< 0.01	0.04	NC	NC
14 day RTI	Puree	3	< 0.01	0.02	< 0.01	< 0.01	0.03	NC	NC
Growth Stages 81 and 87	Paste	3	0.02	0.04	< 0.01	0.011	0.06	NC	NC
125 (×2) g ai/ha	Peel	3	0.02	< 0.02	< 0.01	< 0.01	0.04	NC	NC
600 (×2) L/ha	Peeling water	3	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	NC	NC
	Fruit, peeled	3	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	NC	NC
	Preserve	3	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	NC	NC
	Fruit, dried	3	0.02	0.05	< 0.01	0.018 c0.013	0.07	NC	NC
10-2186-02 (field/RAC),	Fruit (RAC)	3	0.05	< 0.02	< 0.01	< 0.01	0.07	NA	NA
10-3186-02 (processing),	Whole fruit, washed	3	0.05	< 0.02	< 0.01	< 0.01	0.07	1.0	1.0
Catania,	Washings	3	0.01	< 0.02	< 0.01	< 0.01	0.03	0.20	0.43
Italy, 2010	Raw juice	3	0.04	< 0.02	< 0.01	< 0.01	0.06	0.80	0.86
(Missouri—Multiple use variety)	Juice	3	0.03	< 0.02	< 0.01	< 0.01	0.05	0.60	0.71
	Strain rest	3	0.05	< 0.02	< 0.01	< 0.01	0.07	1.0	1.0
2 applications	Raw puree	3	0.08	0.03	< 0.01	0.016	0.13	1.6	1.9
14 day RTI	Puree	3	0.07	0.03	< 0.01	0.016	0.11	1.4	1.5
Growth Stage 73 and 88	Peel	3	0.13	< 0.02	< 0.01	< 0.01	0.15	2.6	2.1
125 (×2) g ai/ha	Peeling water	3	0.01	< 0.02	< 0.01	< 0.01	0.03	0.20	0.43
600 (×2) L/ha	Fruit, peeled	3	0.02	< 0.02	< 0.01	< 0.01	0.04	0.40	0.57
	Preserve	3	0.03	< 0.02	< 0.01	< 0.01	0.05	0.60	0.71
10-2186-03 (field/RAC),	Fruit (RAC)	3	0.08	< 0.02	< 0.01	0.011	0.11	NA	NA
10-3186-03 (processing),	Whole fruit, washed	3	0.09	< 0.02	< 0.01	< 0.01	0.11	1.1	1.0
Alginet,	Washings	3	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.13	< 0.27
Spain, 2010	Raw juice	3	0.04	< 0.02	< 0.01	< 0.01	0.06	0.50	0.55
(Malpica-Tomate de	Juice	3	0.04	< 0.02	< 0.01	< 0.01	0.06	0.50	0.55

Trial No.,	Sample	DAT	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6-CNA		+ 6-CNA
industria)									
	Strain rest	3	0.05	< 0.02	< 0.01	0.018	0.09	0.63	0.82
2 applications	Raw puree	3	0.11	0.04	< 0.01	0.018	0.17	1.4	1.5
14 day RTI	Puree	3	0.11	0.04	< 0.01	0.037	0.19	1.4	1.7
Growth Stage 81 and 83	Peel	3	0.17	< 0.02	< 0.01	< 0.01	0.19	2.1	1.7
125 (×2) g ai/ha	Peeling water	3	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.13	< 0.27
1000 (×2) L/ha	Fruit, peeled	3	0.04	< 0.02	< 0.01	< 0.01	0.06	0.50	0.55
	Preserve	3	0.03	< 0.02	< 0.01	0.013	0.06	0.38	0.55
10-2186-04 (field/RAC),	Fruit (RAC)	3	0.04	< 0.02	< 0.01	< 0.01	0.06	NA	NA
10-3186-04 (processing),	Whole fruit, washed	3	0.02	< 0.02	< 0.01	< 0.01	0.04	0.50	0.67
Salvaterra de Magos,	Washings	3	< 0.01	< 0.02	< 0.01	< 0.01	< 0.03	< 0.25	< 0.50
Portugal, 2010	Raw juice	3	0.02	< 0.02	< 0.01	< 0.01	0.04	0.50	0.67
(H9144-Industry)	Juice	3	0.02	< 0.02	< 0.01	< 0.01	0.04	0.50	0.67
	Strain rest	3	0.04	< 0.02	< 0.01	< 0.01	0.06	1.0	1.0
2 applications	Raw puree	3	0.06	< 0.02	< 0.01	0.01	0.09	1.5	1.5
14 day RTI	Puree	3	0.06	< 0.02	< 0.01	0.011	0.09	1.5	1.5
Growth Stage 85 and 88	Paste	3	0.08	< 0.02	< 0.01	0.016	0.116	2.0	1.9
125 (×2) g ai/ha	Peel	3	0.12	< 0.02	< 0.01	< 0.01	0.14	3.0	2.3
700 (×2) L/ha	Peeling water	3	0.01	< 0.02	< 0.01	< 0.01	0.03	0.25	0.50
	Fruit, peeled	3	0.02	< 0.02	< 0.01	< 0.01	0.04	0.50	0.67
	Preserve	3	0.03	< 0.02	< 0.01	< 0.01	0.05	0.75	0.83
	Fruit, dried	3	0.08	0.03	< 0.01	0.037	0.15	2.0	2.45

LOQ of parent, DFEAF and 6-CNA 0.01, DFA 0.02 mg/kg (parent equivs.) for all processed commodities

Note: No processing factors calculated for Trial 10-2186-01 as no residues were observed in the RAC sample

Soya bean

The effect of processing (laboratory scale) on residues of flupyradifurone in soya beans (Table 151) was investigated (Lenz 2012a, RARVY029). Two field trials were conducted in the USA during the 2010 growing season in which soya beans with incurred residues were obtained where plants were sprayed with two foliar sprays of BYI 02960 200SL. All applications were made using ground-based equipment. A non-ionic surfactant was used in all applications. Single composite samples of soya bean seed (RAC) were collected from treated and control plots at 19 or 21 days after the last application. Soya bean seed RAC was removed and after drying and aspiration was used to firstly generate aspirated grain fractions and the following processed commodities of meal, hulls and refined oil, soy milk and defatted flour. Processing was performed using procedures which simulated commercial processing practices.

Whole soya beans were cleaned by aspiration and screening. A portion of the cleaned soya bean sample was removed for soymilk production. Whole soya beans were then mechanically cracked and the hull material was then separated from the kernel. Kernel material was processed into crude oil, soya bean meal and soya bean flakes. The flakes were ground and sifted to produce defatted flour. Crude oil was sequentially converted to refined oil and soapstock, and the refined oil to bleached oil, deodorised oil and deodoriser distillates. Cleaned whole soya bean were soaked, ground, filtered and heated to give soymilk.

Residues of flupyradifurone and metabolites in soya bean RAC and processed commodities were quantitated by LC/MS/MS using Method 01304. Acceptable concurrent recovery data for soya bean commodities were obtained for each analyte.

Table 151 Residues in soya bean processed fractions from the foliar application of flupyradifurone to soya beans in the USA (Lenz 2012a, RARVY029)

Trial No.,	Sample	DAT	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6-CNA		+ 6-CNA
GAP, USA, Soya bean		21							
RV268-10PA,	Seed, dry (RAC)	21	0.695	0.052	0.058	0.207	0.954	NA	NA
York,	Aspirated grain fractions	21	11	0.428	0.232	0.554	12.0	16	12.5
Nebraska,	Meal	21	0.593	0.062	0.072	0.326	0.98	0.85	1.0
USA, 2010	Hull	21	0.544	0.054	0.056	0.154	0.75	0.78	0.79
(93Y12)	Oil, refined	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.014	< 0.063
	Milk	21	0.029	< 0.05	< 0.01	0.045	0.12	0.042	0.13
2 applications	Defatted flour	21	0.728	0.075	0.084	0.346	1.1	1.0	1.2
9 day RTI									
Growth Stages 79 and 89									
1000 and 998 g ai/ha									
187 (×2) L/ha									
RV269-10PA,	Seed, dry (RAC)	19	0.414	0.306	0.33	4.04	4.76	NA	NA
Springfield,	Aspirated grain fractions	19	4.84	1.47	0.560	1.76	8.0	12	1.7
Nebraska,	Meal	19	0.519	0.442	0.424	7.01	8.0	1.3	1.7
USA, 2010	Hull	19	0.485	0.240	0.297	2.71	3.4	1.2	0.72
(Channel 3051R)	Oil, refined	19	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.024	< 0.012
	Milk	19	0.037	< 0.05	0.022	1.25	1.3	0.089	0.28
2 applications	Defatted flour	19	0.494	0.39	0.381	8.14	9.0	1.2	1.9
8 day RTI									
Growth Stages 79 (×2)									
997 (×2) g ai/ha									
150 (×2) L/ha									

LOQ of parent 0.025, DFEAF, DFA and 6-CNA 0.05 mg/kg (parent equivs.) for aspirated grain fractions

LOQ of parent, DFEAF and 6-CNA 0.01, DFA 0.05 mg/kg (parent equivs.) for all other processed commodities

Potato

The effect of processing (laboratory scale) on residues of flupyradifurone in potatoes (Table 152) was investigated in two trials conducted in the USA during the 2011 growing season (Lenz 2012e, RARVY038). Potatoes with incurred residues were obtained where plants were sprayed with two foliar sprays at exaggerated rates (approximately 5×). All applications were made with ground equipment. A non-ionic surfactant was used in all applications. Single composite potato tuber samples were taken at 7 days after the last application from the treated and control plots. Potato samples were processed per simulated commercial procedures into the following samples: washed tuber, washings, crisps, flakes, wet peel, starch, cooked tuber with peel, peeled tuber, steamed and cooked tuber and cooking water.

A potato RAC sample was taken prior to cleaning. Potatoes were washed in a tub for 5 minutes, inspected by hand and if necessary, culled potatoes were disposed of. A representative sample of wash water and washed potatoes were taken. Aliquots of washed potatoes were removed for processing into potato chips and boiled potatoes and for cooking. An aliquot of washed potatoes was peeled in batches using a pressure steam peeler for 45 seconds, followed by scrubbing with a restaurant style peeler fitted with a rubber scrubber to remove the loosened skin. Water from the scrubber was collected for starch processing. The potatoes were inspected and hand trimmed to remove additional peel, rot, green or otherwise damaged potatoes. A representative peeled tuber sample was removed and the peel was hydraulically pressed then blended with the cut trim waste to form wet peel and trimmings. Starch water was collected from the pressed peel for starch removal.

For potato flake processing, a sample of peeled potatoes were cut into slabs. The slabs were spray washed with cold water to rinse off free surface starch and pre-cooked in a steam jacketed kettle at approximately 70–77 °C for 22 minutes. The slices were cooled to 32 °C using cold running tap water then steam cooked using an atmospherically flowing steam batch style steam cooker at 94–100 °C for 40 minutes. The slices were mashed and an emulsion containing additives was mixed into the remaining mash and the mixture was hand fed into a single drum drier where the mash was dried into a thin sheet. The sheet was broken by hand into large flakes which were then fed into a hammermill for uniform sizing of the finished flakes. If required, the flakes were dried on the fluidized bed dryer until a moisture content less than or equal to 9% was achieved. Water from potato scrubbing and wet peel pressing was reserved for potato starch processing. The starch water was filtered using a series of sieves to remove pieces of pulp and peel and then centrifuged to remove the water from the starch until the required amount of starch was obtained.

For potato chip (crisps) processing, an aliquot of washed potatoes was peeled. The peeled potatoes were inspected by hand and trimmed to remove rot, green or damaged potato tissue. An aliquot of the peeled potatoes was cut into thin slices which were placed into a tub of hot water to remove free starch, drained over a screen and then fried in oil at approximately 163–191 °C for approximately 90 seconds. The fried potatoes were drained, salted and inspected to remove undesirable chips. A sample of crisps was taken.

For unpeeled boiled (cooked) potatoes, an aliquot of unpeeled washed potatoes were cut into quarters and boiled in water until the internal temperature of the potatoes reached 88–92 °C. A representative sample of the cooking water was also taken.

Residues of flupyradifurone and metabolites in potato RAC and processed commodities were quantitated by LC/MS/MS using Method 01304. Acceptable concurrent recovery data for potato commodities were obtained for each analyte.

Table 152 Residues in potato processed fractions from the foliar application of flupyradifurone to potatoes in the USA (Lenz 2012e, RARVY038)

Trial No.,	Sample	DALA	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6CNA		+ 6-CNA
GAP, USA, Potato		7							
RV202-11PA,	Tuber (RAC)	7	0.01	< 0.05	< 0.01	< 0.01	0.060	NA	NA
St John,	Tuber, washed	7	0.012	< 0.05	< 0.01	< 0.01	0.062	1.2	1.0
Kansas	Washings	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 1.0	< 1.0
USA, 2011	Crisps (Am. chips)	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 1.0	< 1.0
(Red Pontiac)	Flakes	7	0.011	< 0.05	< 0.01	< 0.01	0.061	1.1	1.0
	Peel, wet	7	0.013	< 0.05	< 0.01	< 0.01	0.063	1.3	1.1
2 applications	Starch	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 1.0	< 1.0
6 day RTI	Tuber with peel, cooked	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 1.0	< 1.0
Growth Stages 47 and 48	Tuber, peeled	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 1.0	< 1.0
957 and 997 g ai/ha	Tuber, steamed, cooked	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 1.0	< 1.0
309 and 327 L/ha	Cooking water	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 1.0	< 1.0
RV203-11PA,	Tuber (RAC)	7	0.012	0.054	< 0.01	< 0.01	0.066	NA	NA
Ephrata,	Tuber, washed	7	0.014	0.052	< 0.01	0.018	0.084	1.2	1.28
Washington	Washings	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.83	< 0.91
USA, 2011	Crisps (Am. chips)	7	< 0.01	0.074	< 0.01	0.014	0.098	< 0.83	1.5
(Umatilla)	Flakes	7	0.013	0.099	< 0.01	0.025	0.14	1.1	2.1

Trial No.,	Sample	DALA	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6CNA		+ 6-CNA
	Peel, wet	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.83	< 0.091
2 applications	Starch	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.83	< 0.091
7 day RTI	Tuber with peel, cooked	7	< 0.01	< 0.05	< 0.01	0.014	0.074	< 0.83	1.1
Growth Stages 47 and 48	Tuber, peeled	7	< 0.01	0.057	< 0.01	0.014	0.081	< 0.83	1.2
1010 (×2) g ai/ha	Tuber, steamed, cooked	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.83	< 0.091
374 (×2) L/ha	Cooking water	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.83	< 0.091

LOQ of parent, DFEAF, 6-CNA 0.01, DFA 0.05 mg/kg (parent equivs.) for all matrices

Barley

The effect of processing (laboratory scale) on residues of flupyradifurone in barley (Table 153) was investigated in two trials conducted in Germany in the 2010 growing season (Schulte and Ruhl 2012b, 10-3410).

Barley with incurred residues was obtained where plants were sprayed once with BYI 02960 SL 200. Barley bulk RAC samples were harvested 20 or 22 days after the last application.

Barley grain samples were processed per simulated commercial procedures into the following samples: brewer's malt, malt sprouts, brewer's grain, hops draff, brewer's yeast, beer, pearl barley and pearl barley rub-off.

For processing the grain to malt, the field samples did not need to be dried before cleaning as the optimal moisture content of approximately 14% was reached at the sampling date. After cleaning, the grain was sieved (sieve mesh 2.5 mm) then transferred to a steeping vessel where it underwent a combined wet and dry steeping process. The final steeping degree was in the range of 44.2% to 44.4%. The samples then underwent "still" germination for 118 h. The steeped material was turned over continuously at 100% humidity and a mean temperature of 13.8–15.5 °C. After germination, kiln drying was conducted in a dry chamber at a maximum temperature of 80 °C. After kiln-drying the germs were removed mechanically by a trimmer. The brewer's malt and malt sprouts were sampled 2–4 days after malting. The malt was stored at room temperature until beer processing.

For beer processing, the malted barley was dry milled, mixed with brew water then mashed in a heatable tun by a series of heating and resting stages conducted between 46–76 °C. The resultant liquid had become wort and was removed from the brewer's grain using a refining vat (lautering). A sample of the spent grain was taken. After lautering was complete, hops were added to the wort and the mixture was boiled for 90 minutes at normal pressure. After boiling, the flocs (hops draff) were separated in a whirlpool and a sample was taken. The wort was cooled and ventilated using an intra-plant circulation. Pure culture yeast was added to the wort and classical primary fermentation (low fermentation) was carried out in bottom fermentation containers. When the extract content of the fermented young beer was 2% higher than the final attenuation, the storing time began. Samples of brewer's yeast were taken. At the beginning of maturation, the young beer was stored at room temperature in casks. The young beer was then stored under pressure (approximately 0.8–1.8 bar) at approximately 0–2 °C (cold maturation) for about 3–4 weeks. The rack beer was filtered to remove harmful organisms and sludge particles. The final beer product was sampled.

For pearl barley production, it was not necessary to dry or dampen the grain as an optimal moisture content of 15% had been achieved. The spring barley was hulled using a vertical hulling machine until the stipulated abrasion for pearl barley of 30–35% was reached. Pearl barley and pearl barley rub off were sampled.

Residues of flupyradifurone and metabolites in barley RAC and processed commodities were quantitated by LC/MS/MS using Method 01304. Acceptable concurrent recovery data for barley commodities were obtained for each analyte.

Table 153 Residues in barley processed fractions from the foliar application of flupyradifurone to barley in Germany (Schulte and Ruhl 2012b, 10-3410)

Trial No.,	Sample	DAT	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6-CNA		+ 6-CNA
GAP, USA, Barley		21							
10-3410-01,	Grain (RAC)	22	0.84	0.04	0.03	0.018	0.90	NA	NA
Motterwitz,	Malt sprouts	22	0.32	0.49 c0.04	0.16	0.011	0.82	0.38	0.91
Germany, 2010	Brewer's malt	22	0.37	0.04	0.04	< 0.01	0.41	0.44	0.46
(Laverda–spring barley)	Brewer's grain	22	0.07	< 0.02	< 0.01	< 0.01	0.09	0.083	0.10
	Hops draff	22	0.16	< 0.2	< 0.1	< 0.1	0.46	0.19	0.51
1 application	Brewer's yeast	22	0.06	< 0.02	< 0.01	0.037	0.12	0.071	0.13
Growth Stage 83	Beer	22	0.06	< 0.02	< 0.01	< 0.01	0.08	0.071	0.089
460 g ai/ha	Pearl Barley	22	0.12	0.03	0.03	< 0.01	0.15	0.14	0.17
300 L/ha	Pearl barley rub-off	22	2.8	0.08	0.05	0.055	2.94	3.3	3.3
10-3410-02,	Grain (RAC)	20	1.6	0.03	0.02	0.037	1.64	NA	NA
Trossin,	Malt sprouts	20	0.66	0.36	0.14	0.092	1.1	0.41	0.67
Germany, 2010	Brewer's malt	20	0.83	0.02	0.03	< 0.01	0.85	0.52	0.52
(Tocada—spring barley)	Brewer's grain	20	0.04	< 0.02	< 0.01	< 0.01	0.06	0.025	0.037
	Hops draff	20	0.29	< 0.2	< 0.1	< 0.1	0.59	0.18	0.36
1 application	Brewer's yeast	20	0.17	< 0.02	< 0.01	0.018	0.21	0.11	0.13
Growth Stage 85–87	Beer	20	0.08	< 0.02	< 0.01	< 0.01	0.10	0.05	0.061
460 g ai/ha	Pearl Barley	20	0.10	< 0.02	< 0.01	< 0.01	0.12	0.063	0.073
300 L/ha	Pearl barley rub-off	20	4.0	0.05	0.04	0.073	4.12	2.5	2.55

LOQ of parent, DFEAF, 6-CNA 0.01, DFA 0.02 mg/kg (parent equivs.) for all matrices except hops draff

LOQ of parent, DFEAF, 6-CNA 0.1, DFA 0.2 mg/kg (parent equivs.) for hops draff

Wheat

The effect of processing (laboratory scale) on residues of flupyradifurone in wheat (Table 154) was investigated in two trials conducted in Germany in the 2010 growing season (Schulte and Bauer 2012e, 10-3409). Wheat with incurred residues was obtained where plants were sprayed once with BYI 02960 SL 200. Wheat bulk RAC samples were harvested 22 days after the last application.

Wheat grain samples were processed per simulated commercial procedures into the following samples: semolina, semolina bran, white flour bran, white flour, white bread, wholemeal, wholemeal bread and wheat germ.

Frozen field samples were defrosted, dried for 12 hours at 30 °C and then cleaned. If the moisture content of the grain was > 15–16%, the samples were dried for a further 9 hours in a drying cupboard at 30 °C. If the moisture content of the grain was < 15–16%, the samples were moistened with tap water. The final moisture content achieved in the grain was between 15.5–15.6%.

Wheat grain was milled to straight flour, semolina and semolina bran in a closed system with different pairs of smooth rollers and sifter passages. The low-grade meal (toppings) were separated from the semolina bran using a centrifuge/scouring machine. The process resulted in white flour bran and low-grade meal (toppings). After determination of the mineral content of the straight flour and low-grade meal, both fractions were mixed to the final product white flour type 550 until a mineral content of 510–630 g/100 kg flour was reached. Mixing was not required if the mineral content was already in the range of 510–630 g/100 kg. A 1.0 kg loaf of white bread was made by mixing together white flour, yeast, salt and water. The dough was needed, fermented twice and baked into bread at 220 °C for 30 minutes.

Whole meal was generated by the same milling process used to produce white flour type 550. After milling the total bran was cracked with an impact mill into smaller pieces. All milling products were mixed in a flour mixer to form the whole meal. A 1.0 kg loaf of wholemeal bread was made by mixing together whole meal, yeast, salt and water. The dough was kneaded, fermented twice and baked into bread at 220 °C for 30 minutes.

Wheat germ was produced by bruising the grain in a mill (roller mill with 0.5 mm roller distance). The fraction between 400–1000 µm was collected and the fraction > 1000 µm was broken once more (0.3 mm roller distance). The milling/sieving process was performed three times in total with a final roller distance of 0.2 mm. Any fractions < 400 µm and > 1000 µm was excluded from further processing. The fraction between 400–1000 µm was a mixture of bran, semolina and germ. The bran was separated from the semolina and germ using a separator. The semolina/germ mixture was milled to flour and small wheat germ discs (including parts of bran) in a mill with a pair of smooth rollers. The wheat germ with parts of bran was sieved to separate the germ with small parts of bran from the bran. From the separated germ discs, the small parts of bran were removed manually to form the final wheat germ product.

Residues of flupyradifurone and metabolites in wheat RAC and processed commodities were quantitated by LC/MS/MS using Method 01304. Acceptable concurrent recovery data for wheat commodities were obtained for each analyte.

Table 154 Residues in wheat processed fractions from the foliar application of flupyradifurone to wheat in Germany (Schulte and Bauer 2012e, 10-3409)

Trial No.,	Sample	DAT	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6-CNA		+ 6-CNA
GAP, USA, Wheat		21							
10-3409-01,	Grain (RAC)	22	0.23	0.02	< 0.01	< 0.01	0.25	NA	NA
Motterwitz,	Semolina bran	22	1.1	0.5	< 0.01	0.18	1.78	4.8	7.1
Germany, 2010	Semolina	22	0.32	0.03	< 0.01	< 0.01	0.35	1.4	1.4
(Cubus-winter wheat)	White flour bran	22	1.5	0.52	< 0.01	0.22	2.22	6.5	8.9
	White flour	22	0.07	0.03	< 0.01	< 0.01	0.10	0.30	0.40
1 application	White bread	22	0.05	0.02	< 0.01	< 0.01	0.07	0.22	0.28
Growth Stage 83	Whole meal	22	0.36	0.03	< 0.01	< 0.01	0.39	1.6	1.6
460 g ai/ha	Whole meal bread	22	0.22	< 0.02	< 0.01	< 0.01	0.24	0.96	0.96
300 L/ha	Wheat germ	22	0.30	0.03	< 0.01	< 0.01	0.33	1.3	1.3
10-3409-02,	Grain (RAC)	22	0.14	< 0.02	< 0.01	< 0.01	0.16	NA	NA
Machern,	Semolina bran	22	0.61	0.02	< 0.01	0.018	0.648	4.4	4.05
Germany, 2010	Semolina	22	0.06	< 0.02	< 0.01	< 0.01	0.08	0.43	0.50
(Toras-winter wheat)	White flour bran	22	0.81	0.03	< 0.01	< 0.01	0.84	5.8	5.3
	White flour	22	0.02	< 0.02	< 0.01	< 0.01	0.04	0.14	0.25
1 application	White bread	22	0.02	< 0.02	< 0.01	< 0.01	0.04	0.14	0.25
Growth Stage 85	Whole meal	22	0.21	< 0.02	< 0.01	< 0.01	0.23	1.5	1.4
460 g ai/ha	Whole meal bread	22	0.12	< 0.02	< 0.01	< 0.01	0.14	0.86	0.88
300 L/ha	Wheat germ	22	0.11	< 0.02	< 0.01	< 0.01	0.13	0.79	0.81

LOQ of parent, DFEAF, 6-CNA 0.01, DFA 0.02 mg/kg (parent equivs.) for all matrices

The effect of processing (laboratory scale) on residues of flupyradifurone in wheat (Table 155) was also investigated in two trials conducted in USA in the 2010 growing season (Lenz and Fischer 2012, RARVY031). Wheat with incurred residues was obtained where plants were sprayed twice with BYI 02960 SL 200. All applications were made using ground-based equipment. A non-ionic surfactant was used in all applications at 0.25% v/v. Single composite wheat grain RAC samples were harvested 19 or 21 days after the last application.

Wheat grain samples were processed per simulated commercial procedures into the following samples: aspirated grain fractions, germ, bran, middlings, shorts, flour, white bread, whole meal, whole meal bread, starch, gluten, fresh pasta, fresh cooked pasta, dried pasta, dried and cooked pasta and cooking water from both cooked processes.

Unprocessed grain (RAC) samples were taken prior to processing. Drying and aspiration was used to generate aspirated grain fractions.

Following generation of aspirated grain fractions, the whole wheat was cleaned by aspiration and screening. The cleaned wheat was separated into three process lines: germ recovery, whole meal flour and milling for bran, flour and shorts. Cleaned wheat was moisture adjusted (tempered) to 16%. After tempering for 1–1.5 hours the wheat was passed through a disc mill. Ground material was sifted. Material on top of the sieve was aspirated to remove bran from the germ fraction. Germ (with endosperm) was passed through a reduction mill. Germ and reduced endosperm were sifted to separate the germ from the endosperm. Germ material was also aspirated again to remove additional bran and milled/sieved to remove additional endosperm. A 0.5–1.0% recovery of germ was expected.

Cleaned wheat was moisture conditioned to 16.5% and allowed to temper for 24 hours. Tempered wheat was fed through a wheat mill. Breaking of the wheat was accomplished by three rolls. The material was then fed onto the break sifter screen which comprised two sizes [120 mesh (140 micron) and 25 mesh (800 micron)]. Material exiting the break rolls passed over the 120 screen first. Material passing through the 120 screen is “break flour”. Material not passing through was conveyed over the 25 screen. Material passing through the 25 screen is middlings. Material not passing through was conveyed to the other end of the sifter and is bran.

After sampling the middlings, the remainder was fed into the reduction side of the mill. Reduction was achieved through two rolls. After passing through the reduction rolls, the material was fed onto the reduction sifter screen. This screen is 100 mesh (160 micron). Material passing through is reduction flour. Material not passing through and conveyed to the end of the sifter is shorts.

Bran from the break sieve was fed into the reductions side screen of the mill. It was not reduced with the rollers. Bran was conveyed by beater bars over the 100 mesh screen. Material passing through the screen was shorts and was added to shorts produced from the middlings. Material passing over the screen and exiting the end was bran.

A portion of the break and reduction flours were mixed to white flour (straight flour). This flour was used to produce white bread.

Cleaned wheat was ground in a pin mill. Ground material was whole meal flour which was used to produce whole meal bread.

For gluten and starch, break flour was mixed with water. After mixing, dough was rested for two hours. Dough was kneaded as water washed away from the starch, leaving the gluten. Product was water washed until water ran clear, indicating all starch was removed, leaving gluten. Starch and water were separated using centrifugation to separate out the water. Starch was dried in an oven until the moisture content was 15% or less. Dried starch was then ground and collected. Gluten was dried using an oven or drum dryer, then ground and collected.

Equal parts of break and reduction flour were mixed with water and salt to form a dough. This was kneaded and rested and fed through a pasta machine to produce a fresh pasta. A portion was cooked to produce a fresh cooked pasta fraction.

Remaining extruded noodles were dried for eight hours to produce dried pasta. A portion of the dried noodles were removed and collected. Remaining dried noodles were then cooked in boiling water, removed and allowed to cool. After cooling a fraction of dried, cooked pasta was collected. Fractions of the cooking water were collected from both fresh and dried noodles.

Residues of flupyradifurone and metabolites in wheat RAC and processed commodities were quantitated by LC/MS/MS using Method 01304. Acceptable concurrent recovery data for wheat commodities were obtained for each analyte.

Table 155 Residues in wheat processed fractions from the foliar application of flupyradifurone to wheat in the USA (Lenz and Fischer 2012, RARVY031)

Trial No.	Sample	DALA	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6-CNA		+ 6-CNA
GAP, USA, Wheat		21							
RV272-10PA,	Grain (RAC)	21	1.66	2.18	0.022	0.068	3.868	NA	NA
Carlyle,	Aspirated grain fractions	21	22.9 c0.024	19.1	0.21	1.4	43.4	14	11
Illinois	Bran	21	3.95	2.2	0.023	0.11	6.26	2.4	1.6
USA, 2010	Cooking water	21	0.011	0.083	< 0.01	< 0.01	0.094	0.007	0.024
(Branson)	Cooking water 2	21	0.011	0.099	< 0.01	< 0.01	0.11	0.007	0.028
	Germ	21	2.73	2.17	0.029	0.019	4.92	1.6	1.3
2 applications	Gluten	21	0.358	0.40	< 0.01	0.011	0.77	0.22	0.20
7 day RTI	Middlings	21	1.32	1.91	0.021	0.041	3.24	0.80	0.84
Growth Stages 71 and 75	Pasta, cooked	21	0.103	0.367	< 0.01	< 0.01	0.47	0.062	0.12
989 and 1010 g ai/ha	Pasta, dried and cooked	21	0.144	0.529	< 0.01	< 0.01	0.67	0.087	0.17
160 and 240 L/ha	Pasta, dry	21	0.398	1.84	0.015	0.014	2.21	0.24	0.57
	Pasta, fresh	21	0.306	1.43	0.01	0.01	1.71	0.18	0.44
	Shorts	21	2.05	2.08	0.017	0.05	4.15	1.2	1.1
	Starch	21	0.019	0.106	< 0.01	< 0.01	0.13	0.011	0.033
	White bread	21	0.236	1.15	< 0.01	< 0.01	1.4	0.14	0.36
	White flour	21	0.356	1.58	0.013	0.013	1.91	0.21	0.49
	Whole meal	21	2.1	2.04	0.022	0.060	4.1	1.3	1.1
	Wholemeal bread	21	1.25	1.45	0.017	0.048	2.75	0.75	0.71
RV273-10PA,	Grain (RAC)	19	1.29	2.93	0.034	0.16	4.36	NA	NA
Syracuse,	Aspirated grain fractions	19	27.7 c0.171	14.7 c0.281	0.19	2.0 c0.020	44 c0.452	21	10
Nebraska	Bran	19	3.0	3.21	0.028	0.34	6.54	2.3	1.5
USA, 2010	Cooking water	19	0.012	0.144	< 0.01	< 0.01	0.16	0.0093	0.037
(Wesley)	Cooking water 2	19	0.011	0.116	< 0.01	< 0.01	0.13	0.0085	0.030
	Germ	19	2.13	2.86	0.039	0.083	5.08	1.7	1.2
2 applications	Gluten	19	0.655	1.95	0.016	0.022	2.62	0.51	0.60
6 day RTI	Middlings	19	0.78	2.73	0.023	0.073	3.57	0.60	0.82
Growth Stages 83 (×2)	Pasta, cooked	19	0.113	0.561	< 0.01	< 0.01	0.67	0.088	0.15
991 (×2) g ai/ha	Pasta, dried and cooked	19	0.121	0.659	< 0.01	< 0.01	0.78	0.094	0.18
130 (×2) L/ha	Pasta, dry	19	0.423	2.69	0.025	0.034	3.13	0.33	0.72
	Pasta, fresh	19	0.30	2.23	0.017	0.023	2.52	0.23	0.58
	Shorts	19	0.791	2.63	0.022	0.039	3.44	0.61	0.79
	Starch	19	0.011	0.069	< 0.01	< 0.01	0.080	0.009	0.018
	White bread	19	0.293	1.94	0.015	0.019	2.22	0.23	0.51
	White flour	19	0.42	2.46	0.019	0.026	2.93	0.33	0.67
	Whole meal	19	1.45	3.26	0.027	0.15	4.85	1.1	1.1
	Wholemeal bread	19	0.841	1.97	0.019	0.14	2.94	0.65	0.67

LOQ of parent, DFEAF, 6-CNA 0.01, DFA 0.02 mg/kg (parent equivs.) for all matrices except aspirated grain fraction (0.2 mg/kg)

Corn

The effect of processing (laboratory scale) on residues of flupyradifurone in field corn (Table 156) was investigated in two trials conducted in the USA in the 2010 growing season (Lenz 2012b, RARVY030).

Field corn with incurred residues was obtained where plants were sprayed with two foliar sprays of BYI 02960 200 SL. All applications were made using ground-based equipment. A non-ionic surfactant was used in all applications at 0.25% v/v. Single composite field corn grain RAC samples were harvested 21 days after the last application.

Drying and classification of grain using sieves yielded aspirated grain fractions.

For wet and dry milling the corn sample was dried to a moisture content of 10–13%. The samples were cleaned by aspiration and screening.

For dry milling the whole corn was moisture conditioned to 21% and tempered for approximately 2 hours, then the kernel was cracked. Corn stock from the mill was dried then bran, germ and large grits were separated from grits, meal and flour. The bran, germ and large grits were separated. After aspiration and further screening, grits, meal and flour fractions were collected. The germ fractions were dried until moisture was between 14–16%, then were heated and flaked. Flaked kernel material was placed in stainless steel extractors and submerged in hexane. After 30 minutes the miscella (crude oil and hexane) was drained and fresh hexane added to repeat the cycle two times. Residual hexane was removed from the spent flakes. The resultant fractions were miscella and solvent extracted germ meal. Crude oil and hexane in the miscella were separated and the crude oil heated for hexane removal. The crude oil was converted to refined oil and soapstock. The refined oil sequentially gave bleached oil then deodorised oil and distillates.

For wet milling the dried and cleaned corn was steeped in 49–54 °C water containing 0.1–0.2% sulphur dioxide for 22–48 hours. At the end of the steeping period, the whole corn was passed through a disc mill and most the germ and hull removed using a water centrifuge. Germ and hull were dried to give 5–10% moisture. After drying, germ and hull were separated using aspiration and screening. Cornstock (without germ and hull) was ground in the disc mill. Ground material was passed over a screen and material on top of the screen was discarded. Process water (with starch and gluten) passing through the screen was separated into starch and gluten using batch centrifugation. Starch was dried until the moisture was ≤ 15%. Starch fractions were collected. The germ fractions were moisture conditioned to 12%, heated, flaked and pressed to liberate part of the crude oil. Resulting fractions are expelled crude oil and presscake, with residual crude oil. Presscake was placed in stainless steel extractors and submerged in 49–60 °C hexane. After 30 minutes, the miscella was drained and fresh hexane was added to repeat the cycle two times. Residual hexane was removed from the spent presscake. The resultant fractions were miscella and solvent extracted presscake (germ cake). Crude oil and hexane in the miscella were separated and the crude oil heated for hexane removal. The crude oil from expelling and solvent extraction were filtered and combined for refining. The crude oil samples from wet milling were refined, bleached and deodorised as for dry milling.

Residues of flupyradifurone and metabolites in corn RAC and processed commodities were quantitated by LC/MS/MS using Method 01304. Acceptable concurrent recovery data for corn commodities were obtained for each analyte.

Table 156 Residues in field corn processed fractions from the foliar application of flupyradifurone to corn in the USA (Lenz 2012b, RARVY030)

Trial No.	Sample (%DM)	DAT	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6-CNA		+ 6-CNA
GAP, USA, Corn		21							
RV270-10PA,	Grain (RAC)	21	0.012	< 0.05	< 0.01	< 0.01	0.062	NA	NA
Carlyle,	Aspirated grain fractions	21	0.305	< 0.05	< 0.01	< 0.01	0.36	25	5.8

Trial No.	Sample (%DM)	DAT	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6-CNA		+ 6-CNA
Illinois	Bran	21	0.022	< 0.05	< 0.01	< 0.01	0.072	1.8	1.2
USA, 2010	Flour	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06	< 0.83	< 0.97
(Burrus 6H22)	Germ, dry milling	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06	< 0.83	< 0.97
	Germ, wet milling	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06	< 0.83	< 0.97
2 applications	Grits	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06	< 0.83	< 0.97
7 day RTI	Meal, dry milled	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06	< 0.83	< 0.97
Growth Stages 87 (×2)	Oil, dry milled	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06	< 0.83	< 0.97
1010 (×2) g ai/ha	Oil, wet milled	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06	< 0.83	< 0.97
280 and 365 L/ha	Starch	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06	< 0.83	< 0.97
	Steep water	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06	< 0.83	< 0.97
RV271-10PA,	Grain (RAC)	21	0.024	< 0.05	< 0.01	< 0.01	0.074	NA	NA
Atlantic,	Aspirated grain fractions	21	0.498	< 0.05	< 0.01	< 0.01	0.55	21	7.4
Iowa	Bran	21	0.091	< 0.05	< 0.01	< 0.01	0.14	3.8	1.9
USA, 2010	Flour	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06	< 0.42	< 0.81
(Garst 85R08-3000GT)	Germ, dry milling	21	0.029	< 0.05	< 0.01	< 0.01	0.079	1.2	1.1
	Germ, wet milling	21	0.013	< 0.05	< 0.01	< 0.01	0.063	0.54	0.85
2 applications	Grits	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06	< 0.42	< 0.81
8 day RTI	Meal, dry milled	21	0.011	< 0.05	< 0.01	< 0.01	0.061	0.46	0.82
Growth Stage 89 (×2)	Oil, dry milled	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06	< 0.42	< 0.81
1030 and 1010 g ai/ha	Oil, wet milled	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06	< 0.42	< 0.81
299 and 290 L/ha	Starch	21	< 0.01	< 0.05	< 0.01	< 0.01	< 0.06	< 0.42	< 0.81
	Steep water	21	0.012	< 0.05	< 0.01	< 0.01	0.062	0.50	0.84

LOQ of parent, DFEAF, 6-CNA 0.01, DFA 0.02 mg/kg (parent equivs.) for all matrices

Cotton

The effect of processing (laboratory scale) on residues of flupyradifurone in cotton (Table 156) was investigated in two trials (Lenz and Beedle 2012, RARVY033). Cotton with incurred residues was obtained following foliar applications of flupyradifurone to cotton plants at exaggerated rates (approximately 5× the maximum seasonal rate). All applications were made using ground-based equipment. An adjuvant was used in all applications at 0.25% v/v. Cotton RAC samples were harvested at a 13–14 day PHI.

Undelinted cottonseed raw agricultural commodity (RAC) was used to generate meal, hulls and oil (refined, bleached and deodorised). Processing was performed using procedures which simulated commercial processing practices. Seed cotton was cleaned to remove the gin by-products (gin trash) and ginned to separate ginned cottonseed (undelinted cottonseed) and lint in a saw gin. Resulting products were ginned cottonseed, lint and gin trash. Ginned seed fractions were collected and placed into frozen storage. Undelinted cottonseed (ginned) samples were delinted to remove of the most remaining lint and produce delinted cottonseed with approximately 3% lint remaining on the seed.

Delinting produced delinted cottonseed, linters and linter motes. The delinted seed was mechanically cracked and the hull material was then separated from the kernel. Kernel material was processed into meal and crude oil. Degumming and desliming of crude oil produced pre-clarified oil and slime.

Mixing and heating of pre-clarified oil with sodium hydroxide and centrifugation of neutralised oil, separated refined oil and soapstock.

Residues of flupyradifurone and metabolites in cotton RAC and processed commodities were quantitated by LC/MS/MS using Method 01304. Acceptable concurrent recovery data for cotton commodities were obtained for each analyte.

Table 157 Residues in cotton processed fractions from the foliar application of flupyradifurone to cotton plants in the USA (Lenz and Beedle 2012, RARVY033)

Trial No.	Sample	DAT	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6-CNA		+ 6-CNA
GAP, USA, Cotton		14							
RV276-10PA,	Seed undelinted (RAC)	13	1.415	< 0.05	0.038	0.078 c0.072	1.54	NA	NA
Proctor,	Oil refined	13	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.0071	< 0.039
Arkansas	Oil, solv. extracted	13	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.0071	< 0.041
USA, 2010	Crude oil, pre-clarified	13	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.0071	< 0.041
(DG2400RF)	Crude oil, neutralised	13	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.0071	< 0.041
2 applications	Meal	13	0.052	< 0.05	< 0.01	0.17 c0.13	0.27	0.037	0.18
10 day RTI	Hull	13	0.135	< 0.05	< 0.01	0.01	0.20	0.095	0.13
Growth Stages 88 and 89									
1000 (×2) g ai/ha									
93 (×2) L/ha									
RV277-10PA,	Seed undelinted (RAC)	14	0.247	< 0.05	< 0.01	< 0.01	0.297	NA	NA
Uvalde,	Oil refined	14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.040	< 0.20
Texas	Oil, solv. extracted	14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.040	< 0.20
USA, 2010	Crude oil, pre-clarified	14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.040	< 0.20
(Stoneville 5458B2RF)	Crude oil, neutralised	14	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.040	< 0.20
2 applications	Meal	14	0.062	0.065	< 0.01	0.12	0.247	0.25	0.83
9 day RTI	Hull	14	0.232	< 0.05	< 0.01	0.013	0.295	0.94	0.99
Growth Stages 87 and 88									
988 and 989 g ai/ha									
187 and 168 L/ha									

LOQ of parent, DFEAF, 6-CNA 0.01, DFA 0.05 mg/kg (parent equivs.) for all matrices

Peanut

The effect of processing (laboratory scale) on residues of flupyradifurone in peanuts (Table 158) was investigated in two trials carried out in the USA during the 2010 growing season (Lenz 2012c, RARVY032). Peanut nutmeat with incurred residues was obtained where plants were sprayed with two foliar sprays at 991–1020 g ai/ha. Applications were made using ground-based equipment and an adjuvant was added to the spray mixture for all applications.

Bulk peanut samples were processed into processed peanut commodity samples per simulated commercial procedures. The unshelled whole peanuts were dried, screened and cleaned to remove debris. The whole peanuts were shelled to yield the nutmeat kernel and a portion of the kernels was separated to produce the dry roasted whole nutmeat and peanut butter. The nutmeats were roasted and the skins removed to yield the "from the processor" nutmeat sample. Some roasted nutmeat was combined with commercial peanut oil and salt and blended to produce peanut butter. The remaining kernels were pressed in an expeller to produce the crude oil (expeller crude oil) and presscake. The presscake was further ground and extracted with solvent (hexane) to yield more crude oil (solvent-extracted crude oil) after removal of the solvent. The crude oil samples were further treated with alkali, filtered and bleached to yield refined oil. The presscake after solvent extraction was heated to remove any residual solvent yielding the peanut meal sample.

Residues of flupyradifurone and metabolites in peanut RAC and processed commodities were quantitated by LC/MS/MS using Method 01304. Acceptable concurrent recovery data for peanut commodities were obtained for each analyte.

Table 158 Residues in peanut processed fractions from the foliar application of flupyradifurone to peanuts in the USA (Lenz 2012c, RARVY032)

Trial No.	Sample	DAT	Residues as parent (mg/kg)					Processing Factor	
Location,			Parent	DFA	DFEAF	6-CNA	Parent	Parent	Parent
Year							+ DFA		+ DFA
(Variety)							+ 6-CNA		+ 6-CNA
GAP, USA, Peanut		7							
RV274-10PA,	Nut without shell (RAC)	7	0.123	0.309	< 0.01	0.036	0.468	NA	NA
Suffolk, Virginia	Meal	7	0.181	0.394	< 0.01	0.056	0.631	1.5	1.3
USA, 2010	Oil, refined	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.081	0.13
(Champs)	Peanut butter	7	0.026	0.181	< 0.01	0.036	0.243	0.21	0.52
	Peanut, roasted	7	0.024	0.191	< 0.01	0.028	0.243	0.20	0.52
2 applications									
10 day RTI									
Growth Stages 87 and 88									
1000 and 1020 g ai/ha									
112 (×2) L/ha									
RV275-10PA,	Nut without shell (RAC)	7	0.011	< 0.05	< 0.01	< 0.01	0.061	NA	NA
Charlotte, Texas	Meal	7	0.020	< 0.05	< 0.01	< 0.01	0.070	1.8	1.1
USA, 2010	Oil, refined	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.91	< 0.98
(Florida runner 07)	Peanut butter	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.91	< 0.98
	Peanut, roasted	7	< 0.01	< 0.05	< 0.01	< 0.01	< 0.060	< 0.91	< 0.98
2 applications									
8 day RTI									
Growth Stages 88 and 89									
991 and 1000 g ai/ha									
168 (×2) L/ha									

LOQ of parent, DFEAF, 6-CNA 0.01, DFA 0.05 mg/kg (parent equivs.) for all matrices

Coffee beans

The effect of processing (laboratory scale) on residues of flupyradifurone in coffee (Table 159) was investigated in two trials carried out in Brazil and Mexico during the 2011 growing season to determine the residues of flupyradifurone in green coffee beans and then after processing, in roasted coffee beans and instant coffee (Hoag 2012c, RARVP075). A single soil drench application was made followed by three foliar spray applications. All foliar applications were made using ground-based equipment and adjuvant was added at 0.25% v/v.

Coffee cherries were collected from the treated plots at a target pre-harvest interval (PHIs) of 14 days. According to normal commercial practice in Brazil (trial RV235-11PA) and in various regions in Mexico (trial RV247-11PA) coffee cherries were allowed to air-dry before removing the outer hull and parchment using a machine that simulates large-scale commercial production of coffee beans, green. For trial RV235-11PA (Brazil) the cherries were allowed to air-dry for 10-days, before removing the outer hull and parchment. For trial RV247-11PA (Mexico), coffee cherries were placed into forced-air drying ovens at a temperature of 50 °C for four days, followed by air-drying for eight days to yield the required sample size of coffee bean, green after removing the outer hull and parchment.

The remainder of each bulk sample was used to generate the processed commodities, coffee bean, roasted, and coffee, instant. Loose hulls and foreign materials was removed from the green coffee beans. The green coffee beans were batch roasted at temperatures from 215–245 °C then cooled to give the roasted bean sample. Roasted beans were ground then screened. Ground roasted coffee was brewed and the brewed ground coffee was pressed to separate the spent grounds from the coffee extract. The coffee extract recovered from the pressing operation was cycled back into the brewing process. Brewed coffee was transferred to plastic pots. Coffee extract was batch centrifuged then placed into freezing trays. After freezing, the filled pans were taken from the freezer and placed into a freeze dryer to give instant coffee.

Residues of flupyradifurone and metabolites in coffee RAC and processed commodities were quantitated by LC/MS/MS using Method 01304. Acceptable concurrent recovery data for coffee commodities were obtained for each analyte.

Table 159 Residues in coffee processed fractions from the foliar application of flupyradifurone to coffee in Brazil and Mexico (Hoag 2012c, RARVP075)

Trial No.	Sample	DAT	Residues as parent (mg/kg)				
Location,			Parent	DFA	DFEAF	6- CNA	Parent
Year							+ DFA
(Variety)							+ 6-CNA
RV235-11PA,	Bean, green (RAC)	14	0.37	0.118	0.022	< 0.01	0.49
Paulinia,			0.34	0.105	0.019	< 0.01	0.45
São Paulo	Bean, roasted	14	0.20	0.092	0.014	0.013	0.31
Brazil, 2011			0.19	0.081	0.0071	0.022 c0.015	0.29
(Catuai Vermelho)	Coffee, instant	14	0.36	0.381	0.028	0.033	0.74
			0.87	0.573 c0.185	0.029	0.043	1.5
1 [soil drench] + 3 [foliar]							
91, 14 and 14 day RTIs							
Growth Stages 77, 79, 80 and 85							
1225, 409, 396 and 401 g ai/ha							
149, 411, 374 and 398 L/ha							
RV247-11PA,	Bean, green (RAC)	14	0.98	0.501	0.105	0.031	1.5
La Union			1.10	0.523	0.111	0.024	1.6
Zihuateutla,	Bean, roasted	14	0.73	0.55	0.064	0.045	1.3
Puebla,			0.57	0.598	0.049	0.021	1.2
Mexico, 2011	Coffee, instant	14	2.38	2.34	0.266	0.192	4.9
(Caturra)			1.80	3.213 c0.202	0.199	0.095	5.1
1 [soil drench] + 3 [foliar]							
85, 12 and 13 day RTIs							
Growth Stages 72, 81, 81 and 85							
1206, 401, 399 and 398 g ai/ha							
201, 402, 403 and 402 L/ha							

LOQ of parent, DFEAF and 6-CNA 0.01, DFA 0.05 mg/kg (parent equivs.) for coffee bean, green and roasted coffee

LOQ of parent, DFEAF, DFA and 6-CNA 0.05 mg/kg (parent equivs.) for instant coffee

Note: There was no submitted registered use pattern for coffee

Hops

The effect of processing (laboratory scale) on residues of flupyradifurone in hops (Table 160) was investigated (Schulte and Bauer 2012d, 10-3407) in two trials carried out in Germany during the 2010 growing season to determine the residues in green and dried hop cones and then after processing, in beer as well as the additional processing intermediates, hops draff and brewer’s yeast. BYI 02960 SL200 was sprayed once at an application rate of approximately 360 g ai/ha and water volume of 3000 L/ha.

Beer processing simulated industrial brewing practices. Prior to the addition of hops to the wort, ground malted barley and water were mixed in a mash tun (“mashing”) then the wort was separated from the insoluble malt components (“lautering”). Green hop cones were dried to give cone kiln-dried hops (RAC), then milled. Hop pellets were added to the wort, which was then boiled. Filtration gave hops draff. After addition of yeast, the mixture was fermented giving brewer's yeast. The beer is matured, filtered and bottled and then the final product, beer, is sampled.

Residues of flupyradifurone and metabolites in hops RAC and processed commodities were quantitated using LC/MS/MS Method 01304. Acceptable concurrent recovery data for hop commodities were obtained for each analyte.

Table 160 Residues in hops processed fractions from the foliar application of flupyradifurone to hops in Germany (Schulte and Bauer 2012d, 10-3407)

Trial No.,	Sample	DAT	Residues as parent (mg/kg)			
Location,			Parent	DFA	DFEAF	6-CNA
Year						
(Variety)						
GAP, USA, Hops		21				
10-3407-01,	Cone, green	21	0.43	< 0.2	< 0.1	0.29 c0.27
Golzern	Cone kiln-dried (RAC)	21	2.2	0.72	< 0.1	1.58 c1.72
Germany, 2010	Hops draff	21	< 0.1	< 0.2	< 0.1	0.16 c0.15
(Nugget)	Brewer's yeast	21	< 0.1	< 0.2	< 0.1	< 0.1
	Beer	21	0.01	< 0.02	< 0.01	< 0.01
1 application						
Growth Stage 71						
360 g ai/ha						
3000 L/ha						
10-3407-02,	Cone, green	21	1.1	0.37	< 0.1	0.24 c0.16
Hohenebra	Cone kiln-dried (RAC)	21	4.2	0.76	< 0.1	0.77 c1.26
Germany, 2010	Hops draff	21	< 0.1	< 0.2	< 0.1	0.15
(Nordischer Brauer)	Brewer's yeast	21	< 0.1	< 0.2	< 0.1	< 0.1
	Beer	21	0.02	< 0.02	< 0.01	< 0.01
1 application						
Growth Stage 75						
360 g ai/ha						
3000 L/ha						

LOQ of parent, DFEAF 0.01, DFA 0.02 mg/kg (parent equivs.) for beer

LOQ of parent, DFEAF 0.1, and DFA 0.2 mg/kg (parent equivs.) for all matrices except beer

No processing factors were estimated due to significant residues observed in controls of the RAC samples

The results of the processing factors are summarized in Table 161 below:

Table 161 Summary of processing factors for flupyradifurone residues

Raw Agricultural Commodity (RAC)	Processed Commodity	Calculated Processing factors (Parent + DFA + 6-CNA)	Best Estimate Processing Factor (Parent + DFA + 6-CNA)
Oranges	Peel, ripe unwashed	1.6, 1.6, 2.1, 2.5	1.85
	Fruit, washed	0.81, 0.83, 1.1, 1.5	0.965
	Washings	< 0.13, 0.13, 0.16, < 0.21	0.145
	Peel, washed	2.1, 2.8, 2.8, 4.3	2.8
	Peel without oil	1.8, 2.2, 3.0, 4.0	2.6
	Raw juice	0.11, < 0.13, 0.15, < 0.21	0.14
	Juice	0.11, < 0.13, 0.14, < 0.21	0.135
	Oil	< 0.11, < 0.13, < 0.14, < 0.21	0.135
	Strain rest	0.11, 0.14	0.125
	Pulp	0.13, < 0.21, < 0.21, 0.21, 0.89, 1.2	0.21
	Pomace, wet	1.1, 1.3, 1.3, 1.4	1.3
	Pomace, dried	3.9, 4.2, 4.8, 5.2	4.5

Raw Agricultural Commodity (RAC)	Processed Commodity	Calculated Processing factors (Parent + DFA + 6-CNA)	Best Estimate Processing Factor (Parent + DFA + 6-CNA)
	Marmalade	< 0.11, < 0.14, 0.17, 0.21	0.155
Apple	Whole fruit, washed	0.83, 1.1, 1.1, 1.4	1.1
	Washings	< 0.21, < 0.27, < 0.33, < 0.50	0.30
	Raw sauce	0.93, 1.0, 1.0, 1.0	1.0
	Strain rest	1.2, 1.3, 1.4, 1.6	1.35
	Sauce	0.71, 0.78, 0.82, 0.83	0.80
	Pomace, wet	1.1, 1.3, 1.4, 1.6	1.35
	Pomace, dried	3.8, 3.9, 4.0, 4.1	3.95
	Raw juice	0.67, 0.67, 0.86, 1.0	0.765
	Retentate	0.56, 0.67, 0.79, 0.82	0.73
	Juice	0.50, 0.56, 0.64, 0.73	0.60
	Peel	4.4	4.4
	Fruit, peeled	0.56	0.56
	Fruit, dried	1.9	1.9
Grape	Berry	0.47, 0.83, 0.90, 1.2	0.865
	Pomace, grape	1.5, 1.7, 1.8, 1.8	1.75
	Must	0.45, 0.55, 0.85, 0.88	0.70
	Juice, pasteurised	0.50, 0.88	0.69
	Wine at bottling	0.30, 0.45, 0.70, 0.78	0.575
	Wine at first taste test	0.23, 0.33, 0.50, 0.55	0.415
	Jelly	0.21, 0.38	0.295
	Washings	0.076, 0.11	0.093
	Raisin waste	2.4, 2.8	2.6
	Raisin	2.2, 2.8	2.5
Cucumber	Washings	< 0.2, < 0.33	0.265
	Whole fruit, washed	1.2, 1.5	1.35
	Preserve	0.67, 0.89	0.78
	Fruit fermented	0.47, 0.56	0.515
	Brine	0.40, 0.44	0.42
Tomato	Whole fruit, washed	0.67, 1.0, 1.0	1.0
	Washings	< 0.27, 0.43, < 0.50	0.43
	Raw juice	0.55, 0.67, 0.86	0.67
	Juice	0.55, 0.67, 0.71	0.67
	Strain rest	0.82, 1.0, 1.0	1.0
	Raw puree	1.5, 1.5, 1.9	1.5
	Puree	1.5, 1.5, 1.7	1.5
	Paste	1.9	1.9
	Peel	1.7, 2.1, 2.3	2.1
	Peeling water	< 0.27, 0.43, 0.50	0.43
	Fruit peeled	0.55, 0.60, 0.67	0.60
	Preserve	0.55, 0.71, 0.83	0.71
	Fruit dried	2.45	2.45
Soya bean	Aspirated grain fractions	1.7, 12.5	7.1
	Meal	1.0, 1.7	1.35
	Hull	0.72, 0.79	0.76
	Oil, refined	< 0.012, < 0.063	0.038
	Milk	0.13, 0.28	0.21
	Defatted flour	1.2, 1.9	1.55
Potato	Tuber, washed	1.0, 1.28	1.14
	Washings	< 0.91, < 1.0	0.96
	Crisps	< 1.0, 1.5	1.25
	Flakes	1.0, 2.1	1.55
	Peel, wet	< 0.091, 1.1	0.596
	Starch	< 0.091, < 1.0	0.546
	Tuber with peel, cooked	< 1.0, 1.1	1.05
	Tuber, peeled	< 1.0, 1.2	1.1
	Tuber, steamed, cooked	< 0.091, < 1.0	0.546
	Cooking water	< 0.091, < 1.0	0.546
Barley	Malt sprouts	0.67, 0.91	0.79
	Brewer’s malt	0.46, 0.52	0.49

Raw Agricultural Commodity (RAC)	Processed Commodity	Calculated Processing factors (Parent + DFA + 6-CNA)	Best Estimate Processing Factor (Parent + DFA + 6-CNA)
	Brewer's grain	0.037, 0.10	0.069
	Hops draff	0.36, 0.51	0.44
	Brewer's yeast	0.13, 0.13	0.13
	Beer	0.061, 0.089	0.075
	Pearl barley	0.073, 0.17	0.12
	Pearl barley rub-off	2.55, 3.3	2.93
Wheat	Semolina Bran	4.05, 7.1	5.6
	Semolina	0.50, 1.4	0.95
	White flour bran	5.3, 8.9	7.1
	White flour	0.25, 0.40, 0.49, 0.67	0.445
	White bread	0.25, 0.28, 0.36, 0.51	0.32
	Whole meal	1.1, 1.1, 1.4, 1.6	1.25
	Whole meal bread	0.67, 0.71, 0.88, 0.96	0.795
	Wheat germ	0.81, 1.2, 1.3, 1.3	1.25
	Aspirated grain fractions	10, 11	10.5
	Bran	1.5, 1.6	1.55
	Cooking water	0.024, 0.037	0.031
	Cooking water 2	0.028, 0.030	0.029
	Gluten	0.20, 0.60	0.40
	Middlings	0.82, 0.84	0.83
	Pasta, cooked	0.12, 0.15	0.135
	Pasta dried and cooked	0.17, 0.18	0.175
	Pasta, dry	0.57, 0.72	0.645
	Pasta, fresh	0.44, 0.58	0.51
	Shorts	0.79, 1.1	0.945
	Starch	0.018, 0.033	0.026
Corn (Maize)	Aspirated grain fractions	5.8, 7.4	6.6
	Bran	1.2, 1.9	1.55
	Flour	< 0.81, < 0.97	0.89
	Germ, dry milling	< 0.97, 1.1	1.035
	Germ, wet milling	0.85, < 0.97	0.91
	Grits	< 0.81, < 0.97	0.89
	Meal, dry milled	0.82, < 0.97	0.895
	Oil, dry milled	< 0.81, < 0.97	0.89
	Oil, wet milled	< 0.81, < 0.97	0.89
	Starch	< 0.81, < 0.97	0.89
	Steep water	0.84, < 0.97	0.905
Cotton	Oil, refined	< 0.20	0.20
	Oil, solv. extracted	< 0.20	0.20
	Crude oil, pre-clarified	< 0.20	0.20
	Crude oil, neutralised	< 0.20	0.20
	Meal	0.83	0.83
	Hull	0.99	0.99
Peanuts	Meal	1.1, 1.3	1.2
	Oil, refined	0.13, < 0.98	0.56
	Peanut butter	0.52, < 0.98	0.75
	Peanut, roasted	0.52, < 0.98	0.75

Residues in animal commodities

Dairy cattle transfer study (Moore and Harbin 2012, RARVP050-1)

A group of 19 Holstein dairy cows (approximately 2.5–3.5 years of age) were dosed with flupyradifurone by capsule daily after the morning milking for 29 consecutive days. Two cows served as controls (Group 1), four were dosed at 4.81 ppm (Group 2, average of 0.184 mg ai/kg bw/day), three at 23.1 ppm (Group 3, average of 0.896 mg ai/kg bw/ day), three at 49.6 ppm (Group 4, average of 1.83 mg ai/kg bw/ day) and seven at 135 ppm (Group 5, average of 4.89 mg ai/kg bw/ day). The overall average feed consumption was 20.2 kg/cow/day (dry weight). The weight of the cattle at the start of dosing ranged from 462–613 kg and from 453–583 kg at the end of dosing.

Milk was collected twice daily from all dose groups and was a composite sample of the evening milk and the following morning milk prior to administration of the daily dose. Milk from the 135 ppm group was sub-sampled for analysis on days 0, 2, 4, 7, 10, 14, 17, 19, 25, and 28, and from the other groups on Day 28. A portion of the day 25 milk from a control animal and three of the 135 ppm group was processed into cream (milk fat) and whey (skim milk) for analysis.

On Day 29, one of the control cows (Group 1), all the Groups 2, 3, and 4 cows, and four of the Group 5 dose group cows were sacrificed. Representative samples of liver (each lobe), kidney (centre and ends), fat (approximately equal composite of omental, renal, and subcutaneous), and muscle (composite of loin, round, and flank) were collected. The remaining cows (one control and three from the Group 5 dose level) entered the depuration phase of the study, and were sacrificed 3, 7, and 14 days post-dosing (Days 32, 36, and 43). One cow from Group 5 was sacrificed at each interval and the control cow (Group 1) was sacrificed on Day 43. Milk from Days 30, 31 and 35 and tissues from the cow sacrificed at each interval were analysed to monitor the decline of BYI 02960 residues.

Residues of flupyradifurone and its metabolites BYI 02960-hydroxy, BYI 02960-acetyl-AMCP and difluoroacetic acid were quantitated using LC/MS/MS Method RV-004-A11-04. The LOQ was 0.01 mg/kg (parent equivalents) for flupyradifurone, BYI 02960-OH and BYI 02960-AMCP and 0.020 mg/kg for DFA in milk, cream, fat, kidney, liver and muscle. The LOQ was 0.01 mg/kg (parent equivalents) for flupyradifurone, BYI 02960-OH and BYI 02960-AMCP and 0.05 mg/kg for DFA in whey.

Residues found in milk are summarized below in Table 162. Residues in tissues are summarized in Table 163. The total BYI 02960 (parent + DFA) residues in milk from Group 5 (135 mg/kg feed) reached plateau levels within two to four days of consecutive dosing and declined rapidly, from 0.81 mg/kg to < LOQ at 6 days after cessation of dose administration. The residue in milk was primarily (60–90%) parent flupyradifurone.

In addition, Day-25 milk samples from Group 5 were separated into cream and whey, to determine if the residues preferentially collect in more aqueous or fatty compartments. The processing/transfer factors (PF) for the total flupyradifurone (parent + DFA) residues indicated a very slight preference for more aqueous conditions, with a PF of 1.15 to whey and 0.79 to cream.

Table 162 Flupyradifurone residues in milk, cream and whey

Sampling Day	Flupyradifurone (mg/kg)	DFA [a] (mg/kg)	Total (Flupyradifurone + DFA) Residues (mg/kg)
MILK: Group 5 Treated Dairy Cows (average 135 ppm in the feed)			
2 Day	0.840	0.087	0.927
	0.774	0.080	0.854
	0.895	0.092	0.987
	0.625	0.078	0.703
	0.619	0.074	0.693
	0.840	0.062	0.902
	0.630	0.091	0.721
Average			0.827
4 Day	0.832	0.108	0.940
	0.674	0.114	0.788
	0.977	0.128	1.105
	0.358	0.052	0.410
	0.739	0.144	0.883
	0.844	0.088	0.932
	1.656	0.098	1.754
Average			0.973
7 Day	0.714	0.106	0.820
	0.665	0.105	0.770
	0.763	0.135	0.898
	0.480	0.104	0.584

Sampling Day	Flupyradifurone (mg/kg)	DFA [a] (mg/kg)	Total (Flupyradifurone + DFA) Residues (mg/kg)
	0.812	0.236	1.048
	0.905	0.115	1.02
	0.476	0.163	0.639
Average			0.826
10 Day	0.800	0.107	0.907
	0.708	0.112	0.820
	0.983	0.159	1.142
	0.547	0.096	0.643
	0.844	0.232	1.076
	0.727	0.127	0.854
	0.733	0.127	0.860
Average			0.900
14 Day	0.885	0.129	1.014
	0.752	0.126	0.878
	0.915	0.166	1.081
	0.696	0.100	0.796
	0.681	0.249	0.930
	0.756	0.153	0.909
	0.799	0.135	0.934
Average			0.935
17 Day	0.896	0.119	1.015
	0.745	0.121	0.866
	0.996	0.156	1.152
	0.554	0.102	0.656
	1.019	0.241	1.26
	0.886	0.125	1.011
	0.720	0.135	0.855
Average			0.974
19 Day	0.888	0.109	0.997
	0.746	0.112	0.858
	0.888	0.149	1.037
	0.742	0.100	0.842
	1.102	0.185	1.287
	0.850	0.110	0.96
	0.556	0.145	0.701
Average			0.955
25 Day	0.689	0.092	0.781
	0.666	0.093	0.759
	0.730	0.141	0.871
	0.595	0.095	0.690
	0.532	0.192	0.724
	0.707	0.095	0.802
	0.636	0.094	0.730
Average			0.765
28 Day	0.695	0.094	0.789
	0.671	0.099	0.770
	0.919	0.136	1.055
	0.615	0.100	0.715
	0.654	0.168	0.822
	0.854	0.106	0.960
	0.826	0.266	1.092
Average			0.886
MILK: Group 4 Treated Dairy Cows (average 49.6 ppm in the feed)			
28 Day	0.290	0.046	0.336
	0.238	0.042	0.280
	0.273	0.035	0.308
Average			0.308
MILK: Group 3 Treated Dairy Cows (average 23.1 ppm in the feed)			
28 Day	0.125	0.023	0.148
	0.109	< 0.020	0.129

Sampling Day	Flupyradifurone (mg/kg)	DFA [a] (mg/kg)	Total (Flupyradifurone + DFA) Residues (mg/kg)
	0.090	< 0.020	0.110
Average			0.129
MILK: Group 2 Treated Dairy Cows (average 4.81 ppm in the feed)			
28 Day	0.025	< 0.020	0.045
	0.026	< 0.020	0.046
	0.023	< 0.020	0.043
	0.019	< 0.020	0.039
Average			0.043
MILK: Depuration Phase: Group 5 Treated Dairy Cows (average 135 ppm in the feed)			
29 Day	0.651	0.141	0.792
	0.606	0.165	0.771
	0.743	0.113	0.856
Average:			0.806
30 Day Depuration	0.063	0.063	0.126
	0.043	0.104	0.147
	0.071	0.067	0.138
Average:			0.137
31 Day Depuration	< 0.01	0.041	0.051
	< 0.01	0.053	0.063
	< 0.01	0.035	0.045
Average:			0.053
35 Day Depuration	< 0.01	< 0.020	< 0.030
	< 0.01	< 0.020	< 0.030
Average:			< 0.030
CREAM: Group 5 Treated Dairy Cows (average 135 ppm in the feed)			
25 Days	0.527	0.042	0.569
	0.528	0.039	0.567
	0.604	0.068	0.672
Average:			0.603
WHEY: Group 5 Treated Dairy Cows (average 135 ppm in the feed)			
25 Days	0.749	0.107	0.856
	0.721	0.112	0.833
	0.803	0.151	0.954
Average:			0.881

Residues of BYI 02960-OH and BYI 02960-AMCP were < 0.01 mg/kg at all doses

[a] Calculated as parent equivalents.

Table 163 Flupyradifurone residues in fat, liver, kidney and muscle

Sampling Day	Flupyradifurone (mg/kg)	DFA [a] (mg/kg)	BYI 02960-OH [a] (mg/kg)	Total (Flupyradifurone + DFA) Residues (mg/kg)
Group 5: Treated Dairy Cows (average 135 ppm in the feed)				
Fat				
29 Days	0.916	0.427	0.022	1.343
	0.856	0.204	0.015	1.06
	0.767	0.375	< 0.01	1.142
	1.370	0.560	0.031	1.93
	Average:			1.37
Fat—Depuration Phase				
32 Days	< 0.01	0.100	< 0.01	0.11
36 Days	< 0.01	< 0.020	< 0.01	< 0.030
43 Days	< 0.01	< 0.020	< 0.01	< 0.030
Kidney				
29 Days	4.26	0.528	0.106	4.788
	4.80	0.379	0.068	5.179
	4.16	0.630	0.119	4.79
	5.66	0.693	0.117	6.353
	Average:			5.28

Sampling Day	Flupyradifurone (mg/kg)	DFA [a] (mg/kg)	BYI 02960-OH [a] (mg/kg)	Total (Flupyradifurone + DFA) Residues (mg/kg)
Depuration Phase				
32 Days	0.045	0.141	< 0.01	0.186
36 Days	< 0.01	< 0.020	< 0.01	< 0.030
43 Days	< 0.01	< 0.020	< 0.01	< 0.030
Liver				
29 Days	3.56	0.348	0.043	3.908
	2.96	0.284	0.042	3.244
	3.89	0.507	0.039	4.397
	3.39	0.457	0.014	3.847
	Average:			3.849
Liver—Depuration Phase				
32 Days	0.033	0.106	< 0.01	0.139
36 Days	< 0.01	< 0.020	< 0.01	< 0.030
43 Days	< 0.01	< 0.020	< 0.01	< 0.030
Muscle				
29 Days	1.35	0.368	0.018	1.718
	1.25	0.302	0.016	1.552
	1.54	0.473	< 0.01	2.013
	1.88	0.396	< 0.01	2.276
	Average:			1.890
Muscle—Depuration Phase				
32 Days	0.017	0.095	< 0.01	0.112
36 Days	< 0.01	< 0.020	< 0.01	< 0.030
43 Days	< 0.01	< 0.020	< 0.01	< 0.030
Group 4: Treated Dairy Cows (average 49.6 ppm in the feed)				
Fat				
29 Days	0.377	0.112	< 0.01	0.489
	0.248	0.111	< 0.01	0.359
	0.231	0.073	< 0.01	0.304
Average:				0.384
Kidney				
29 Days	2.15	0.217	0.031	2.367
	1.31	0.221	0.041	1.531
	1.91	0.170	0.062	2.08
Average:				1.99
Liver				
29 Days	2.00	0.169	0.021	2.169
	1.25	0.120	0.021	1.37
	1.79	0.106	0.017	1.896
Average:				1.81
Muscle				
29 Days	0.740	0.170	< 0.01	0.910
	0.490	0.128	< 0.01	0.618
	0.562	0.109	< 0.01	0.671
Average:				0.733
Group 3: Treated Dairy Cows (average 23.1 ppm in the feed)				
Fat				
29 Days	0.097	0.033	< 0.01	0.13
	0.110	0.05	< 0.01	0.16
	0.120	0.030	< 0.01	0.15
Average:				0.147
Kidney				
29 Days	0.754	0.099	0.027	0.853
	0.711	0.075	0.028	0.786
	0.894	0.068	0.022	0.962
Average:				0.867
Liver				
29 Days	0.821	0.071	< 0.01	0.892
	0.714	0.054	0.012	0.768

Sampling Day	Flupyradifurone (mg/kg)	DFA [a] (mg/kg)	BYI 02960-OH [a] (mg/kg)	Total (Flupyradifurone + DFA) Residues (mg/kg)
	0.730	0.045	0.012	0.775
Average:				0.812
Muscle				
29 Days	0.248	0.066	< 0.01	0.314
	0.260	0.057	< 0.01	0.317
	0.242	0.039	< 0.01	0.281
Average:				0.304
Group 2: Treated Dairy Cows (average 4.81 ppm in the feed)				
Fat				
29 Days	0.021	< 0.020	< 0.01	0.041
	0.018	< 0.020	< 0.01	0.038
	0.028	< 0.020	< 0.01	0.048
	0.017	< 0.020	< 0.01	0.037
Average:				0.041
Kidney				
29 Days	0.122	0.022	0.013	0.144
	0.134	< 0.020	< 0.01	0.154
	0.222	< 0.020	0.042	0.242
	0.158	< 0.020	< 0.01	0.178
Average:				0.180
Liver				
29 Days	0.172	< 0.020	< 0.01	0.192
	0.133	< 0.020	< 0.01	0.153
	0.156	< 0.020	< 0.01	0.176
	0.119	< 0.020	< 0.01	0.139
Average:				0.165
Muscle				
29 Days	0.044	< 0.020	< 0.01	0.064
	0.041	< 0.020	< 0.01	0.061
	0.048	< 0.020	< 0.01	0.068
	0.038	< 0.020	< 0.01	0.058
Average:				0.063

Residues of BYI 02960-AMCP were < 0.01 mg/kg at all doses

[a] Calculated as parent equivalents.

The results of the study are summarized in the following table.

Table 164 Residues of flupyradifurone and DFA in milk and tissues

Animal Commodity	Dose Level of Flupyradifurone (ppm)	Flupyradifurone Residue (mg/kg)		DFA Residue (mg/kg) [a]		Flupyradifurone + DFA Residue as Parent Equivalents (mg/kg)	
		Mean	Highest	Mean	Highest	Mean	Highest [b]
Fat	4.81	0.021	0.028	< 0.020	< 0.020	0.041	0.048
	23.1	0.109	0.120	0.038	0.05	0.147	0.16
	49.6	0.285	0.377	0.099	0.112	0.384	0.489
	135	0.977	1.370	0.392	0.560	1.37	1.93
	(+3 days depuration)		< 0.01		0.100		0.11
	(+7 days depuration)		< 0.01		< 0.020		< 0.030
	(+14 days depuration)		< 0.01		< 0.020		< 0.030
Kidney	4.81	0.159	0.222	0.0205	0.022	0.180	0.242
	23.1	0.786	0.894	0.081	0.099	0.867	0.962
	49.6	1.79	2.15	0.203	0.221	1.99	2.37
	135	4.72	5.66	0.558	0.693	5.28	6.35
	(+3 days depuration)		0.045		0.141		0.186
	(+7 days depuration)		< 0.01		< 0.020		< 0.030
	(+14 days depuration)		< 0.01		< 0.020		< 0.030

Animal Commodity	Dose Level of Flupyradifurone (ppm)		Flupyradifurone Residue (mg/kg)		DFA Residue (mg/kg) [a]		Flupyradifurone + DFA Residue as Parent Equivalents (mg/kg)	
			Mean	Highest	Mean	Highest	Mean	Highest [b]
Liver	4.81		0.145	0.172	< 0.020	< 0.020	0.165	0.192
	23.1		0.755	0.821	0.057	0.071	0.812	0.892
	49.6		1.68	2.00	0.132	0.169	1.81	2.17
	135		3.45	3.89	0.399	0.507	3.85	4.40
	(+3 days depuration)			0.033		0.106		0.139
	(+7 days depuration)			< 0.01		< 0.020		< 0.030
	(+14 days depuration)			< 0.01		< 0.020		< 0.030
Muscle	4.81		0.043	0.048	< 0.020	< 0.020	0.063	0.068
	23.1		0.250	0.260	0.054	0.066	0.304	0.317
	49.6		0.597	0.740	0.136	0.170	0.733	0.910
	135		1.51	1.88	0.385	0.473	1.89	2.28
	(+3 days depuration)			0.017		0.095		0.112
	(+7 days depuration)			< 0.01		< 0.020		< 0.030
	(+14 days depuration)			< 0.01		< 0.020		< 0.030
Milk	4.8	Day 28	0.023	0.026	< 0.020	< 0.020	0.043	0.046
	23.1	Day 28	0.108	0.125	0.021	0.023	0.129	0.148
	49.6	Day 28	0.267	0.290	0.041	0.046	0.308	0.336
	135	Day 2	0.746	0.895	0.081	0.092	0.827	0.987
		Day 4	0.869	1.656	0.105	0.144	0.973	1.75
		Day 7	0.688	0.905	0.138	0.236	0.826	1.05
		Day 10	0.763	0.983	0.137	0.232	0.900	1.14
		Day 14	0.783	0.915	0.151	0.249	0.935	1.08
		Day 17	0.831	1.019	0.143	0.241	0.974	1.26
		Day 19	0.825	1.102	0.130	0.185	0.955	1.29
		Day 25	0.651	0.730	0.115	0.192	0.765	0.871
		Day 28	0.748	0.919	0.138	0.266	0.886	1.09
		Day 29	0.667	0.743	0.140	0.165	0.806	0.856
		Day 30 Depuration	0.059	0.071	0.078	0.104	0.137	0.147
		Day 31 Depuration	< 0.01	< 0.01	0.043	0.053	0.053	0.063
		Day 35 Depuration	< 0.01	< 0.01	< 0.020	< 0.020	< 0.030	< 0.030
Cream (Day 25)	135		0.553	0.604	0.05	0.068	0.603	0.672
Whey (Day 25)	135		0.758	0.803	0.123	0.151	0.881	0.954

[a] Calculated as parent equivalents.

[b] The highest total residues are from one animal, not from the addition of highest flupyradifurone + DFA from more than one animal

Laying hen transfer study (Wade and Netzband 2012, RARVP041)

Eighty-four mature laying hens were dosed orally, *via* capsule, for 29 consecutive days with flupyradifurone at dose rates of 0 ppm (control, 12 hens, Group 1), 1.5 ppm (12 hens, Group 2, average of 0.10 mg ai/kg bw/day), 6.5 ppm (12 hens, Group 3, average of 0.45 mg ai/kg bw day), 19.4 ppm (12 hens, Group 4, average of 1.31 mg/kg bw/day) and 65.1 ppm (24 hens, Group 5, average of 4.54 mg/kg bw/day). The average feed consumption (fresh weight) was 128 g/bird/day. The weight of the hens ranged from 1.15–1.64 kg at the start of dosing and from 1.25–1.73 kg at termination.

Eggs were collected twice daily (afternoon and morning). The eggs collected in the afternoon from each sub-group were combined with the eggs collected the following morning from the same sub-group. On Day 29 of the study, twelve hens from the control and Group 5, and all hens in the Group 2–4 dose groups were sacrificed by CO_2 asphyxiation within 24 hours of the administration of the final dose. Liver (entire), muscle (thigh and breast), and fat (abdominal and subcutaneous) were collected. Twenty-four hens (12 from the control group and 12 from Group 5) entered a 21-day depuration phase following the administration of the final dose. Egg and tissue samples were collected on study days 35, 42, and 49 for analysis.

Residues of flupyradifurone and its metabolites BYI 02960-hydroxy, BYI 02960-acetyl-AMCP and difluoroacetic acid were quantitated using LC/MS/MS Method RV-004-A11-04. The LOQ was 0.01 mg/kg for each analyte in eggs and poultry tissues. Residues in eggs are summarized below in Table 165. Residues in eggs during the depuration phase are summarized in Table 166. Residues in tissues are summarized in Table 167, with the depuration phase in Table 168.

Table 165 Flupyradifurone residues in eggs

Sampling Day / Group	Flupyradifurone (mg/kg)	DFA [a] (mg/kg)	BYI 02960-AMCP [a] (mg/kg)	BYI 02960-OH [a] (mg/kg)	Total (Flupyradifurone + DFA) residues (mg/kg)
Eggs: LOD	0.004	0.003	0.003	0.003	
0 Day/65.1 ppm	< LOD	< LOD	< LOD	< LOD	< LOD
	< LOD	< LOD	< LOD	< LOD	< LOD
	< LOD	< LOD	< LOD	< LOD	< LOD
	< LOD	< LOD	< LOD	< LOD	< LOD
	< LOD	< LOD	< LOD	< LOD	< LOD
	< LOD	< LOD	< LOD	< LOD	< LOD
Average					< LOD
2 Day/65.1 ppm	0.057	0.381	0.018	0.033	0.438
	0.049	0.345	0.019	0.023	0.394
	0.034	0.342	0.023	0.022	0.376
	0.060	0.358	0.019	0.023	0.418
	0.036	0.266	0.014	0.016	0.302
	0.052	0.310	0.019	0.024	0.362
Average					0.382
4 Day/65.1 ppm	0.038	0.808	0.037	0.021	0.846
	0.095	1.000	0.060	0.041	1.095
	0.053	0.952	0.051	0.022	1.005
	0.056	0.977	0.043	0.020	1.033
	0.051	0.757	0.038	0.017	0.808
	0.112	0.896	0.043	0.038	1.008
Average					0.966
7 Day/65.1 ppm	0.047	1.070	0.042	0.022	1.117
	0.072	1.100	0.055	0.041	1.172
	0.044	1.030	0.044	0.026	1.074
	0.045	1.070	0.033	0.022	1.115
	0.057	0.933	0.041	0.023	0.990
	0.056	0.930	0.036	0.020	0.986
Average					1.076
10 Day/65.1 ppm	0.062	1.350	0.053	0.044	1.412
	0.063	1.260	0.057	0.040	1.323
	0.070	1.270	0.059	0.051	1.340
	0.063	1.340	0.056	0.042	1.403
	0.048	0.977	0.039	0.029	1.025
	0.081	1.070	0.049	0.040	1.151
Average					1.276
14 Day/65.1 ppm	0.077	1.030	0.041	0.039	1.107
	0.075	1.120	0.046	0.049	1.195
	0.068	0.982	0.043	0.046	1.050
	0.044	1.030	0.035	0.026	1.074
	0.043	0.735	0.027	0.028	0.778
	0.070	0.934	0.037	0.038	1.004
Average					1.035
17 Day /65.1 ppm	0.091	1.020	0.040	0.053	1.111
	0.128	1.320	0.049	0.078	1.448

Sampling Day / Group	Flupyradifurone (mg/kg)	DFA [a] (mg/kg)	BYI 02960-AMCP [a] (mg/kg)	BYI 02960-OH [a] (mg/kg)	Total (Flupyradifurone + DFA) residues (mg/kg)
Eggs: LOD	0.004	0.003	0.003	0.003	
	0.082	1.090	0.047	0.060	1.172
	0.053	1.240	0.041	0.035	1.293
	0.065	1.170	0.039	0.049	1.235
	0.063	1.180	0.040	0.052	1.243
Average					1.250
21 Day/65.1 ppm	0.055	1.220	0.044	0.040	1.275
	0.098	1.200	0.05	0.065	1.298
	0.081	1.290	0.041	0.064	1.371
	0.049	1.230	0.029	0.036	1.279
	0.055	1.160	0.046	0.044	1.215
	0.089	1.110	0.048	0.052	1.199
Average					1.273
24 Day/1.5 ppm	< LOD	0.053	< LOD	< LOD	0.057
	< LOD	0.049	< LOD	< LOD	0.053
	< LOD	0.05	< LOD	< LOD	0.054
Average					0.055
24 Day/6.5 ppm	< LOD	0.158	0.006	< LOD	0.162
	< LOD	0.169	0.006	0.005	0.173
	< LOD	0.137	0.007	0.003	0.141
Average					0.159
24 Day/19.4 ppm	0.011	0.449	0.014	0.01	0.460
	0.025	0.508	0.018	0.016	0.533
	0.020	0.534	0.018	0.015	0.554
Average					0.516
24 Day/65.1 ppm	0.070	1.390	0.053	0.048	1.460
	0.069	1.460	0.068	0.044	1.529
	0.114	1.580	0.056	0.058	1.694
	0.096	1.680	0.087	0.067	1.776
	0.128	1.470	0.056	0.063	1.598
	0.125	1.520	0.060	0.059	1.645
	0.048	1.350	0.039	0.034	1.398
	0.054	1.510	0.042	0.037	1.564
	0.046	1.390	0.082	0.043	1.436
	0.05	1.410	0.056	0.030	1.460
	0.095	1.570	0.054	0.059	1.665
	0.085	1.500	0.059	0.055	1.585
Average					1.568
28 Day/1.5 ppm	< LOD	0.058	< LOD	< LOD	0.0548
	< LOD	0.0394	< LOD	0.0031	0.0434
	< LOD	0.0498	< LOD	< LOD	0.0538
Average					0.057
28 Day/6.5 ppm	0.007	0.171	0.006	0.009	0.178
	0.004	0.160	0.008	0.004	0.164
	0.008	0.158	0.007	0.009	0.166
Average					0.169
28 Day/19.4 ppm	0.020	0.496	0.013	0.018	0.516
	0.01	0.474	0.017	0.011	0.484
	0.040	0.555	0.016	0.026	0.595
Average					0.532
28 Day/65.1 ppm	0.169	1.370	0.053	0.082	1.539
	0.167	1.450	0.064	0.084	1.617
	0.201	1.670	0.056	0.103	1.871
	0.185	1.690	0.067	0.091	1.875
	0.067	1.540	0.056	0.05	1.607
	0.069	1.480	0.052	0.055	1.549
	0.283	1.420	0.041	0.089	1.703
	0.243	1.300	0.034	0.113	1.543

Sampling Day / Group	Flupyradifurone (mg/kg)	DFA [a] (mg/kg)	BYI 02960-AMCP [a] (mg/kg)	BYI 02960-OH [a] (mg/kg)	Total (Flupyradifurone + DFA) residues (mg/kg)
Eggs: LOD	0.004	0.003	0.003	0.003	
	0.181	1.310	0.040	0.093	1.491
	0.144	1.130	0.039	0.080	1.274
	0.191	1.320	0.051	0.077	1.511
	0.174	1.290	0.056	0.094	1.464
Average					1.587

[a] Calculated as parent equivalents.

Table 166 Residues in eggs in the depuration phase

Sampling Day / Group	Flupyradifurone (mg/kg)	DFA [a] (mg/kg)	BYI 02960-AMCP [a] (mg/kg)	BYI 02960-OH [a] (mg/kg)	Total (Flupyradifurone + DFA) residues (mg/kg)
Eggs: LOD	0.004	0.003	0.003	0.003	
35 Day/65.1 ppm	0.0088	0.1010	< LOD	0.0039	0.1098
	< LOD	0.1580	< LOD	< LOD	0.162
	< LOD	0.1320	< LOD	0.0030	0.1360
Average					0.136
42 Day/65.1 ppm	< LOD	0.0064	< LOD	< LOD	0.014
	< LOD	0.0079	< LOD	< LOD	0.0119
Average					0.0112
49 Day/ 65.1 ppm	< LOD	< LOD	< LOD	< LOD	< 0.007

[a] Calculated as parent equivalents.

Table 167 Flupyradifurone residues in chicken tissues

Sampling Day / Group	Flupyradifurone (mg/kg)	DFA [a] (mg/kg)	BYI 02960-AMCP [a] (mg/kg)	BYI 02960-OH [a] (mg/kg)	Flupyradifurone + DFA (mg/kg)
Fat LOD:	0.003	0.002	0.003	0.002	
29 Day/1.5 ppm	< LOD	0.0350	< LOD	< LOD	0.0380
	< LOD	0.0265	< LOD	< LOD	0.0295
	< LOD	0.0253	< LOD	< LOD	0.0283
Average					0.0319
29 Day/6.5 ppm	< LOD	0.1130	< LOD	< LOD	0.116
	< LOD	0.1240	< LOD	< LOD	0.127
	< LOD	0.1150	< LOD	< LOD	0.118
Average					0.120
29 Day/19.4 ppm	< LOD	0.2870	0.0070	< LOD	0.290
	< LOD	0.2540	0.0056	< LOD	0.257
	< LOD	0.2760	0.0053	< LOD	0.279
Average					0.275
29 Day/65 ppm	0.5550	0.8580	0.0182	0.0152	1.413
	0.0086	0.9710	0.0166	0.0023	0.9796
	0.0134	1.1900	0.0291	0.0137	1.203
Average					1.199
Liver LOD:	0.003	0.003	0.003	0.002	
29 Day/1.5 ppm	< LOD	0.1120	< LOD	< LOD	0.1150
	< LOD	0.1010	< LOD	< LOD	0.1040
	< LOD	0.0988	< LOD	< LOD	0.1018
Average					0.1069
29 Day/6.5 ppm	< LOD	0.4100	0.017	< LOD	0.413
	0.0115	0.4040	0.018	0.0144	0.4155
	< LOD	0.4240	0.0112	< LOD	0.427
Average					0.4185
29 Day/19.4 ppm	< LOD	0.9570	0.0222	< LOD	0.9600

Sampling Day / Group	Flupyradifurone (mg/kg)	DFA [a] (mg/kg)	BYI 02960-AMCP [a] (mg/kg)	BYI 02960-OH [a] (mg/kg)	Flupyradifurone + DFA (mg/kg)
	< LOD	1.0000	0.0270	0.0083	1.003
	< LOD	1.0800	0.0257	0.0047	1.083
Average					1.015
29 Day/65 ppm	0.0614	2.9700	0.0625	0.0728	3.031
	0.0060	3.7400	0.1000	0.0259	3.746
	0.0293	3.2300	0.0851	0.0557	3.259
Average					3.345
Muscle LOD:	0.004	0.003	0.003	0.003	
29 Day/ 1.5 ppm	< LOD	0.0959	0.0031	< LOD	0.0999
	< LOD	0.0832	< LOD	< LOD	0.0872
	< LOD	0.0694	< LOD	< LOD	0.0734
Average					0.0868
29 Day/6.5 ppm	< LOD	0.2770	0.0094	< LOD	0.281
	< LOD	0.2910	0.010	< LOD	0.295
	< LOD	0.3030	0.016	< LOD	0.3070
Average					0.294
29 Day/19.4 ppm	< LOD	0.6950	0.0178	< LOD	0.6990
	< LOD	0.6830	0.0291	0.0057	0.687
	0.0049	0.7780	0.0258	0.0057	0.7829
Average					0.7230
29 Day/65 ppm	0.0583	1.590	0.0404	0.0075	1.648
	0.013	2.500	0.0753	0.0302	2.510
	0.0483	2.720	0.0904	0.0580	2.768
Average					2.309

[a] Values reported in parent equivalents.

Table 168 Residues in chicken tissues depuration phase

Sampling Day / Group	Flupyradifurone (mg/kg)	DFA [a] (mg/kg)	AMCP [a] (mg/kg)	BYI 02960-OH [a] (mg/kg)	Total (Flupyradifurone + DFA) residues (mg/kg)
Fat LOD:	0.003	0.002	0.003	0.002	
35 Day (65 mg/kg feed)	< LOD	0.0407	< LOD	< LOD	0.0437
42 Day (65 mg/kg feed)	< LOD	0.0053	< LOD	< LOD	0.0083
49 Day (65 mg/kg feed)	< LOD	0.0086	< LOD	< LOD	0.0116
Liver LOD:	0.003	0.003	0.003	0.002	
35 Day (65 mg/kg feed)	< LOD	0.0848	< LOD	< LOD	0.0878
42 Day (65 mg/kg feed)	< LOD	0.0078	< LOD	< LOD	0.018
49 Day (65 mg/kg feed)	< LOD	< LOD	< LOD	< LOD	< 0.006
Muscle LOD:	0.004	0.003	0.003	0.003	0.0040
35 Day (65 mg/kg feed)	< LOD	0.052	< LOD	< LOD	0.0542
42 Day (65 mg/kg feed)	< LOD	0.0060	< LOD	< LOD	0.01
49 Day (65 mg/kg feed)	< LOD	< LOD	< LOD	< LOD	< 0.007

[a] Values reported in parent equivalents.

The results of the study are summarized in the following table.

Table 169 Residues of flupyradifurone and DFA in eggs and tissues

Animal Commodity	Dose Level of Flupyradifurone (ppm)		Flupyradifurone Residue (mg/kg)		DFA Residue [a] (mg/kg)		Flupyradifurone + DFA Residues as Parent Equivalents (mg/kg)	
			Mean	Highest	Mean	Highest	Mean	Highest [b]
Egg	1.5	Day 24	< 0.004	< 0.004	0.051	0.053	0.055	0.057
		Day 28	< 0.004	< 0.004	0.047	0.058	0.057	0.0548
	6.5	Day 24	< 0.004	< 0.004	0.155	0.169	0.159	0.173
		Day 28	0.006	0.008	0.163	0.171	0.169	0.178
	19.4	Day 24	0.019	0.025	0.497	0.534	0.516	0.554
		Day 28	0.023	0.040	0.508	0.555	0.532	0.595
	65.1	Day 0	< 0.004	< 0.004	< 0.003	< 0.003	< 0.007	< 0.007
		Day 2	0.048	0.060	0.334	0.381	0.382	0.438
		Day 4	0.068	0.112	0.898	1.000	0.966	1.10
		Day 7	0.054	0.072	1.02	1.100	1.08	1.17
		Day 10	0.065	0.081	1.21	1.350	1.28	1.41
		Day 14	0.063	0.077	0.972	1.120	1.04	1.20
		Day 17	0.080	0.128	1.17	1.320	1.25	1.45
		Day 21	0.071	0.098	1.202	1.290	1.27	1.37
		Day 24	0.082	0.128	1.49	1.680	1.57	1.78
		Day 28	0.173	0.283	1.414	1.690	1.59	1.88
		Day 35 Depuration	0.0056	0.0088	0.130	0.1580	0.136	0.162
		Day 42 Depuration	< 0.004	< 0.004	0.0072	0.0079	0.0112	0.0119
		Day 49 Depuration		< 0.004		< 0.003		< 0.007
Fat	1.5		< 0.003	< 0.003	0.0289	0.0350	0.0319	0.0380
	6.5		< 0.003	< 0.003	0.117	0.1240	0.120	0.127
	19.4		< 0.003	< 0.003	0.272	0.2870	0.275	0.290
	65.1		0.192	0.5550	1.006	1.190	1.20	1.41
	(+6 days depuration)			< 0.003		0.0407		0.0437
	(+13 days depuration)			< 0.003		0.0053		0.0083
	(+20 days depuration)			< 0.003		0.0086		0.0116
Liver	1.5		< 0.003	< 0.003	0.1039	0.1120	0.107	0.115
	6.5		0.0058	0.0115	0.4127	0.4240	0.419	0.427
	19.4		< 0.003	< 0.003	1.012	1.080	1.02	1.08
	65.1		0.0322	0.0614	3.313	3.7400	3.35	3.75
	(+6 days depuration)			< 0.003		0.0848		0.0878
	(+13 days depuration)			< 0.003		0.0078		0.018
	(+20 days depuration)			< 0.003		< 0.003		< 0.006
Muscle	1.5		< 0.004	< 0.004	0.0828	0.0959	0.0868	0.0999
	6.5		< 0.004	< 0.004	0.2903	0.3030	0.294	0.307
	19.4		0.0043	0.0049	0.7187	0.7780	0.723	0.783
	65.1		0.0390	0.0583	2.270	2.720	2.31	2.77
	(+6 days depuration)			< 0.004		0.052		0.0542
	(+13 days depuration)			< 0.004		0.0060		0.01
	(+20 days depuration)			< 0.004		< 0.003		< 0.007

[a] Values reported in parent equivalents.

[b] The highest total residues are from one animal, not from the addition of highest flupyradifurone + DFA from more than one animal

NATIONAL RESIDUE DEFINITIONS

USA, Canada, Mexico, Japan:

Definition of the residue (for compliance with the MRL for plant and animal commodities): *Flupyradifurone*

Europe:

Definition of the residue (for compliance with the MRL for plant and animal commodities):

Two separate definitions–*Flupyradifurone, expressed as flupyradifurone* and *Difluoroacetic acid (DFA), expressed as DFA*

USA, Canada, Mexico, Europe, Japan:

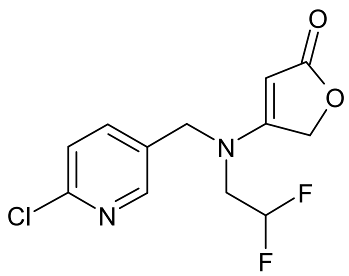
Definition of the residue (for estimation of dietary intake for plant and animal commodities): *Flupyradifurone and difluoroacetic acid, expressed as flupyradifurone*

APPRAISAL

Flupyradifurone (fpd) is an insecticide belonging to the chemical class of butenolides. It acts as an agonist of the nicotinic acetylcholine receptor. It was scheduled for evaluation as a new compound by the 2015 JMPR at the 46th Session of the CCPR (2014). It was evaluated for toxicology in 2015. An ADI of 0–0.08 mg/kg bw and an ARfD of 0.2 mg/kg bw were established.

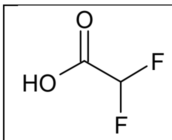
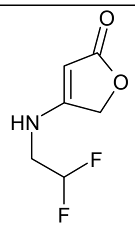
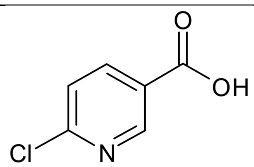
The manufacturer supplied information on identity, metabolism and environmental fate, methods of residue analysis, freezer storage stability, registered use patterns, supervised residue trials, fate of residues in processing and farm animal feeding studies.

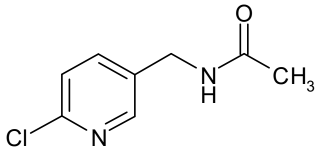
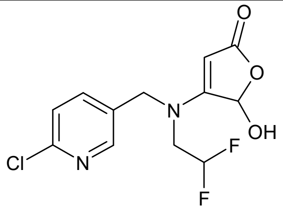
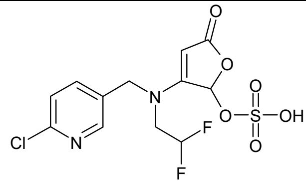
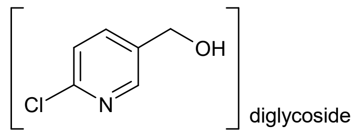
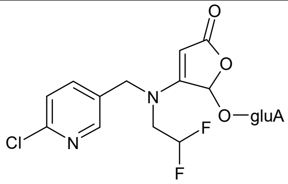
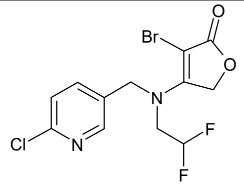
The IUPAC name is 4-[(6-chloro-3-pyridylmethyl)(2,2-difluoroethyl)amino]furan-2(5H)-one.



Flupyradifurone

The structure of the key metabolites discussed, are shown below:

		
Difluoroacetic acid (DFA)	fpd-difluoroethyl-amino-furanone (DFEAF)	6- chloronicotinic acid (6-CNA)

 fpd-acetyl-AMCP	 fpd-OH	 fpd-OH-SA
 fpd-CHMP-di-glyc	 fpd-OH-glu	 fpd-bromo

Plant Metabolism

Flupyradifurone (fpd) metabolism in primary crops was investigated following either foliar applications (apple, cotton, and rice), soil granule/drench applications (tomato, potato, and rice) or by seed dressing (potato). Studies were conducted using [^{14}C]flupyradifurone labelled on the furanone or pyridinylmethyl moiety and using application rates representative of the supported uses. One study on tomato using soil drench applications and with ^{14}C-labelling on the difluoroethyl amino group was also submitted.

Acetonitrile and water extraction of tomato, potatoes, apples, cotton and paddy rice resulted in extraction efficiencies of 84.8–99.5% (tomato fruit), 93.6–98.3% (tomato flowers), 67.0–93.4% (potatoes), 59.8–98.7% (apple fruits), 94.3–98.4% (apple leaves), 66.9–89.7% (gin trash), 96.6–99.2% (cotton lint), 21.9–38.5% (cotton seed), 19.2–88.4% (rice kernel), 75.6–90.4% (rice husks) and 79.0–83.7% (rice straw).

[Furanone-4-^{14}C]-fpd or [pyridinylmethyl-^{14}C]-fpd was applied twice by soil drench applications to four tomato plants at rates of 300 g ai/ha at BBCH 15 (= 5th leaf on main shoot unfolded) and BBCH 51 (= first inflorescence visible and first bud erect). Parent was the main residue in both fruit and flowers (24–36% and 66–78% of the TRR respectively). The compound fpd-CHMP-di-glyc was observed in tomato fruit at 37% TRR. In a similar study, three tomato plants were treated twice at 300 g ai/ha by soil drench application with [ethyl-1-^{14}C]-fpd. The label specific metabolite difluoroacetic acid (DFA) was the main residue in both extracts (87% TRR in fruit and 60% TRR in flowers). Parent was present at 10% TRR in fruit and 33% TRR in flowers.

Two different methods of application to potatoes were described. In one experiment potato seed pieces were treated with either [furanone-4-^{14}C]-fpd or [pyridinylmethyl-^{14}C]-fpd at 254 or 274 g ai/ha. In the other experiment either [furanone-4-^{14}C]-fpd or [pyridinylmethyl-^{14}C]-fpd was sprayed in-furrow onto soil at 626 g ai/ha prior to planting of potato seed pieces. Parent was the main residue in extracts of both seed piece and in-furrow treatment for both labels (40–51% of the TRR). For the furanone label two metabolites, difluoroethyl-amino-furanone (DFEAF) and fpd-OH-glyc, were present at < 10% TRR (< 0.01 mg/kg equiv.). For the pyridinylmethyl label a number of metabolites were identified, the major being 6-chloropyridine-3-carboxylic acid (6-chloronicotinic acid or 6-CNA) at 18 and 22% TRR.

Apple trees were subjected to two different methods of application. In one experiment two apple trees were treated once with either [furanone-4-^{14}C]-fpd or [pyridinylmethyl-^{14}C]-fpd at 86 or 87 g ai/ha/ per metre canopy height at the end of flowering (BBCH 69). In another experiment, one apple tree was treated twice with both labelled parent at 86 or 87 g ai/ha/ per metre canopy height at the end of flowering (BBCH 69) and at 14 days before harvest. The main compound in apple fruits from the single application furanone experiment was glucose (or a corresponding isomeric carbohydrate) at 50% TRR while parent was detected at 7.4% TRR. In apple leaves, parent was detected at 26% TRR. Glucose was also a major component found in the apple fruits from the double application experiment (14% TRR. Parent was the main component in the extracts of apple fruits (71–74% TRR and leaves (58% TRR) of the double application furanone experiment. Parent was the main compound in apple fruits and leaves of the single (43% and 25% TRR respectively) and the double application (86–88% and 48% TRR respectively) experiments using the pyridinylmethyl label.

Two different methods of application to cotton were described. In one experiment cotton plants were treated once with either [furanone-4-^{14}C]-fpd or [pyridinylmethyl-^{14}C]-fpd at 209 or 206 g ai/ha at BBCH 16. In another type of experiment, one cotton plant was treated twice at 209 g ai/ha (furanone label) or 206 g ai/ha (pyridinylmethyl label) at BBCH 15–16 (unfolding of the 5th to 8th true leaf) and a second time at 176–177 g ai/ha at 14 or 15 days before harvest of the cotton bolls (BBCH 95–97). In both experiments cotton seeds with lint, lint and gin trash were harvested at maturity of seed (BBCH 99). The main compound in all cotton matrices for experiments with both labels, except for seeds of the single application experiment, was parent (23–73% TRRs), even after only one treatment with a long PHI (169 days). The fraction comprising the metabolites fpd-OH-glyc and fpd-acetic acid represented a major portion of the TRR in all sample matrices, except for seeds, while another major compound in gin trash of the first application experiment was identified as fpd-OH. In seeds of the single application pyridinylmethyl label experiment, 6-CNA was the only component identified (16.2% TRR). It was also observed in gin trash of the single application pyridinylmethyl label experiment (18.5% TRR).

Two different methods of application to paddy rice were described. One consisted of a single granular treatment with either 409 g ai/ha [furanone-4-^{14}C]-fpd or 434 g ai/ha [pyridinylmethyl-^{14}C]-fpd applied during the transplanting of the rice seedlings. In the second experiment, [furanone-4-^{14}C]-fpd or [pyridinylmethyl-^{14}C]-fpd was applied twice as a spray treatment onto plants as well as the water surface. The first spray application took place directly after transplanting of the rice seedlings at a rate of 175–178 g ai/ha and the second approximately one month before harvest at a rate of 236–240 g ai/ha. In both experiments, the rice (kernels, husks and straw) was harvested at maturity (BBCH 89–92) at 127 DAT (granular) or 29 DAT (foliar). The main compound was parent in all rice matrices (57–78% of the TRR), except for rice kernels after granular application for the furanone label (23% TRR), where the main metabolite was the natural compound glucose/carbohydrates (27% TRR). In rice straw for both labels, fpd-bromo which was identified as the only major metabolite present (8–12% TRR) co-eluted with fpd-chloro.

A study was conducted to determine the fate of the difluoroethane moiety of parent flupyradifurone by determination of the non-radiolabelled difluoroacetic acid (DFA) content of extracts from the plant metabolism studies (apples, potatoes, cotton and rice) conducted with either [furanone-4-^{14}C]- or [pyridinylmethyl-^{14}C]-fpd. The application technique did not significantly influence the level, with residues of a similar order of magnitude after soil or foliar application. Residues in apple fruits were 0.04–0.23 mg/kg, in apple leaves 0.45–0.62 mg/kg, in potato tubers 0.13–0.18 mg/kg, in cotton seeds 0.02–0.03 mg/kg, in cotton gin trash 0.02–0.04 mg/kg, in rice straw 0.12–0.39 mg/kg, in rice husk 0.20–0.46 mg/kg and in rice grains 0.02–0.08 mg/kg.

High difluoroacetic acid concentrations after foliar spray application indicate that this metabolite is also formed in plants and not only in soil. Difluoroacetic acid represents a significant proportion of the residue in all edible matrices of primary crops when considering the results of the studies conducted with [^{14}C]flupyradifurone.

Summary of plant metabolism

Metabolism in primary crops was similar in all plant groups investigated. In the studies using [furanone-4-^{14}C]-fpd and [pyridinylmethyl-^{14}C]-fpd, parent flupyradifurone was consistently observed to be the major component of the radioactive residues, accounting for approximately 23 to 88% of the TRR in all plant parts analysed. As well as flupyradifurone, the following metabolites were identified in different plant matrices, the conjugate fpd-CHMP-diglycoside, up to 37% TRR (0.05 mg/kg equiv.) in tomato fruit and the metabolite 6-CNA in the range of 13–22% TRR (0.02 mg/kg equiv.) in tomato fruit, potato tuber and cotton seed, both resulting from the cleavage of the molecule at the ethylamine bond and containing the pyridinyl moiety. In contrast, metabolites containing the furanone moiety were close to non-detect and the radioactivity in the [^{14}C]furanone studies was mostly recovered as incorporated in natural glucoside and carbohydrate components, indicating an extensive degradation of the furanone counterpart.

The study conducted on tomato with the ^{14}C labelling on the difluoroethyl amino group showed that following soil drench application, significant proportions (87% TRR) and levels (0.17 mg/kg) of difluoroacetic acid (DFA) were present in tomato fruits. Samples from the radiolabelled studies were therefore re-analysed for non-radiolabelled DFA and residues, expressed as DFA equivalents, were measured in the range of 0.02–0.23 mg/kg in apple fruits, potato tuber, cotton seed and rice grain, irrespective of mode of application.

Confined rotational crop studies

Studies were undertaken to investigate the metabolism of flupyradifurone in the representative crops wheat, Swiss chard and turnips from three consecutive rotations using either [furanone-4-^{14}C]-fpd or [pyridinylmethyl-^{14}C]-fpd sprayed onto the soil of a planting container at 436 and 433 g ai/ha respectively. The crops were each sown at 29, 135 and 296 days after the soil application, representing the first, second and third rotation. Raw agricultural commodities sampled included the immature samples forage and hay from wheat, and the immature samples from Swiss chard. All other samples (wheat straw and wheat grain, Swiss chard, turnip leaves and turnip roots) were harvested in each rotation at maturity. No studies were undertaken with labelling on the difluoroethyl amino group. A similar metabolic profile as observed for primary crops was observed in the confined rotational studies.

Parent compound and about 30 (furanone label) or 50 (pyridinylmethyl label) metabolites were detected in the conventional and exhaustive extracts of the various samples of the three rotations. Parent compound was the main component detected in all matrices of all rotations, except for wheat grain. Parent accounted for 34–64% (furanone label) or 28–62% (pyridinylmethyl label) of the TRR in the commodities at a 29 day PBI, 28–68% (furanone label) or 25–67% (pyridinylmethyl label) TRR at a 135 day PBI, and 18–72% (furanone label) or 20–69% (pyridinylmethyl label) TRR at a 296 day PBI, not considering grains. In wheat grains, only trace amounts of parent compound were detected (< 1% and 2% of the TRR) in the furanone label experiments and 1–14% of the TRR in the pyridinylmethyl label experiments. The highest proportion of parent compound was always detected in turnip leaves.

Samples from the confined rotational studies were also analysed for the presence of non-radiolabelled difluoroacetic acid (DFA). Significant levels of DFA were detected in all plant matrices of the first and second rotation except turnip roots. DFA represented a major proportion of the residues in the edible crops wheat grain, Swiss chard and turnip roots, as well as in wheat hay.

Field rotational crops

In the USA the total maximum seasonal application rate for a large range of crops is 409 g ai/ha. In Central America the total maximum seasonal application rate for potatoes, tomatoes and chilli peppers and melon, cucumbers and watermelons is 600 g ai/ha. Field crop rotational trials conducted in Europe have been made available to the Meeting.

In a European study, applications were either made to bare soil or to lettuce (primary crop or target crop) at 200 g ai/ha. In each rotation (25–33 days, 60–200 days and 260–330 days), three different crops were planted: a root crop (carrots or turnips), a leafy crop (lettuce), or a cereal (barley). Samples of the rotational crops were taken at their harvest times, as well as at one earlier interval and were analysed for residues of parent, DFA, DFEAF and 6-CNA.

In general parent compound was either absent or present at low levels (< 0.1 mg/kg). DFEAF was not detected in all commodities at all PHIs, while DFA was observed in most commodities at all PHIs (up to 0.63 mg/kg in cereal grain and up to 0.12 mg/kg in lettuce and root crops). Low residues of 6-CNA were detected in barley straw only.

Additional European studies involved two applications to bare soil at 125 g ai/ha with the rotational crops potatoes, cucumber, leek, French bean, onion, pea and winter rape planted approximately 30 days after the last application. Samples were taken at typical harvest maturity. No residues of parent or DFEAF were detected. Residues of 6-CNA were < 0.005 mg/kg (as 6-CNA) except in bean pods (up to 0.016 mg/kg) and pea dry (up to 0.017 mg/kg). DFA residues were detected in most samples: up to 0.25 mg/kg in potato; up to 0.23 mg/kg in leek; up to 0.41 mg/kg in cucumber; up to 0.16 mg/kg in onion, up to 1.1 mg/kg in French bean; up to 2.3 mg/kg in field pea and up to 0.15 mg/kg in winter rape.

The field rotational crop studies indicate that while parent and 6-CNA may be observed at low levels, DFA is the major component of the residues.

Environmental fate in soil

The Meeting received information on soil photolysis, aerobic and anaerobic soil metabolism, field dissipation and adsorption/desorption behaviour in different soils. Only those studies relevant to the current evaluation were considered.

A soil photolysis study showed that light was found to have little influence on the behaviour and degradation of flupyradifurone in soils. Flupyradifurone was found to be hydrolytically stable in aqueous solution at pH 4 to pH 9 (50 °C) for five days.

Aerobic laboratory studies were conducted in numerous soils from the USA, Europe and Brazil at 20 ± 2 °C and for periods of 117–120 days, using four different labelling positions. The studies showed that flupyradifurone is degraded in soil under aerobic conditions. The major routes of degradation are cleavage of the difluoroethyl group producing difluoroacetic acid, cleavage of the molecule at the pyridinylmethyl bridge, with subsequent oxidation to 6-chloronicotinic acid and mineralisation to CO_2 and formation of non-extractable residues. DT_{50} values ranged from 33 to 374 days, while DT_{90} values ranged from 209 to > 1000 days. Degradation of the flupyradifurone metabolite, 6-chloronicotinic acid (6-CNA), was studied under aerobic conditions in three English soils over 119 days. It was seen to degrade to CO_2 (84–92% of the applied dose at the end of the study). The rapid breakdown of 6-CNA (DT_{50} values 2.9 to 5.3 days) demonstrated that it will not persist in aerobic soils. DT_{50} values for difluoroacetic acid ranged from 32.0–73.6 days while DT_{90} values ranged from 149.0–244.5 days.

Field dissipation studies were carried out at six sites in Europe and three sites in both Canada and the USA. In general, the field behaviour of flupyradifurone was consistent with the model developed from the laboratory studies. The studies showed that the major routes of dissipation for flupyradifurone are biodegradation to 6-chloronicotinic acid and difluoroacetic acid and non-extractable residues followed by mineralisation to carbon dioxide. When DFA and 6-CNA were detected, the amounts always decreased over the period of the study. Flupyradifurone dissipated with DT_{50} values of 8 to 251 days in the field studies indicating that flupyradifurone has a potential for residue carry over to the following cropping season if application is performed annually.

Animal metabolism

The Meeting received animal metabolism studies with flupyradifurone in rats, hens and goats.

Rats

Evaluation of the metabolism studies in rats was carried out by JMPR 2015 and is not fully considered here.

In studies conducted in rats using [^{14}C]flupyradifurone, the majority (up to 90%) of radioactivity was excreted in urine within 24 hours. There was no evidence of tissue accumulation. While parent was the main compound detected in excreta (up to 50% of radioactivity in males and 70% in females) flupyradifurone was metabolized to eight identified metabolites and up to 19 unidentified metabolites involving hydroxylation, conjugation and cleavage reactions. The extent of metabolism was greater in male than female rats. Significant plant metabolites, including DFA, DFEAF, 6-CNA and fpd-OH were also observed in the rat metabolism studies.

Goats

Two studies on the metabolism of flupyradifurone were conducted with the test compound labelled in either the [furanone-4-^{14}C] or the [pyridinylmethyl-^{14}C]-position. In each study a lactating goat was dosed orally once daily for five consecutive days, at 28.8 or 24.4 ppm respectively. Milk was sampled twice daily, in the morning and afternoon. Animals were sacrificed approximately 6 hours after the last dose.

A total of 72 and 85% of the total administered dose was eliminated in the faeces and urine (0–102 hours) while TRRs in tissues were 1.7 and 1.2 mg/kg equiv. in liver, 1.5 and 1.9 mg/kg equiv. in kidney, 0.54 and 0.36 mg/kg equiv. in muscle and 0.27 and 0.11 mg/kg equiv. in total fat for the furanone and pyridinylmethyl labels respectively.

Acetonitrile and water extraction of liver, kidney, muscle, fat and milk resulted in extraction efficiencies of 73.3–92.8% (liver), 89.1–98.8% (kidney), 88.4–99.2% (fat), 95.0–99.4% (muscle) and 90.7–99.3% (milk).

Metabolism in ruminants was very limited. Parent was the only significant residue in muscle, fat and liver (60–88% TRR for the furanone label and 85–99 % TRR for the pyridinylmethyl label). It was also the major component in milk with the pyridinyl label (89% TRR), but lactose was the major component in milk for the furanone label (67% TRR), while parent was found at 24 % of the TRR.

Significant metabolism was demonstrated only in kidney, in which parent was present at 35–50.5% TRR for the pyridinylmethyl and furanone labels respectively, while the metabolite fpd-OH (hydroxylation in the 5 position of the furanone ring) was present at 15–16% TRR. Fpd-glucuronides, from hydroxylation followed by conjugation with glucuronic acid, were observed at 13–31% of the TRR in total, with four isomers up to 9% TRR each.

Hens

Two studies on the metabolism of flupyradifurone were conducted with the test compound labelled in either the [furanone-4-^{14}C] or the [pyridinylmethyl-^{14}C]-position. In each study six laying hens were dosed orally once daily for 14 consecutive days, at 17.1 or 16.2 ppm for the furanone and pyridinylmethyl labels respectively. Animals were sacrificed approximately 6 hours after the last dose.

A total of 78 and 96% of the total administered dose was eliminated in the excreta for the furanone and pyridinylmethyl labels respectively (cumulative after 14 days). In eggs 2.4 and 0.24% of the administered dose was recovered (cumulative after 14 days). A plateau level of approximately 1.0 mg/kg equiv. was reached at 9 days after the first administration for the furanone label and 0.08 mg/kg equiv. at six days after the first administration for the pyridinylmethyl label. TRRs in tissues were 2.2 and 0.44 mg/kg equiv. in liver, 1.1 and 1.1 mg/kg equiv. in kidney, 0.18 and 0.070 mg/kg equiv. in total skeletal muscle and 0.43 and 0.021 mg/kg equiv. in total fat for the furanone and pyridinylmethyl labels respectively.

Acetonitrile and water extraction of egg, muscle, fat and liver resulted in extraction efficiencies of 65.0–96.1% (eggs), 38.6–92.6% (muscle), 79.7–98.5% (fat) and 72.1–74.6% (liver).

Parent was generally a minor component in hen matrices, with the exception of muscle for the furanone label (14% TRR) and fat (15% TRR) and eggs (20% TRR) for the pyridinylmethyl label only. Fatty acids were the major metabolic product with the furanone label for eggs, fat, and liver (52–96% TRR). With the pyridinylmethyl label, fpd-acetyl-AMCP, from cleavage of both the furanone and difluoroethyl groups, was the major residue in eggs (23% TRR), muscle (40% TRR) and fat (29% TRR), while the major residue in liver was fpd-OH-SA from hydroxylation of the furanone (23% TRR).

Summary of animal metabolism

Whilst parent was the main compound detected in excreta of rats, flupyradifurone was metabolized to eight identified metabolites and up to 19 unidentified metabolites involving hydroxylation, conjugation and cleavage reactions.

Metabolism in ruminants was very limited. Parent flupyradifurone was the major portion of the residue in fat, muscle, and liver (60–99% TRR). The parent was also the major component in milk with the pyridinylmethyl label (89% TRR). Significant metabolism was demonstrated only in the kidney.

Metabolism in poultry was far more extensive than in ruminants. Parent was generally a minor component in hen matrices, with the exception of muscle for the furanone label (14% of the TRR) and fat (15% of the TRR) and eggs (20% of the TRR) for the pyridinylmethyl label only. Fatty acids were the major metabolic product with the furanone label for eggs, fat, and liver. With the pyridinylmethyl label, fpd-acetyl-AMCP, was the major residue in eggs, muscle and fat and the major residue in liver was fpd-OH-SA (23% TRR).

Difluoroacetic acid would not have been detected in the hen and goat metabolism studies as a labelled residue due to the positions of the radiolabels.

Methods of analysis

The Meeting received information on analytical methods suitable for the determination of residues of the active substance flupyradifurone and the metabolites DFA, DFEAF and 6-CNA in plant matrices and flupyradifurone and the metabolites DFA, fpd-acetyl-AMCP and fpd-OH in animal matrices.

The methods used for data collection and proposed for enforcement for plant and animal commodities are based on LC-MS/MS for plant and animal matrices. All methods involve extraction with acetonitrile/water. LOQs were generally 0.01 mg/kg for parent, DFEAF and 6-CNA and 0.02 mg/kg for DFA in plant commodities. In animal commodities, LOQs were generally 0.01 mg/kg for parent, fpd-acetyl-AMCP and fpd-OH and 0.01 or 0.02 mg/kg for DFA. Radio-validation experiments were carried out on the analytical methods for plant and animal commodities confirming the acceptability of the methods.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the freezer storage stability of flupyradifurone in plant commodities.

Residues trial data are supported by the supplied storage studies which showed that flupyradifurone, DFEAF and DFA residues are stable for at least 18 months in high water, high acid, high oil, high protein and high starch content matrices, when stored frozen at approximately –18 °C. The storage periods in the storage stability studies cover the sample storage intervals in the residue trials.

All samples in the laying hen feeding study were analysed within thirty days of collection. Therefore, there was no necessity for freezer storage stability data. In the dairy cattle feeding study, tissues and milk samples were analysed within 25 days of collection for residues of parent, fpd-OH and fpd-AMCP and therefore freezer storage stability data was not required for these analytes. DFA residues were shown to be stable in animal commodities for up to 43 days in frozen storage.

Definition of the residue

Plants

In the metabolism studies on primary crops using [furanone-4-^{14}C]-fpd and [pyridinylmethyl-^{14}C]-fpd, flupyradifurone was consistently observed to be the major component of the radioactive residues, accounting for approximately 23–88% of the TRR in all plant parts analysed. Parent compound was the main component detected in almost all matrices of all rotations in the confined rotational crop trials accounting for 18–72% of the TRR.

The Meeting therefore considered that a residue definition of *Flupyradifurone* is appropriate for plant commodities for compliance with MRLs (enforcement).

Due to the fact that parent flupyradifurone was consistently the predominant residue in plants in the metabolism studies and was one of the two predominant components in the supervised field trials over a wide range of crops, parent should also be in the definition for risk assessment.

There are a number of possible candidates for inclusion in the risk assessment definition.

DFA was detected at significant levels in apple fruit, potato tuber, cotton seed and rice grain, irrespective of method of application in the metabolism studies. Significant levels of DFA were also detected in all plant matrices of the first and second rotation, except turnip roots, in the confined rotational study. DFA was found in most crops included in the field rotation studies and was the only significant residue. DFA is considered to be 3× more potent than flupyradifurone (Toxicology report—JMPR 2015). DFA was also included in the data generation method and should be in the definition for risk assessment.

The metabolite fpd-difluoroethyl-amino-furanone (DFEAF), was a metabolite found in Swiss chard in all rotations of the confined rotational crop study at up to 17% TRR and at much lower levels in other crops. However, it was not detected in any of the field rotation studies. It was analysed in all residue trials conducted in primary and succeeding crops. DFEAF was usually not detected or residues were generally very low (< 5%) in most crops in comparison to the total observed residues. DFEAF was observed in the rat metabolism studies and was observed to be no more toxic than parent flupyradifurone. The Meeting therefore considered that DFEAF need not be included in the risk assessment residue definition for plants.

The metabolite 6-CNA was present at > 10% TRR in a number of plant metabolism studies, (13–22% TRR in tomato fruit, potato tuber and cotton seed) but was generally present at low residue levels (approximately 0.02 mg/kg equiv.). It was included in the residue trial analyses making a significant contribution to flupyradifurone residues in a number of crops such as beans, soya beans and cotton seed. 6-CNA is found to be no more toxic than parent flupyradifurone and will be included in the flupyradifurone residue definition for dietary assessment.

Given that DFA is considered to be 3× more potent than flupyradifurone (Toxicology report—JMPR 2015), the sum of parent, 6-CNA and 3× DFA is the recommended residue definition for commodities of plant origin for risk assessment (dietary exposure assessment). Adjusting residues of DFA to parent equivalents, means there is no necessity for applying the toxicity factor of 3, as the molecular weight of parent flupyradifurone (288.68) is 3.0× the molecular weight of DFA (96.03). Residues of DFA have been expressed as parent equivalents in the submitted field trials.

The proposed residue definition for plant commodities for dietary risk assessment is the *Sum of flupyradifurone, difluoroacetic acid and 6-chloronicotinic acid, expressed as parent equivalents*.

Animals

Metabolism in ruminants was very limited. Parent flupyradifurone was the major portion of the residue in fat, muscle, and liver (60–88% TRR for the furanone label and 85–99% TRR for the pyridinylmethyl label) and was also the major component in milk for the pyridinylmethyl label (89% TRR). Parent was present in the kidney at 35–50% TRR.

Metabolism in poultry was far more extensive than in ruminants. Parent was generally a minor component in hen matrices, with the exception of muscle for the furanone label (14% TRR) and fat (15% TRR) and eggs (20% TRR) for the pyridinylmethyl label only. Fatty acids were the major metabolic product for the furanone label in eggs, fat, and liver. With the pyridinylmethyl label, fpd-acetyl-AMCP, was the major residue in eggs (23% TRR) and the major residue in liver was fpd-OH-SA (23% TRR).

In terms of the estimated livestock animal dietary burden, fpd-OH-SA is expected to be present in poultry liver at up to 0.1 mg parent equiv./kg, compared to the HR for total flupyradifurone of 0.89 mg parent equiv./kg. This contribution to the total toxicological burden is considered to be minor since it is unlikely to be of greater toxicity than the unconjugated alcohol, which is covered by the parent. Therefore, fpd-OH-SA does not have to be included in the definition for dietary intake for animal commodities.

Difluoroacetic acid was not analysed for in the hen and goat metabolism studies. Residues of parent, DFA, fpd-OH and fpd-AMCP were determined in the lactating dairy cattle and laying hen feeding studies. No quantifiable residues of fpd-OH or fpd-AMCP were observed in any milk sample. The residue in milk was primarily (60–90%) parent. No quantifiable residues of fpd-AMCP were observed in any cow tissues and residues of fpd-OH (when present in cow tissues), were always much lower than residues of parent (the dominant residue at approximately 70–90%) and DFA. In the hen feeding study residues of parent, fpd-OH and fpd-AMCP were much lower than residues of DFA (usually greater than 90%) in eggs and tissues. After feeding at 19.4 ppm for 29 days, for example, mean residues in fat, liver and muscle were 0.275, 1.02 and 0.723 mg/kg of which 0.272, 1.01 and 0.719 mg/kg were DFA.

The proposed residue definition for animal commodities for compliance with MRLs (enforcement) is *Sum of flupyradifurone and difluoroacetic acid, expressed as parent equivalents*.

A residue definition of *Sum of flupyradifurone and 3× difluoroacetic acid* is appropriate for commodities of animal origin for dietary risk assessment. As the MWt of flupyradifurone is 3.0× the MWt of DFA, it is not necessary to apply a toxicity factor for DFA, if the residues of DFA are expressed as parent equivalents.

The proposed residue definition for animal commodities for dietary risk assessment is *Sum of flupyradifurone and difluoroacetic acid, expressed as parent equivalents*.

The ratio of total residues (flupyradifurone + DFA) in muscle and fat in the livestock feeding studies support the conclusion that the total residue is not fat soluble (mean residues of fat/ muscle in the dairy cattle study at four feeding levels 0.48–0.72 and 0.37–0.52 in the laying hen study at four feeding levels). There is no evidence to suggest that there is significant potential for bioaccumulation in fat tissues.

Definition of the residue (for compliance with the MRL for plant commodities): *Flupyradifurone.*

Definition of the residue (for estimation of dietary intake for plant commodities): *Sum of flupyradifurone, difluoroacetic acid and 6-chloronicotinic acid, expressed as parent equivalents.*

Definition of the residue (for compliance with the MRL and for estimation of dietary intake for animal commodities): *Sum of flupyradifurone and difluoroacetic acid, expressed as parent equivalents.*

The Meeting agreed that the residue be designated as not fat soluble.

Results of supervised residue trials on crops

Supervised trials were available for the use of flupyradifurone on citrus fruits (grapefruit, lemons, mandarins and oranges), pome fruits (apples and pears), berries and other small fruits (blueberries, grapes and strawberries), assorted tropical and sub-tropical fruit—inedible peel (prickly pear), bulb vegetables (bulb onion and green onions), Brassica vegetables (broccoli, cabbage, and cauliflower), cucurbits (cucumber, melons and summer squash), fruiting vegetables other than cucurbits (tomatoes, chilli and sweet peppers and sweet corn), leafy vegetables (mustard greens, head and leafy lettuce and spinach), legume vegetables (common bean, snow peas, lima beans and garden peas), pulses (peas, beans and soya beans), root and tuber vegetables (carrots, radishes and potatoes), stalk and stem vegetables (celery), cereals (barley, wheat, sorghum and maize), tree nuts (almonds and pecans), oilseeds (cotton and peanuts), coffee and hops.

For maximum residue level estimation (compliance) residues of flupyradifurone parent have been considered. For dietary intake assessment (risk assessment), residues of flupyradifurone, DFA and 6-chloronicotinic acid expressed as parent equivalents (referred to as total residues of flupyradifurone), have been considered.

Product labels were available from the United States of America and Central America.

The Central American GAPs were the critical GAPs for citrus fruit, melon, cucumber and watermelon, tomatoes and chilli peppers and potatoes. None of the submitted trial data matched the Central American GAPs for any of these crops, so these will not be referred to further. The USA label has foliar and soil application GAPs for some crop groups. Only one of these methods of application is allowed per crop.

The following crops are listed on the USA label under rotational crops for immediate plant-back: Cereal grains (except rice), cotton, non-grass animal feeds (alfalfa and clover only), peanut, root vegetables (except sugar beet), tuberous and corm vegetables, leafy vegetables, Brassica (cole) leafy vegetables, legume vegetables (succulent or dried), fruiting vegetables, cucurbit vegetables, hops, citrus fruit, pome fruit, bush berry (except cranberry), low growing berry (except cranberry), small fruit vine climbing (except fuzzy kiwifruit), tree nut (except almond), prickly pear/ cactus pear.

Bulb vegetables (Group 3-07, *Allium* Spp.) are on the current USA Sivanto 200SL label as a rotational crop only, with immediate plant-back interval.

The label states that for crops not listed in the immediate plant-back section of this label, or for crops for which no tolerances for the active ingredient have been established, a 12-month plant-back interval must be observed.

Residue trial data were submitted for bulb onions, green onions, almonds, almond hulls and coffee. As there is no submitted GAP for these crops, the trial data for these crops will not be discussed further.

Where mean values have been considered, if an individual observation is higher than the highest mean, this is also listed (as the HR). Where parent or DFA residues were not detected or were less than the LOQ (*i.e.* < 0.01 mg/kg for parent or 0.05 mg/kg for DFA) the LOQ value was utilized for maximum residue estimation and dietary intake assessment. For 6-CNA, values less than the LOQ were not added for calculation of total residues of flupyradifurone.

The following table shows how residues in the trials were added to give total residues of flupyradifurone.

Parent	DFA	6-CNA	Total
< 0.01	0.05	0.01	0.07
0.01	< 0.05	0.01	0.07
< 0.01	< 0.05	< 0.01	< 0.06
0.01	0.05	< 0.01	0.06
0.01	0.05	0.01	0.07

Citrus fruits, pome fruits, bush berries, grapes and tree nuts are normally cultivated as permanent crops and are not expected to be subject to a potential uptake of flupyradifurone residues from the soil.

The Meeting noted that the submitted plant metabolism studies show that parent flupyradifurone metabolizes to a number of other compounds which, like parent, would be converted to 6-CNA by a common moiety imidacloprid analytical method. It is noted that the submitted residues trials indicate that the use of flupyradifurone results in residues of 6-CNA in a number of crops which would require changes to established imidacloprid MRLs or the establishment of new imidacloprid MRLs. As the submitted trials did not quantify total residues containing the 6-chloropyridyl moiety, the Meeting agreed that any attempt to estimate maximum residue levels for imidacloprid, resulting from the use of flupyradifurone, would not be robust. The Meeting recommends that the residue definition for imidacloprid be changed from that requiring a common moiety analytical method.

Alternatively, some future trials of flupyradifurone could measure total residues containing 6-chloropyridyl, using a common moiety method.

Citrus Fruit—Grapefruit, Lemons, Mandarins and Oranges

The USA foliar GAP for citrus fruit is two applications at 205 g ai/ha, 10 day RTI, 1-day PHI). The USA soil application GAP for citrus fruit is one application at 409 g ai/ha, 30-day PHI).

Residue data for mandarins, grapefruit, lemons and oranges have been submitted according to the USA GAPs for foliar and soil application. Foliar applications were made using either concentrated or dilute sprays. The highest residue observations after foliar application have been selected from each trial for estimation of maximum residue levels and for dietary intake purposes, as these were always greater than the soil application residue observations.

The Meeting noted that the use in the USA is for the citrus fruit group. Although the median residues for each fruit differed by a factor of less than five, the Meeting decided to recommend maximum residue levels for the individual sub-groups of citrus fruit, as there are sufficient trials for each sub-group.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in mandarins from supervised trials (foliar use pattern) according to the GAP in the USA was 0.12, 0.16, 0.35, 0.39, 0.51, 0.61 and 0.90 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in mandarins from supervised trials (foliar use pattern) according to the GAP in the USA was 0.14, 0.21, 0.40, 0.44, 0.56, 0.66 and 0.99 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for the subgroup mandarins of 1.5, 0.44 and 0.99 mg/kg respectively.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in grapefruit from supervised trials (foliar use pattern) according to the GAP in the USA was 0.16, 0.19, 0.19, 0.19, 0.27 and 0.29 mg/kg.

The ranked order of total residues of flupyradifurone in grapefruit from supervised trials (foliar use pattern) according to the GAP in the USA was 0.18, 0.21, 0.21, 0.21, 0.31 and 0.32 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for the subgroup pomelo and grapefruit of 0.7, 0.21 and 0.32 mg/kg respectively.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in lemons from supervised trials (foliar use pattern) according to the GAP in the USA was 0.12, 0.18, 0.23, 0.30, 0.35, 0.44 and 0.71 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in lemons from supervised trials (foliar use pattern) according to the GAP in the USA was 0.14, 0.20, 0.25, 0.32, 0.37, 0.55 and 0.73 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for the subgroup lemons and limes of 1.5, 0.32 and 0.73 mg/kg respectively.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in oranges from supervised trials (foliar use pattern) according to the GAP in the USA was 0.067, 0.19, 0.25, 0.29, 0.34, 0.63, 0.70, 0.88, 1.2 and 2.1 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in oranges from supervised trials (foliar use pattern) according to the GAP in the USA was 0.087, 0.21, 0.27, 0.31, 0.36, 0.65, 0.72, 0.91, 1.2 and 2.2 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for the subgroup oranges, sweet, sour of 4, 0.505 and 2.2 mg/kg respectively.

Pome Fruit—Apples and Pears

Residue trials were conducted in apples in the USA according to the critical GAP in the USA for pome fruit (two foliar applications at 205 g ai/ha, 10-day RTI, 14-day PHI). Applications were made using either concentrated or dilute sprays. The highest observations for estimation of maximum residue levels and for dietary intake purposes have been selected from each trial.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in apples from supervised trials according to the GAP in the USA was 0.060, 0.084, 0.097, 0.12, 0.13, 0.14, 0.15, 0.18, 0.21, 0.22, 0.22, 0.25 and 0.30 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in apples from supervised trials according to the GAP in the USA was 0.11, 0.13, 0.15, 0.17, 0.18, 0.19, 0.23, 0.25, 0.27, 0.28, 0.28, 0.30 and 0.62 mg/kg.

Residue trials were conducted in pears in the USA and Canada according to the critical GAP in the USA for pome fruit (two foliar applications at 205 g ai/ha, 10-day RTI, 14-day PHI).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in pears from supervised trials according to the GAP in the USA was 0.18, 0.19, 0.20, 0.21, 0.22, 0.26, 0.32, 0.39 and 0.47 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in pears from supervised trials according to the GAP in the USA was 0.23, 0.29, 0.32, 0.44, 0.45, 0.49, 0.59, 0.63 and 0.69 mg/kg.

The use pattern in the USA is for Crop Group 11-10 (Pome Fruit). To consider a maximum residue level for a group, residues in individual crops should be similar (e.g. medians should not differ by more than 5×). In deciding whether to combine the datasets for apples and pears or to only utilize the data from the commodity with the highest residues, the Meeting noted that the populations of residues in apples and pears are sufficiently different (Mann-Whitney U-Test) and decided to use the data from pears to estimate a maximum residue level for the group Pome fruit.

Based on pear data only, the Meeting estimated a maximum residue level, an STMR and an HR of 0.9, 0.45 and 0.69 mg/kg for pome fruit.

Berries and other Small Fruit—Blueberries, Grapes and Strawberries

Residue trials were conducted in blueberries (four low bush, 21 highbush and one rabbit eye) in the USA (nine trials), Canada (four), Australia (three), Chile (three), New Zealand (two), United Kingdom (two) and one each in Italy, Spain and Denmark, most according to the GAP in the USA for Crop Subgroup 13-07B (except cranberry) (two foliar applications at 205 g ai/ha, 7-day RTI, 3-day PHI).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in blueberries from supervised trials carried out in the USA and Canada according to the GAP in the USA was 0.35, 0.42, 0.45, 0.57, 0.78, 1.2, 1.6 and 2.5 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in blueberries from supervised trials carried out in the USA and Canada according to the GAP in the USA was 0.40, 0.47, 0.50, 0.62, 0.83, 1.2, 1.6 and 2.6 mg/kg.

The Meeting noted that blueberries can be used as a representative crop for bush berries and estimated an STMR of 0.725 mg/kg, an HR of 2.6 mg/kg and a sub-group maximum residue level of 4 mg/kg for flupyradifurone on bush berries.

Residue trials were conducted in grapes in the USA and Canada according to the critical GAP in the USA for foliar application for subgroup 13-07F (two applications at 205 g ai/ha, 10-day RTI, 0-day PHI). In addition, soil drench trials were carried out according the USA GAP for soil application for subgroup 13-07F (one application at 409 g ai/ha, 30-day PHI). Residue observations in grapes arising from the foliar use pattern were higher than those arising from the soil use pattern in every trial, so these have been considered for estimation of the maximum residue level.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in grapes from supervised trials (foliar use pattern) according to the GAP in the USA was 0.31, 0.39, 0.46, 0.52, 0.57, 0.58, 0.69, 0.80, 1.0, 1.1 and 1.9 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in grapes from supervised trials (foliar use pattern) according to the GAP in the USA was 0.36, 0.44, 0.51, 0.57, 0.63, 0.63, 0.74, 0.85, 1.2, 1.2 and 2.0 mg/kg (HR 2.3 mg/kg).

Based on the dataset, the Meeting estimated a maximum residue level, an STMR and an HR for grapes of 3, 0.63 and 2.3 mg/kg respectively for grapes.

Residue trials were conducted in strawberries in the USA and Canada according to the critical GAP in the USA for application for sub-group 13-07G (two foliar applications at 205 g ai/ha, 10-day RTI, 0-day PHI).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in strawberries from supervised trials according to the GAP in the USA was 0.23, 0.33, 0.38, 0.38, 0.43, 0.51, 0.54, 0.54, 0.58 and 0.62 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in strawberries from supervised trials according to the GAP in the USA was 0.33, 0.38, 0.43, 0.43, 0.48, 0.57, 0.59, 0.59, 0.63 and 0.90 mg/kg (HR 0.94 mg/kg).

Based on the dataset, the Meeting estimated a maximum residue level, a median and a highest residue for strawberries of 1.5, 0.525 and 0.94 mg/kg respectively.

For strawberries no data from studies on relevant follow crops are available.

In field studies on succeeding crops, the mean, median and highest total residues in French beans were 0.98, 0.80 and 1.80 mg/kg respectively. In the absence of data for relevant follow crops the Meeting decided to add the mean residue found in French beans in field studies on succeeding crops of 0.98 mg/kg to the median residue obtained from strawberry residue trials of 0.525 mg/kg for an overall STMR for flupyradifurone in strawberries of 1.505 mg/kg. The Meeting also decided to add the highest residue of 1.80 mg/kg for French beans in the succeeding crop trials to 0.94 mg/kg (the highest residue found in strawberry field trials) for an overall HR of 2.74 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for strawberries of 1.5, 1.505 and 2.74 mg/kg respectively.

Assorted tropical and sub-tropical fruit – inedible peel —Prickly Pear

Residue trials were conducted in prickly pear cactus in the USA according to the critical GAP in the USA (two applications at 205 g ai/ha, 7-day RTI, 21-day PHI). Residues were determined in fruit and pads. Four trials were carried out for each but only two trials for fruit (observed parent residues 0.10 and 0.12 mg/kg and total residues 0.15 and 0.17 mg/kg) and two for pads (observed parent residues 0.20 and 0.25 mg/kg and total residues 0.25 and 0.30 mg/kg) can be considered independent.

No maximum residue level was recommended due to the limited data (n = 2 independent trials).

Bulb vegetables—Bulb onion and Green Onions

There is no registered use for bulb onions or green onions as a primary crop. Bulb vegetables (Group 3-07, *Allium* Spp.) are on the USA Sivanto 200SL label as a rotational crop only, with immediate plant-back interval.

Data from studies on onions and leeks as follow crops are available. In field studies on succeeding crops, mean, median and highest total residues in onions were 0.14, 0.12 and 0.28 mg/kg respectively. Mean, median and highest residues in leeks were 0.18, 0.13 and 0.39 mg/kg respectively. No residues of parent flupyradifurone were observed.

Based on the leek data (mean and highest residues), the Meeting estimated a maximum residue level, an STMR and an HR for bulb vegetables except fennel bulb of *0.01, 0.18 and 0.39 mg/kg respectively.

Brassica vegetables—Broccoli, Cabbage and Cauliflower

Residue trials were conducted in broccoli (four trials), cabbage (10 trials) and cauliflower (six trials) in the USA and Canada according to the GAP in the USA for Crop Group 5 (two foliar applications at 205 g ai/ha, 7-day RTI, 1-day PHI).

The Meeting noted that the GAP was for the Brassica vegetables group, and considered a group maximum residue level, however the medians for the broccoli and cauliflower data sets differed by greater than 5×.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in broccoli heads and stalks from supervised trials according to the GAP in the USA was 0.37, 0.40, 0.95 and 1.9 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in broccoli from supervised trials according to the GAP in the USA was 0.46, 1.2, 2.5 and 3.3 mg/kg (HR 3.5 mg/kg).

The Meeting considered the data insufficient for estimating a maximum residue level for broccoli.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in cauliflower heads and stalks from supervised trials according to the GAP in the USA was 0.022, 0.032, 0.087, 0.11, 2.1 and 2.4 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in cauliflower heads and stalks from supervised trials according to the GAP in the USA was 0.11, 0.20, 0.26, 0.46, 2.2 and 2.5 mg/kg (HR 2.6 mg/kg).

The Meeting estimated a maximum residue level, a median and a highest residue for cauliflower of 6, 0.36 and 2.6 mg/kg respectively.

For Brassica vegetables, no data from studies on follow crops are available. In field studies on succeeding crops, the overall highest mean, median and highest total residues in lettuce were 0.12, < 0.06 and 0.41 mg/kg respectively.

The Meeting decided to add the mean residue for lettuce in field studies on succeeding crops of 0.12 mg/kg to the median residue obtained from supervised cauliflower residue trials of 0.36 mg/kg for an overall STMR for flupyradifurone in cauliflower of 0.48 mg/kg.

It was also decided to add 2.6 mg/kg (the highest residue found in supervised field trials for cauliflower) and the highest residue of 0.41 mg/kg for lettuce in the succeeding crop trials.

The Meeting estimated a maximum residue level, an STMR and an HR for cauliflower of 6, 0.48 and 3.01 mg/kg respectively for cauliflower.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in cabbage head from supervised trials (foliar use pattern) according to the GAP in the USA was 0.11, 0.12, 0.32, 0.33, 0.38, 0.45, 0.69, 0.82 and 0.83 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in cabbage head from supervised trials according to the GAP in the USA was 0.20, 0.24, 0.44, 0.60, 0.67, 0.79, 0.93, 1.0 and 1.1 mg/kg (HR 1.3 mg/kg).

The Meeting estimated a maximum residue level, a median and a highest residue for cabbage, head of 1.5 mg/kg, 0.67 and 1.3 mg/kg respectively.

It was decided to add the mean residue for lettuce in field studies on succeeding crops of 0.12 mg/kg to the median residue obtained from supervised cabbage residue trials of 0.67 mg/kg for an overall STMR for flupyradifurone in cabbage of 0.79 mg/kg.

It was also decided to add 1.3 mg/kg (the highest residue found in supervised field trials for cabbage) and the highest residue of 0.41 mg/kg for lettuce in the succeeding crop trials.

The Meeting estimated a maximum residue level, an STMR and an HR for cabbage of 1.5, 0.79 and 1.71 mg/kg respectively for cabbage.

Cucurbits—Cucumber, Melons and Summer Squash

Residue trials were conducted in cucumbers (nine trials), summer squash (eight trials) and muskmelons (five trials) in the USA and Canada according to the foliar GAP in the USA for Crop Group 9 (two foliar applications at 205 g ai/ha, 7-day RTI, 1-day PHI) and also according to the soil application GAP in the USA (one application at 409 g ai/ha, 21-day PHI). The highest observations for estimation of maximum residue levels and for dietary intake purposes have been selected from each trial for both the foliar application and the soil application.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in cucumber from supervised trials (foliar use pattern) according to the GAP in the USA was 0.039, 0.081, 0.083, 0.092, 0.10, 0.11, 0.13, 0.19 and 0.23 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in cucumber from supervised trials (foliar use pattern) according to the GAP in the USA was 0.16, 0.18, 0.28, 0.28, 0.34, 0.45, 0.62, 0.85 and 1.0 mg/kg (HR 1.1 mg/kg).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in cucumber from supervised trials (soil use pattern) according to the GAP in the USA was < 0.010, < 0.010, < 0.010, < 0.010, 0.011, 0.012, 0.015, 0.022 and 0.027 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in cucumber from supervised trials (soil use pattern) according to the GAP in the USA was < 0.060, 0.061, 0.066, 0.13, 0.18, 0.24, 0.42, 0.47 and 1.0 mg/kg (HR 1.5 mg/kg).

The Meeting estimated a maximum residue level for cucumbers of 0.4 mg/kg (based on the foliar use pattern).

The Meeting noted that in some decline trials conducted with flupyradifurone on cucumbers, there was no indication that the total residues (flupyradifurone + DFA + 6-CNA) had reached a maximum. Therefore, it was not possible to estimate an STMR and HR for cucumbers.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in summer squash from supervised trials (foliar use pattern) according to the GAP in the USA was 0.032, 0.033, 0.048, 0.054, 0.055, 0.075, 0.081 and 0.10 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in summer squash from supervised trials (foliar use pattern) according to the GAP in the USA was 0.10, 0.14, 0.16, 0.21, 0.22, 0.61, 0.95 and 1.1 mg/kg (HR 1.4 mg/kg).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in summer squash from supervised trials (soil use pattern) according to the GAP in the USA was < 0.010, < 0.010, < 0.010, < 0.010, 0.020, 0.024, 0.031 and 0.057 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in summer squash from supervised trials (soil use pattern) according to the GAP in the USA was < 0.060 (4), 0.16, 1.1, 1.1 and 1.4 mg/kg (HR 1.5 mg/kg).

The Meeting estimated a maximum residue level, a median and a highest residue for summer squash of 0.2 mg/kg, 0.215 and 1.5 mg/kg respectively.

In field studies on succeeding crops, mean, median and highest total residues in cucumbers were 0.44, 0.51 and 0.69 mg/kg respectively. The Meeting decided to add the mean residue found in field studies on succeeding crops of 0.44 mg/kg to the median residue obtained from supervised cucurbit fruiting vegetables residue trials of 0.215 mg/kg for an overall STMR for flupyradifurone in summer squash of 0.655 mg/kg.

It was also decided to add 1.5 mg/kg (the highest residue found in supervised field trials) and the highest residue of 0.69 mg/kg for cucumbers in the succeeding crop trials.

The Meeting estimated a maximum residue level, an STMR and an HR of 0.2, 0.655 and 2.19 mg/kg respectively for summer squash.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in melon fruit from supervised trials (foliar use pattern) according to the GAP in the USA was 0.061, 0.088, 0.11, 0.15 and 0.19 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in melon fruit from supervised trials (foliar use pattern) according to the GAP in the USA was 0.20, 0.22, 0.38, 0.52 and 0.68 mg/kg (HR 0.71 mg/kg).

For dietary intake purposes the ranked order of total residues of flupyradifurone in melon pulp from supervised trials (foliar use pattern) according to the GAP in the USA was < 0.060 (2), 0.062, 0.075 and 0.095 mg/kg.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in melon fruit from supervised trials (soil use pattern) according to the GAP in the USA was < 0.010, 0.012, 0.012, 0.017 and 0.028 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in melons from supervised trials (soil use pattern) according to the GAP in the USA was 0.082, 0.088, 0.23, 0.43 and 0.87 mg/kg (HR 1.2 mg/kg).

For dietary intake purposes the ranked order of total residues of flupyradifurone in melon pulp from supervised trials (soil use pattern) according to the GAP in the USA was < 0.060 (2), 0.13, 0.22 and 0.38 mg/kg (HR 0.38 mg/kg).

The Meeting estimated a maximum residue level for melons of 0.4 mg/kg (based on the foliar use pattern). The Meeting estimated a median and highest residue for melon of 0.13 and 0.38 mg/kg respectively (based on the soil use pattern melon pulp data).

In field studies on succeeding crops, mean, median and highest total residues in cucumbers were 0.44, 0.51 and 0.69 mg/kg respectively.

The Meeting decided to add the mean residue found in field studies on succeeding crops of 0.44 mg/kg to the median residue obtained from supervised melon residue trials of 0.13 mg/kg for an overall STMR for flupyradifurone in melons of 0.57 mg/kg.

It was also decided to add 0.38 mg/kg (the highest residue found in supervised field trials) and the highest residue of 0.69 mg/kg for cucumbers in the succeeding crop trials.

The Meeting estimated a maximum residue level, an STMR and an HR of 0.4, 0.57 and 1.07 mg/kg respectively for melon, except watermelons.

Fruiting vegetables other than Cucurbits—Tomatoes, Sweet and Chili peppers

Residue trials were conducted in tomatoes (19 trials), sweet peppers (10 trials) and chilli peppers (four trials) in the USA and Canada, according to the GAP in the USA for Crop Group 8-10 (two foliar applications at 205 g ai/ha, 7-day RTI, 1-day PHI) and also according to the soil application GAP for Crop Group 8-10 (1 application at 409 g ai/ha, 45-day PHI). The highest observations for estimation of maximum residue levels and for dietary intake purposes have been selected from each trial for both the foliar application and the soil application.

The Meeting noted that in some decline trials conducted with flupyradifurone on fruiting vegetables other than cucurbits, there was no indication that the total residues (flupyradifurone + DFA

+ 6-CNA) had reached a maximum. The Meeting therefore decided that although the GAP was for the fruiting vegetables other than cucurbits group, no Crop Group MRL will be considered.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in tomatoes from supervised trials (foliar use pattern) according to the GAP in the USA was 0.055, 0.057, 0.059, 0.068, 0.086, 0.088, 0.11, 0.13, 0.14, 0.14, 0.14, 0.15, 0.23, 0.27, 0.28, 0.31, 0.45, 0.57 and 0.73 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in tomatoes from supervised trials (foliar use pattern) according to the GAP in the USA was 0.11, 0.11, 0.11, 0.15, 0.15, 0.18, 0.19, 0.19, 0.22, 0.27, 0.29, 0.30, 0.32, 0.33, 0.40, 0.46, 0.53, 0.62 and 0.91 mg/kg (HR 1.1 mg/kg).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in tomatoes from supervised trials (soil use pattern) according to the GAP in the USA was < 0.010 (7), 0.010, 0.011, 0.012, 0.013, 0.014, 0.015, 0.015, 0.029, 0.031, 0.034, 0.069 and 0.24 mg/kg

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in tomatoes from supervised trials (soil use pattern) according to the GAP in the USA was < 0.060, < 0.060, 0.064, 0.070, 0.079, 0.10, 0.13, 0.15, 0.15, 0.19, 0.20, 0.24, 0.34, 0.68, 0.81, 0.81, 0.90, 1.1 and 1.9 mg/kg (HR 2.1 mg/kg).

The Meeting estimated a maximum residue level of 1 mg/kg based on the foliar use pattern. The Meeting estimated a median and highest residue for tomato of 0.27 and 2.1 mg/kg.

For tomatoes, no data from studies on follow crops are available. In field studies on succeeding crops, mean, median and highest total residues in cucumbers (fruiting vegetables, cucurbits) were 0.44, 0.51 and 0.69 mg/kg respectively. The Meeting decided to add the mean residue found in cucumber field studies on succeeding crops of 0.44 mg/kg to the median residue obtained from supervised tomato residue trials of 0.27 mg/kg for an overall STMR for flupyradifurone in tomatoes of 0.71 mg/kg. It was also decided to add 2.1 mg/kg (the highest residue found in supervised tomato field trials) to the highest residue of 0.69 mg/kg for cucumbers in the succeeding crop trials.

The Meeting estimated a maximum residue level, an STMR and an HR for tomatoes of 1, 0.71 and 2.79 mg/kg respectively.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in peppers from supervised trials (foliar use pattern) according to the GAP in the USA was 0.030, 0.051, 0.070, 0.073, 0.083, 0.087, 0.12, 0.12, 0.12, 0.29, 0.30, 0.37, 0.47 and 0.53 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in peppers from supervised trials (foliar use pattern) according to the GAP in the USA was 0.11, 0.12, 0.14, 0.17, 0.17, 0.20, 0.22, 0.26, 0.35, 0.39, 0.42, 0.44, 0.52 and 0.68 mg/kg (HR 0.81 mg/kg).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in peppers from supervised trials (soil use pattern) according to the GAP in the USA was < 0.010, < 0.010, < 0.010, < 0.010, < 0.010, 0.011, 0.011, 0.011, 0.013, 0.024, 0.027, 0.035, 0.047 and 0.18 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in peppers from supervised trials (soil use pattern) according to the GAP in the USA was 0.071, 0.098, 0.10, 0.13, 0.13, 0.13, 0.14, 0.16, 0.17, 0.36, 0.52, 0.72, 0.92 and 1.6 mg/kg (HR 1.7 mg/kg).

The Meeting estimated a maximum residue level of 0.9 mg/kg for peppers based on the foliar use pattern. The Meeting estimated a median and highest residue of 0.24 and 1.7 mg/kg.

For peppers, no data from studies on follow crops are available. In field studies on succeeding crops, mean, median and highest total residues in cucumbers (fruiting vegetables, and cucurbits) were 0.44, 0.51 and 0.69 mg/kg respectively. The Meeting decided to add the mean residue found in cucumber field studies on succeeding crops of 0.44 mg/kg to the median residue obtained from supervised pepper residue trials of 0.24 mg/kg for an overall STMR for flupyradifurone in peppers of 0.68 mg/kg. It was also decided to add 1.7 mg/kg (the highest residue found in supervised field trials) to the highest residue of 0.69 mg/kg for cucumbers in the succeeding crop trials.

The Meeting estimated a maximum residue level, an STMR and an HR for peppers of 0.9, 0.68 and 2.39 mg/kg respectively.

The Meeting used the pepper data and a default processing factor of 10 to estimate a maximum residue level, STMR and HR for flupyradifurone in chilli pepper (dried) of 9, 6.8 and 23.9 mg/kg.

Fruiting vegetables other than Cucurbits—Sweet corn

Residue trials were conducted in sweet corn (13 trials) in the USA and Canada, according to the GAP in the USA for Crop Group 15 (two foliar applications at 205 g ai/ha, 7-day RTI, 7-day PHI). Three trials also included plots to measure residues following the planting of seed treated with flupyradifurone.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in sweet corn kernels and cob husked from supervised trials (foliar use pattern) according to the GAP in the USA was < 0.01 (9), 0.016, 0.018, 0.026 and 0.038 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in sweet corn kernels and cob husked from supervised trials (foliar use pattern) according to the GAP in the USA was < 0.060 (3), 0.068, 0.099, 0.12, 0.13, 0.13, 0.15, 0.19, 0.21, 0.25 and 0.28 mg/kg (HR 0.29 mg/kg).

The Meeting estimated a maximum residue level, a median and a highest residue for sweet corn (corn-on-the-cob) of 0.05, 0.13 and 0.29 mg/kg respectively.

For sweet corn, no data on follow crops is available. In field studies on succeeding crops, the overall mean, median and highest total residues in barley grain were 0.43, 0.22 and 1.30 mg/kg respectively. In the absence of follow crop data for sweet corn the Meeting decided to add the mean residue found in barley grain in field studies on succeeding crops of 0.43 mg/kg to the median residue obtained from the sweet corn residue trials of 0.13 mg/kg for an overall STMR for flupyradifurone in sweet corn (corn-on-the-cob) of 0.56 mg/kg.

It was also decided to add 0.29 mg/kg (the highest residue found in supervised field trials) and the highest residue of 1.30 mg/kg for barley grain in the succeeding crop trials.

The Meeting estimated a maximum residue level, an STMR and an HR for sweet corn (corn-on-the-cob) of 0.05, 0.56 and 1.59 mg/kg respectively.

Brassica leafy vegetables

Residue trials were conducted in mustard greens (eight trials) in the USA according to the GAP in the USA for Crop Group 5 (two foliar applications at 205 g ai/ha, 7-day RTI, 1-day PHI).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in mustard greens from supervised trials according to the GAP in the USA was 6.1, 7.3, 10, 11, 12, 15, 18 and 24 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in mustard greens from supervised trials according to the GAP in the USA was 6.2, 7.7, 11, 12, 12, 15, 18 and 25 mg/kg (HR 25 mg/kg).

The Meeting estimated a maximum residue level of 40 mg/kg for flupyradifurone in mustard greens, together with an STMR and an HR of 12 and 25 mg/kg respectively.

Short term intake assessment showed that residues in mustard greens exceed the acute reference dose (ARfD) of 0.2 mg/kg bw, at 250% of the ARfD for the general population and 610% for children. No alternative GAP for mustard greens was available.

Leafy vegetables—Head and Leafy lettuce and Spinach

Residue trials were conducted in spinach (nine trials), leaf lettuce (nine trials) and head lettuce (eight trials) in the USA and Canada, according to the foliar GAP in the USA for Crop Group 4 (two foliar applications at 205 g ai/ha, 7-day RTI, 1-day PHI).

In field studies on succeeding crops, the highest mean, median and highest total residues in lettuce were 0.12, < 0.06 and 0.41 mg/kg respectively. The Meeting concluded that residues in leafy vegetables due to an additional uptake via the roots are insignificant in comparison to residue levels following direct treatment.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in spinach from supervised trials according to the GAP in the USA was 2.0, 3.8, 6.4, 6.7, 7.9, 8.8, 9.8, 17 and 17 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in spinach from supervised trials according to the GAP in the USA was 2.1, 3.8, 6.5, 7.0, 8.5, 8.9, 9.9, 17 and 18 mg/kg (HR 19 mg/kg).

The Meeting estimated a maximum residue level of 30 mg/kg for flupyradifurone in spinach, together with an STMR and an HR of 8.5 and 19 mg/kg respectively.

Short term intake assessment showed that residues in spinach exceed the acute reference dose (ARfD) of 0.2 mg/kg bw, at 130% of the ARfD for the general population and 420% for children. No alternative GAP for spinach was available.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in leaf lettuce from supervised trials according to the GAP in the USA was 1.1, 1.8, 2.1, 2.3, 2.7, 3.8, 6.3 and 7.3 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in leaf lettuce from supervised trials according to the GAP in the USA was 1.2, 2.1, 2.2, 2.4, 2.8, 3.9, 6.5 and 7.5 mg/kg (HR 8.0 mg/kg).

The Meeting estimated a maximum residue level of 15 mg/kg for flupyradifurone in lettuce, leaf, together with an STMR and an HR of 2.6 and 8.0 mg/kg respectively.

Short term intake assessment showed that residues in leaf lettuce exceed the acute reference dose (ARfD) of 0.2 mg/kg bw, at 250% for children. No alternative GAP for leaf lettuce was available.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in head lettuce from supervised trials according to the GAP in the USA was 0.31, 0.69, 0.76, 1.2, 1.3, 1.6, 2.0 and 2.3 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in head lettuce from supervised trials according to the GAP in the USA was 0.38, 0.77, 0.83, 1.2, 1.4, 1.7, 2.1 and 2.4 mg/kg (HR 2.4 mg/kg).

The Meeting estimated a maximum residue level of 4 mg/kg for flupyradifurone in lettuce, head, together with an STMR and an HR of 1.3 and 2.4 mg/kg respectively.

Legume vegetables without pods

Residue trials were conducted in peas (six trials) and lima beans (nine trials) in the USA and Canada (beans only), according to the foliar GAP in the USA for Crop Group 6 (two foliar applications at 205 g ai/ha, 10-day RTI, 7-day PHI).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in peas from supervised trials according to the GAP in the USA was 0.12, 0.25, 0.51, 0.62, 0.77 and 1.5 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in peas from supervised trials according to the GAP in the USA was 0.86, 1.0, 1.7, 1.9, 1.9 and 3.9 mg/kg (HR 3.9 mg/kg).

The Meeting estimated a maximum residue level of 3 mg/kg for flupyradifurone in peas, shelled (succulent seeds), together with a median and a highest residue of 1.8 and 3.9 mg/kg respectively.

In field studies on succeeding crops, the mean, median and highest total residues in French beans were 0.98, 0.80 and 1.80 mg/kg respectively. The Meeting decided to add the mean residue found in field studies on succeeding crops of 0.98 mg/kg to the median residue obtained from garden peas residue trials of 1.8 mg/kg for an overall STMR for flupyradifurone in peas, shelled (succulent seeds) of 2.78 mg/kg.

The Meeting decided to add the highest residue of 1.80 mg/kg for French beans in the succeeding crop trials to 3.9 mg/kg (the highest residue found in supervised field trials) for an overall HR of 5.8 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for peas, shelled (succulent seeds) of 3, 2.78 and 5.7 mg/kg respectively.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in lima beans from supervised trials according to the GAP in the USA was < 0.010, < 0.010, 0.011, 0.012, 0.025, 0.027, 0.062, 0.10 and 0.11 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in lima beans from supervised trials according to the GAP in the USA was < 0.070, 0.10, 0.13, 0.14, 0.19, 0.20, 0.41, 0.56 and 0.95 mg/kg (HR 0.97 mg/kg).

The Meeting estimated a maximum residue level of 0.2 mg/kg for flupyradifurone in beans, shelled (succulent = immature seeds), together with a median and highest residue of 0.19 and 0.97 mg/kg respectively.

In field studies on succeeding crops, the mean, median and highest total residues in French beans were 0.98, 0.80 and 1.80 mg/kg respectively. It was decided to add the mean residue found in field studies on succeeding crops of 0.98 mg/kg to the median residue obtained from supervised lima beans residue trials of 0.19 mg/kg for an overall STMR for flupyradifurone in beans, shelled (succulent = immature seeds) of 1.17 mg/kg.

The Meeting decided to add the highest residue of 1.80 mg/kg for French beans in the succeeding crop trials to 0.97 mg/kg (the highest residue found in supervised field trials) for an overall HR of 2.77 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for beans, shelled (succulent = immature seeds) of 0.2, 1.17 and 2.77 mg/kg respectively.

Legume vegetables with pods

Residue trials were conducted in common beans (eight trials), and snow peas (six trials) in the USA and Canada according to the foliar GAP in the USA for Crop Group 6 (two foliar applications at 205 g ai/ha, 10-day RTI, 7-day PHI).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in common beans from supervised trials according to the GAP in the USA was 0.012, 0.063, 0.13, 0.16, 0.18, 0.21, 0.24 and 0.81 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in common beans from supervised trials according to the GAP in the USA was 1.3, 1.5, 1.5, 1.6, 1.7, 1.7, 2.4 and 3.0 mg/kg (HR 3.3 mg/kg).

The Meeting estimated a maximum residue level of 1.5 mg/kg for flupyradifurone in beans, except broad bean and soya bean (green pods and immature seeds) together with a median and highest residue of 1.65 and 3.3 mg/kg respectively.

In field studies on succeeding crops, the mean, median and highest total residues in French beans were 0.98, 0.80 and 1.80 mg/kg respectively. It was decided to add the mean residue found in field studies on succeeding crops of 0.98 mg/kg to the median residue obtained from supervised snow peas residue trials of 1.65 mg/kg for an overall STMR for flupyradifurone in peas (pods and succulent = immature seeds) of 2.63 mg/kg.

The Meeting decided to add the highest residue of 1.80 mg/kg for French beans in the succeeding crop trials to 3.3 mg/kg (the highest residue found in supervised field trials) for an overall HR of 5.1 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for beans, except broad bean and soya bean (green pods and immature seeds) of 1.5, 2.63 and 5.1 mg/kg respectively.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in snow peas from supervised trials according to the GAP in the USA was 0.57, 0.58, 0.95, 0.98, 1.2, and 1.2 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in snow peas from supervised trials according to the GAP in the USA was 1.5, 1.6, 1.6, 1.8, 2.2 and 3.3 mg/kg (HR 3.7 mg/kg).

The Meeting estimated a maximum residue level of 3 mg/kg for flupyradifurone in peas (pods and succulent = immature seeds), together with a median and highest residue of 1.7 and 3.7 mg/kg respectively.

In field studies on succeeding crops, the mean, median and highest total residues in French beans were 0.98, 0.80 and 1.80 mg/kg respectively. It was decided to add the mean residue found in field studies on succeeding crops of 0.98 mg/kg to the median residue obtained from supervised snow peas residue trials of 1.7 mg/kg for an overall STMR for flupyradifurone in peas (pods and succulent = immature seeds) of 2.68 mg/kg.

The Meeting decided to add the highest residue of 1.80 mg/kg for French beans in the succeeding crop trials to 3.7 mg/kg (the highest residue found in supervised field trials) for an overall HR of 5.5 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for peas (pods and succulent = immature seeds) of 3, 2.68 and 5.5 mg/kg respectively.

Pulses—Peas and Beans

Residue trials were conducted in beans (10 trials, one in which only forage was collected), and peas (10 trials) in the USA and Canada, according to the foliar GAP in the USA for Crop Group 6 (two foliar applications at 205 g ai/ha, 10-day RTI, 7-day PHI).

The Meeting noted that the GAP was for USA Crop Group 6 which includes the Codex pulses group and considered a group maximum residue level for the pulses group, however the median residues for peas (dry) and beans (dry) differed by > 5-fold.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in shelled dried beans from supervised trials according to the GAP in the USA was < 0.010, 0.011, 0.019, 0.036, 0.036, 0.043, 0.070, 0.12 and 0.24 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in shelled dried beans from supervised trials according to the GAP in the USA was 0.12, 0.49, 0.64, 0.73, 0.73, 0.87, 1.2, 1.5 and 7.4 mg/kg (HR 7.4 mg/kg).

The Meeting estimated a maximum residue level of 0.4 mg/kg for beans (dry), together with a median residue of 0.73 mg/kg.

In field studies on succeeding crops, the mean, median and highest total residues in dry field peas were 2.49, 2.56 and 3.77 mg/kg respectively. The Meeting decided to add the mean residue found in field studies on succeeding crops of 2.49 mg/kg to the median residue obtained from dried beans residue trials of 0.73 mg/kg for an overall STMR for flupyradifurone in beans (dry) of 3.22 mg/kg.

The Meeting estimated a maximum residue level and an STMR for beans (dry) of 0.4 and 3.22 mg/kg respectively.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in shelled dried peas from supervised trials according to the GAP in the USA was 0.017, 0.13, 0.38, 0.45, 0.47, 0.67, 0.81, 1.0, 1.2 and 1.3 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in shelled dried peas from supervised trials according to the GAP in the USA was 0.067, 0.42, 0.69, 0.82, 0.83, 1.4, 1.7, 2.0, 2.1 and 6.2 mg/kg (HR 6.6 mg/kg).

The Meeting estimated a maximum residue level of 3 mg/kg for peas (dry), together with a median residue of 1.115 mg/kg.

The Meeting decided to add the mean residue found in field studies on succeeding crops of 2.49 mg/kg to the median residue obtained from dried peas residue trials of 1.115 mg/kg for an overall STMR for flupyradifurone in peas (dry) of 3.605 mg/kg.

The Meeting estimated a maximum residue level and an STMR for peas (dry) of 3 and 3.605 mg/kg respectively.

Noting that the use pattern is for the USA Subgroup 6C, the Meeting agreed that an STMR of 2.49 mg/kg (mean residue found in dried peas in field studies on succeeding crops) should apply to pulse crops on the USA label, which are not covered by the recommended Beans (dry) and Peas (dry) MRLs.

Pulses—Soya beans

Residue trials were conducted in soya beans (20 trials) in the USA and Canada, according to the GAP in the USA for dry soya bean seed (two foliar applications at 205 g ai/ha, 10-day RTI, 21-day PHI).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in soya beans from supervised trials according to the GAP in the USA was < 0.010 (4), 0.012, 0.015 (2), 0.019, 0.034, 0.053, 0.068, 0.069, 0.082, 0.15, 0.22, 0.25, 0.28, 0.36, 0.61 and 1.0 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in soya beans from supervised trials according to the GAP in the USA was < 0.060, 0.082, 0.085, 0.090, 0.11, 0.13, 0.25, 0.26, 0.66, 0.70, 1.2, 1.5, 1.7, 1.8, 1.8, 2.0, 3.7, 3.9, 4.1 and 11 (HR 11) mg/kg.

The Meeting estimated a maximum residue level and a median residue of 1.5 and 0.95 mg/kg respectively.

In field studies on succeeding crops, the mean, median and highest total residues in dry field peas were 2.49, 2.56 and 3.77 mg/kg respectively. The Meeting decided to add the mean residue found in field studies on succeeding crops of 2.49 mg/kg to the median residue obtained from soya beans residue trials of 0.95 mg/kg for an overall STMR for flupyradifurone in soya beans of 3.44 mg/kg.

The Meeting estimated a maximum residue level and an STMR for soya beans of 1.5 and 3.44 mg/kg respectively.

Root and tuber vegetables

Carrots and Radishes

Residue trials were conducted in carrots in the USA and Canada according to the critical foliar GAP in the USA for Crop Subgroup 1B (two foliar applications at 205 g ai/ha, 10-day RTI, 7-day PHI).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in carrots from supervised trials according to the GAP in the USA was < 0.010 (2), 0.014, 0.017, 0.020, 0.021, 0.027, 0.037, 0.059 and 0.60 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in carrots from supervised trials according to the GAP in the USA was 0.076, 0.11, 0.11, 0.13, 0.21, 0.23, 0.24, 0.46, 0.60 and 0.68 mg/kg (HR 1.1 mg/kg).

Residue trials were conducted in radishes in the USA and Canada according to the critical foliar GAP in the USA for Crop Subgroup 1B (two foliar applications at 205 g ai/ha, 10-day RTI, 7-day PHI).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in radishes from supervised trials according to the GAP in the USA was 0.024, 0.029, 0.031, 0.037, 0.040, 0.043 and 0.046 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in radishes from supervised trials according to the GAP in the USA was 0.10, 0.11, 0.11, 0.12, 0.19, 0.28 and 0.31 mg/kg (HR 0.32 mg/kg).

The Meeting noted that the GAP was for the subgroup (root and tuber vegetables except potatoes), and considered a group maximum residue level. The median values for the carrot and radish data sets differed by less than 5-fold, and the data sets were statistically similar (Mann-Whitney test), therefore the Meeting agreed to combine the data sets:

For maximum residue level estimation: < 0.010 (2), 0.014, 0.017, 0.020, 0.021, 0.024, 0.027, 0.029, 0.031, 0.037 (2), 0.040, 0.043, 0.046, 0.059 and 0.60 mg/kg.

For dietary intake assessment: 0.076, 0.10, 0.11, 0.11, 0.11, 0.11, 0.12, 0.13, 0.19, 0.21, 0.23, 0.24, 0.28, 0.31, 0.46, 0.60 and 0.68 mg/kg (HR 1.1 mg/kg).

The Meeting estimated a maximum residue level of 0.7 mg/kg for flupyradifurone in root and tuber vegetables (except potato), together with a median and highest residue of 0.19 and 1.1 mg/kg.

In field studies on succeeding crops, the overall mean, median and highest total residues in carrot and turnip roots were 0.10, 0.08 and 0.27 mg/kg respectively. The Meeting decided to add the mean residue found in field studies on succeeding crops of 0.10 mg/kg to the median residue obtained from carrot and radish residue trials of 0.19 mg/kg for an overall STMR for flupyradifurone in root and tuber vegetables (except potato) of 0.29 mg/kg.

The Meeting decided to add the highest residue of 0.27 mg/kg for carrot and turnip roots in the succeeding crop trials to 1.1 mg/kg (the highest residue found in supervised field trials) for an overall HR of 1.37 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for root and tuber vegetables (except potato) of 0.7, 0.29 and 1.37 mg/kg respectively.

Potatoes

Residue trials were conducted in potatoes in the USA and Canada, according to the foliar GAP in the USA for Crop Subgroup 1C (two foliar applications at 205 g ai/ha, 7-day RTI, 7-day PHI).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in potatoes from supervised trials according to the GAP in the USA was < 0.010 (12), 0.010 (2), 0.012, 0.020 (2), 0.022 and 0.037 (2) mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in potatoes from supervised trials according to the GAP in the USA was < 0.060 (9), 0.060, 0.062, 0.070, 0.070, 0.071, 0.072, 0.080, 0.088, 0.096, 0.12 and 0.12 mg/kg (HR 0.14 mg/kg).

The Meeting estimated a maximum residue level of 0.05 mg/kg for flupyradifurone in potatoes, together with a median and highest residue of 0.061 and 0.14 mg/kg respectively.

In field studies on succeeding crops, the mean, median and highest total residues in potatoes were 0.23, 0.20 and 0.43 mg/kg respectively. The Meeting decided to add the mean residue found in field studies on succeeding crops of 0.23 mg/kg to the median residue obtained from potato residue trials of 0.061 mg/kg for an overall STMR for flupyradifurone in potatoes of 0.291 mg/kg.

The Meeting decided to add the highest residue of 0.43 mg/kg for potatoes in the succeeding crop trials to 0.14 mg/kg (the highest residue found in supervised field trials) for an overall HR of 0.57 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for potatoes of 0.05, 0.291 and 0.56 mg/kg respectively.

The Meeting noted that the GAP in the USA for Crop Subgroup 1C also includes sweet potato and agreed that the results of the USA and Canada potato trials matching this GAP could be used to estimate a maximum residue level for sweet potato. The Meeting estimated a maximum residue level, an STMR and an HR for sweet potatoes of 0.05, 0.291 and 0.57 mg/kg respectively.

Stalk and stem vegetables—Celery

Residue trials were conducted in celery (10 trials) in the USA and Canada, according to the GAP in the USA for Crop Group 4 (two foliar applications at 205 g ai/ha, 7-day RTI, 1-day PHI).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in celery stalk from supervised trials according to the GAP in the USA was 0.22, 1.1, 2.0, 2.1, 2.2, 2.4, 3.2, 3.5 and 6.0 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in celery stalk from supervised trials according to the GAP in the USA was 0.27, 1.1, 2.0, 2.2, 2.2, 2.4, 3.2, 3.6 and 6.1 mg/kg (HR 6.8 mg/kg).

The Meeting estimated a maximum residue level of 9 mg/kg for flupyradifurone in celery, together with a median and highest residue and of 2.2 and 6.8 mg/kg (based on the total residue data for untrimmed stalks).

No data from studies on follow crops are available for celery and other stalk and stem vegetables.

In field studies on succeeding crops, the mean, median and highest total residues in leeks were 0.18, 0.13 and 0.39 mg/kg respectively. The Meeting decided to add the mean residue found in field studies on succeeding crops of 0.18 mg/kg to the median residue obtained from celery residue trials of 2.2 mg/kg for an overall STMR for flupyradifurone in celery of 2.38 mg/kg.

The Meeting decided to add the highest residue of 0.39 mg/kg for leeks in the succeeding crop trials to 6.8 mg/kg (the highest residue found in supervised field trials) for an overall HR of 7.19 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for celery of 9, 2.38 and 7.19 mg/kg respectively.

Short term intake assessment showed that residues in celery exceed the acute reference dose (ARfD) of 0.2 mg/kg bw, at 120% for children. No alternative GAP for celery was available.

Cereal grains—Barley, Maize, Sorghum and Wheat

Twenty residue trials were conducted in barley in the USA and Canada according to the GAP in the USA for Crop Group 15 (except rice) (two foliar applications at 205 g ai/ha, 7-day RTI, 21-day PHI). Three trials which showed residues after planting seed treated with flupyradifurone are not considered as there is no registered use pattern.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in barley grain from supervised trials according to the GAP in the USA was 0.038, 0.065, 0.096, 0.21, 0.24, 0.25, 0.27, 0.30, 0.31, 0.44, 0.46, 0.48, 0.68 (2), 0.71, 0.81, 0.84, 1.2, 1.7 and 2.3 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in barley grain from supervised trials according to the GAP in the USA was 0.53, 0.55, 0.67, 0.69, 0.74, 0.78, 0.79, 0.82, 0.83, 0.88, 0.89, 0.91, 1.1, 1.3, 1.3, 1.3, 1.4, 1.5, 1.8 and 2.4 mg/kg (HR 2.5 mg/kg).

Twenty-nine residue trials were conducted in wheat in the USA and Canada, twenty-eight according to the GAP in the USA for Crop Group 15 (except rice) (two foliar applications at 205 g ai/ha, 7-day RTI, 21-day PHI). Three trials which showed residues after planting seed treated with flupyradifurone are not considered as there is no registered use pattern.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in wheat grain from supervised trials according to the GAP in the USA was 0.016, 0.024, 0.031, 0.033, 0.034, 0.040, 0.050, 0.059, 0.074, 0.090 (2), 0.10 (2), 0.15 (2), 0.16 (2), 0.17, 0.18, 0.21, 0.22, 0.23, 0.26, 0.34, 0.37, 0.58, 0.61 and 0.73 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in wheat grain from supervised trials according to the GAP in the USA was 0.083, 0.090, 0.10, 0.21, 0.24, 0.27, 0.30, 0.30, 0.38, 0.57, 0.60, 0.66, 0.67, 0.70, 0.74, 0.77, 0.78, 0.79, 0.80, 0.83, 0.90, 0.93, 1.1, 1.2, 1.6, 1.9, 2.6 and 2.7 mg/kg (HR 2.8 mg/kg).

Nine residue trials were conducted in sorghum in the USA, according to the GAP in the USA for Crop Group 15 (except rice) (two foliar applications at 205 g ai/ha, 7-day RTI, 21-day PHI). Three trials which showed residues after planting seed treated with BYI 02960 480FS are not considered as there is no registered use pattern.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in sorghum grain from supervised trials according to the GAP in the USA was 0.34, 0.46 (2), 0.50, 0.51, 0.79, 0.86, 1.4 and 1.5 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in sorghum grain from supervised trials according to the GAP in the USA was 0.41, 0.54, 0.58, 0.67, 0.70, 0.89, 0.94, 1.5 and 1.6 mg/kg (HR 1.9 mg/kg).

Twenty residue trials were conducted in field corn in the USA, according to or approximating the GAP in the USA for Crop Group 15 (except rice) (two foliar applications at 205 g ai/ha, 7-day RTI, 21-day PHI).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in field corn grain from supervised trials according to the GAP in the USA was < 0.01 (15) and 0.011 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in field corn grain from supervised trials according to the GAP in the USA was < 0.060 (14), 0.061 and 0.13 mg/kg (HR 0.21 mg/kg).

The Meeting noted that the GAP was for the cereal grains group, other than rice and considered a group maximum residue level. The median residues for barley, wheat and sorghum differed by less than 5-fold, although the data sets were not statistically similar (Kruskal-Wallis test).

The Meeting estimated a maximum residue level of 3 mg/kg for flupyradifurone in cereal grains (except maize and rice) based on the data set for barley, together with a median residue of 0.885 mg/kg.

In field studies on succeeding crops, the overall mean, median and highest total residues in barley grain were 0.43, 0.22 and 1.3 mg/kg respectively. The Meeting decided to add the mean residue found in field studies on succeeding crops of 0.43 mg/kg to the median residue obtained from the barley residue trials of 0.885 mg/kg for an overall STMR for flupyradifurone in cereal grains (except maize and rice) of 1.315 mg/kg.

The Meeting estimated a maximum residue level and an STMR for cereal grains (except maize and rice) of 3 and 1.315 mg/kg respectively.

The Meeting estimated a maximum residue level of 0.015 mg/kg for flupyradifurone in maize, together with a median residue of 0.06 mg/kg.

In field studies on succeeding crops, the overall mean, median and highest total residues in barley grain were 0.43, 0.22 and 1.3 mg/kg respectively. The Meeting decided to add the mean residue found in field studies on succeeding crops of 0.43 mg/kg to the median residue obtained from the maize residue trials of 0.06 mg/kg for an overall STMR for flupyradifurone in maize of 0.49 mg/kg.

The Meeting estimated a maximum residue level and an STMR for maize of 0.015 and 0.49 mg/kg respectively.

Tree nuts—Pecans

Five residue trials were conducted in pecans in the USA according to the critical foliar GAP in the USA for Crop Group 14 except almonds (two foliar applications at 205 g ai/ha, 14-day RTI, 7-day PHI). Both concentrated and dilute applications were made. The highest observations for estimation of maximum residue levels and for dietary intake purposes have been selected from each trial.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in pecans nutmeat without shell from supervised trials according to the GAP in the USA was < 0.010 (4) and 0.012 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in pecans nutmeat without shell from supervised trials according to the GAP in the USA was < 0.060 (4) and 0.062 mg/kg (HR 0.063 mg/kg).

Based on the dataset, the Meeting estimated a maximum residue level, an STMR and an HR of 0.015, 0.060 and 0.063 mg/kg respectively, for flupyradifurone in pecans.

Oilseeds—Cotton and Peanuts

Twelve residue trials were conducted in cotton in the USA, eleven according to or approximating the GAP in the USA for cotton (two foliar applications at 205 g ai/ha, 10-day RTI, 14-day PHI). Three of these trials also included plots to measure residues following planting of seed treated with BYI 02960 480 FS. The latter method of application is not registered and is not discussed further.

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in cotton seed from supervised trials (foliar use pattern) according to the GAP in the USA was 0.014, 0.018, 0.074, 0.081, 0.12, 0.13, 0.18, 0.20, 0.40 and 0.49 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in cotton seed from supervised trials (foliar use pattern) according to the GAP in the USA was 0.072, 0.12, 0.13, 0.21, 0.23, 0.24, 0.27, 0.47, 0.63 and 0.74 mg/kg (HR 0.86 mg/kg).

Based on the dataset, the Meeting estimated a maximum residue level and a median residue of 0.8 and 0.235 mg/kg respectively.

In field studies on succeeding crops, the overall mean, median and highest total residues in an oilseed crop (rape seed) were 0.16, 0.15 and 0.26 mg/kg respectively. The Meeting decided to add the mean residue found in rape seed field studies on succeeding crops of 0.16 mg/kg to the median residue obtained from the cotton residue trials of 0.235 mg/kg for an overall STMR for flupyradifurone in cotton seed of 0.395 mg/kg.

The Meeting estimated a maximum residue level and an STMR for cotton seed of 0.8 and 0.395 mg/kg respectively.

Twelve residue trials were conducted in peanuts in the USA, eleven according to or approximating the GAP in the USA for peanuts (two foliar applications at 205 g ai/ha, 10-day RTI, 7-day PHI).

For the estimation of the maximum residue level the ranked order of residues of flupyradifurone in peanuts from supervised trials according to the GAP in the USA was < 0.01 (5), 0.011, 0.014, 0.017 and 0.027 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in peanuts from supervised trials according to the GAP in the USA was < 0.060 (4), 0.065, 0.067, 0.069, 0.082 and 0.087 mg/kg (HR 0.090 mg/kg

Based on the dataset, the Meeting estimated a maximum residue level, a median and highest residue of 0.04, 0.065 and 0.090 mg/kg respectively.

In field studies on succeeding crops, the overall mean, median and highest total residues in rape seed were 0.16, 0.15 and 0.26 mg/kg respectively. The Meeting decided to add the mean residue found in field studies on succeeding crops of 0.16 mg/kg to the median residue obtained from the peanut residue trials of 0.065 mg/kg for an overall STMR for flupyradifurone in peanuts of 0.225 mg/kg.

The Meeting decided to add the highest residue of 0.26 mg/kg for rape seed in the succeeding crop trials to 0.090 mg/kg (the highest residue found in supervised field trials) for an overall HR of 0.35 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for peanuts of 0.04, 0.225 and 0.35 mg/kg respectively.

Hops

Residue trials were conducted in hops in the USA according to the critical GAP in the USA for hops (one foliar application at 153 g ai/ha, 21-day PHI). Both concentrated and dilute applications were made. The highest observations for estimation of maximum residue levels and for dietary intake purposes have been selected from each trial.

The ranked order of residues of flupyradifurone in dried hops from supervised trials according to the GAP in the USA were in rank order 2.4, 2.7 and 4.7 mg/kg.

For the estimation of dietary intake, the ranked order of total residues of flupyradifurone in dried hops from supervised trials according to the GAP in the USA were in rank order 3.3, 3.3 and 8.0 mg/kg.

No maximum residue level was recommended due to the limited data (n = 3 trials).

Animal feeds

The Meeting received supervised trials data for alfalfa forage and hay, clover forage and hay, pea, bean and soya bean forage and hay, peanut hay, barley hay and straw, maize and sweet corn forage and stover, sorghum forage and stover, wheat forage, hay and straw, almond hull and cotton gin by-products.

Where available, supplied moisture content values have been used to calculate residues on a dry weight basis. Where no moisture content for samples was provided, the default OECD values for moisture content were used.

Legume forages

No data from studies on legume forages as follow crops are available. In field studies on succeeding crops, the overall mean, median and highest total residues in barley forage were 0.19, 0.08 and 0.80 mg/kg respectively. Assuming a dry matter content of 30%, dry weight mean, median and highest residues in barley forage are 0.63, 0.27 and 2.7 mg/kg. The Meeting concluded that residues in legume forages due to an additional uptake *via* the roots, are insignificant in comparison to residue levels following direct treatment.

Bean forage

Residue trials were conducted in beans (10 trials) in the USA and Canada according to the GAP in the USA for Crop Group 6 (two foliar applications at 205 g ai/ha, 10-day RTI and a 7-day PHI for bean forage).

For the calculation of the livestock animal dietary burden, the ranked order of total residues (fresh weight) of flupyradifurone in bean forage from supervised trials according to the GAP in the USA was 0.27, 1.0, 1.2, 1.4, 1.7, 1.7, 1.8, 2.8, 3.2 and 4.1 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues in bean forage samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 1.1, 7.9, 9.2, 9.5, 12, 13, 14, 17, 18 and 21 mg/kg (HR 21 mg/kg).

The Meeting estimated median and highest residues values for flupyradifurone in bean forage of 12.5 and 21 mg/kg respectively (dry weight).

Pea vines (forage)

Residue trials were conducted in peas (10 trials) in the USA according to or approximating the GAP in the USA for Crop Group 6 (two foliar applications at 205 g ai/ha, 10-day RTI, and a 7-day PHI for pea forage).

For the calculation of the livestock animal dietary burden, the ranked order of total residues (fresh weight) of flupyradifurone in pea vines from supervised trials according to the GAP in the USA was 2.2, 3.3, 3.8, 3.9, 4.1, 4.4, 5.0, 5.1, 5.3 and 5.3 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues in pea vines samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 14, 16, 17, 20, 22, 23, 25, 27, 28 and 43 mg/kg (HR 44 mg/kg).

The Meeting estimated median and highest residues values for flupyradifurone in pea forage of 22.5 and 44 mg/kg respectively (dry weight).

Soya bean forage

Residue trials were conducted in soya beans (20 trials) in the USA and Canada according to the GAP in the USA for Crop Group 6 (two foliar applications at 205 g ai/ha, 10-day RTI and a 7-day PHI for soya bean forage).

For the calculation of the livestock animal dietary burden, the ranked order of total residues (fresh weight) of flupyradifurone in soya bean forage from supervised trials according to the GAP in the USA was 1.7, 2.1, 2.5, 3.3, 3.6, 3.8, 4.1, 4.4 (2), 4.5, 4.9, 5.0, 5.1, 5.3, 5.3, 5.4, 5.6, 5.6, 5.9 and 8.2 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues in soya bean forage samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 8.8, 11, 14, 15, 16, 19, 21, 21, 22, 23, 23, 23, 24, 24, 24, 29, 31, 35, 36 and 41 mg/kg (HR 46 mg/kg).

The Meeting estimated median and highest residues values for flupyradifurone in soya bean forage of 23 and 46 mg/kg respectively (dry weight).

Alfalfa forage

Residue trials were conducted in alfalfa (13 trials) in the USA according to the GAP in the USA for alfalfa (two foliar applications at 205 g ai/ha, 10-day RTI, and a 7-day PHI for alfalfa forage).

For the calculation of the livestock animal dietary burden, the ranked order of total residues (fresh weight) of flupyradifurone in alfalfa forage from supervised trials according to the GAP in the USA was 1.4, 1.6, 2.9, 3.8, 4.0, 4.4, 4.4, 5.0, 5.0, 5.3, 5.6, 6.9 and 8.8 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues in alfalfa forage samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 4.9, 7.9, 12, 16, 18, 19, 20, 22, 25, 26, 27, 34 and 46 mg/kg (HR 51 mg/kg).

The Meeting estimated median and highest residues values for flupyradifurone in alfalfa forage of 20 and 51 mg/kg respectively (dry weight).

Clover forage

Residue trials were conducted in clover (four trials) in the USA according to the GAP in the USA for clover (two foliar applications at 205 g ai/ha, 10-day RTI and a 7-day PHI for clover forage).

For the calculation of the livestock animal dietary burden, the ranked order of total residues (fresh weight) of flupyradifurone in clover forage from supervised trials according to the GAP in the USA at three locations was 4.7, 5.9 and 6.2 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues (dry weight) of flupyradifurone in clover forage from supervised trials according to the GAP in the USA at three locations was 16, 20 and 21 mg/kg.

The Meeting considered that there were insufficient data on which to base estimates of the median and highest residues.

Legume fodders

No data from studies on legume fodders as follow crops are available. In field studies on succeeding crops, the overall mean, median and highest total residues in barley fodder (straw) were 0.22, < 0.12 and 0.78 mg/kg respectively. Assuming a dry matter content of 89%, dry weight mean, median and highest residues in barley straw are 0.25, 0.13 and 0.88 mg/kg. The Meeting concluded that residues in legume fodders due to an additional uptake *via* the roots, are insignificant in comparison to residue levels following direct treatment.

Bean hay

Residue trials were conducted in beans (nine trials) in the USA and Canada according to the GAP in the USA for Crop Group 6 (two foliar applications at 205 g ai/ha, 10-day RTI and a 7-day PHI for bean hay).

For the calculation of the maximum residue level, the ranked order of residues (fresh weight) of flupyradifurone in bean hay from supervised trials according to the GAP in the USA was < 0.040, 0.71, 2.2, 2.7, 3.0, 4.7, 7.6, 7.9 and 9.8 mg/kg.

For the calculation of the maximum residue level the ranked order of residues in bean hay samples converted to a dry weight basis from supervised trials according to the GAP in the USA was < 0.049, 0.80, 2.8, 3.2, 4.2, 5.9, 8.6, 12 and 15 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues (fresh weight) of flupyradifurone in bean hay from supervised trials according to the GAP in the USA was < 0.24, 2.0, 2.4, 3.7, 4.1, 5.9, 8.7, 10 and 11 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues in bean hay samples converted to a dry weight basis from supervised trials according to the GAP in the USA was < 0.30, 2.3, 3.1, 4.4, 5.7, 7.4, 9.9, 15 and 16 mg/kg (HR 17 mg/kg).

The Meeting estimated maximum residue level, median and highest residues values for flupyradifurone in bean hay of 30, 5.7 and 17 mg/kg (dry weight) respectively.

Pea hay

Residue trials were conducted in peas (10 trials) in the USA and Canada according to the GAP in the USA for Crop Group 6 (two foliar applications at 205 g ai/ha, 10-day RTI and a 7-day PHI for pea hay).

For the calculation of the maximum residue level, the ranked order of residues (fresh weight) of flupyradifurone in pea hay from supervised trials according to the GAP in the USA was 4.7, 5.0, 6.4, 6.8, 8.0, 8.2, 9.1, 9.9, 10 and 15 mg/kg.

For the calculation of the maximum residue level, the ranked order of residues in pea hay samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 7.8, 8.9, 9.8, 11, 12, 16, 19, 24, 26 and 33 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues (fresh weight) of flupyradifurone in pea hay from supervised trials according to the GAP in the USA was 5.9, 6.8, 8.8, 9.8, 11, 11, 11, 12, 13 and 19 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues in pea hay samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 11, 12, 14, 14, 17, 22, 22, 30, 33 and 36 mg/kg (HR 36 mg/kg).

The Meeting estimated maximum residue level, median and highest residues values for flupyradifurone in pea hay of 50, 19.5 and 36 mg/kg (dry weight) respectively.

Soya bean hay

Residue trials were conducted in soya beans (20 trials) in the USA and Canada according to the GAP in the USA for Crop Group 6 (two foliar applications at 205 g ai/ha, 10-day RTI and a 7-day PHI for soya bean hay).

For the calculation of the maximum residue level, the ranked order of residues (fresh weight) of flupyradifurone in soya bean hay from supervised trials according to the GAP in the USA was 1.5, 2.6, 3.9, 4.2, 6.2, 6.4, 6.6, 6.6, 6.7, 6.9, 8.1, 8.3, 8.5, 8.5, 9.3, 11, 12, 13, 15 and 17 mg/kg.

For the calculation of the maximum residue level the ranked order of residues in soya bean hay samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 2.1, 4.4, 4.6, 6.2, 7.2, 8.2, 8.6, 8.8, 9.3, 10, 12, 15, 15, 15, 15, 16, 18, 19, 20 and 29 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues (fresh weight) of flupyradifurone in soya bean hay from supervised trials according to the GAP in the USA was 3.4, 6.7, 8.4, 9.4, 9.5, 9.8, 10, 10, 10, 11, 11, 11, 11, 13, 13, 15, 16, 18, 20 and 21 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues in soya bean hay samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 4.8, 11, 13, 13, 13, 13, 13, 13, 14, 15, 16, 18, 21, 22, 22, 23, 23, 24, 26 and 36 mg/kg (HR 41 mg/kg).

The Meeting estimated maximum residue level, median and highest residues values for flupyradifurone in soya bean hay of 40, 15.5 and 41 mg/kg (dry weight) respectively.

Alfalfa hay

Residue trials were conducted in alfalfa (13 trials) in the USA according to the GAP in the USA for alfalfa (two foliar applications at 205 g ai/ha, 10-day RTI and a 7-day PHI for alfalfa hay).

For the calculation of the maximum residue level, the ranked order of residues (fresh weight) of flupyradifurone in alfalfa hay from supervised trials according to the GAP in the USA was 2.5, 2.7, 4.2, 4.8, 5.5, 5.9, 6.1, 6.2, 7.4, 8.4, 9.3, 9.4 and 9.5 mg/kg.

For the calculation of the maximum residue level, the ranked order of residues in alfalfa hay samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 3.0, 3.2, 5.1, 7.3, 7.9, 9.0, 9.2, 9.3, 10, 11, 13, 13 and 15 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues (fresh weight) of flupyradifurone in alfalfa hay from supervised trials according to the GAP in the USA was 7.1, 7.4, 7.4, 7.7, 8.2, 8.3, 10, 10, 11, 13, 14, 15 and 25 mg/kg.

For the calculation of the livestock animal dietary burden the ranked order of total residues in alfalfa hay samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 7.6, 9.1, 9.4, 12, 13, 13, 14, 16, 18, 18, 19, 19 and 36 mg/kg (HR 42 mg/kg).

The Meeting estimated maximum residue level, median and highest residues values for flupyradifurone in alfalfa hay of 30, 14 and 42 mg/kg (dry weight) respectively.

Clover hay

Residue trials were conducted in clover (four trials) in the USA according to the GAP in the USA for clover (two foliar applications at 205 g ai/ha, 10-day RTI and a 14-day PHI for clover hay) and also at approximately 0.5× the registered rate.

Sampling of four clover hay samples at the registered rate was conducted at 11, 12, 14 and 17-PHI. As the 12 and 14 day samples are from the same location, the Meeting considered that insufficient data was available at GAP to recommend an MRL or dietary parameters.

Peanut hay

Residue trials were conducted in peanuts (12 trials), in the USA according to the GAP in the USA for peanuts (two foliar applications at 205 g ai/ha, 10-day RTI, and a 7-day PHI for peanut hay).

For the calculation of the maximum residue level, the ranked order of residues (fresh weight) of flupyradifurone in peanut hay from supervised trials according to the GAP in the USA was 1.7, 2.0, 2.7, 3.7, 4.5, 5.0, 9.1, 10, 11 and 11 mg/kg.

For the calculation of the maximum residue level, the ranked order of residues in peanut hay samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 2.0, 4.1, 5.4, 5.4, 5.7, 6.4, 10, 12, 13 and 16 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked total order of total residues (fresh weight) of flupyradifurone in peanut hay from supervised trials according to the GAP in the USA was 3.8, 5.4 (2), 5.5, 6.0 (2), 12 and 13 (3) mg/kg.

For the calculation of the livestock animal dietary burden the ranked order of total residues in peanut hay samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 6.0, 6.4, 7.2, 7.7, 10, 12, 14, 15 (2) and 19 mg/kg (HR 20 mg/kg).

The Meeting estimated maximum residue level, median and highest residues values for flupyradifurone in peanut hay of 30, 11 and 20 mg/kg (dry weight) respectively.

Wheat, sorghum, maize and sweet corn forages

In field studies on succeeding crops, the overall mean, median and highest total residues in barley forage were 0.19, 0.08 and 0.80 mg/kg respectively. Assuming a dry matter content of 30%, dry weight mean, median and highest residues in barley forage are 0.63, 0.27 and 2.7 mg/kg. The Meeting concluded that residues in cereal forages due to an additional uptake *via* the roots, are insignificant in comparison to residue levels following direct treatment.

Wheat forage

Residue trials were conducted in wheat (29 trials) in the USA and Canada according to the GAP in the USA for Crop Group 15 (except rice) (two foliar applications at 205 g ai/ha, 7-day RTI, and a 7-day PHI for wheat forage).

For the calculation of the livestock animal dietary burden, the ranked order of total residues (fresh weight) of flupyradifurone in wheat forage from supervised trials according to the GAP in the USA was 0.095, 0.11, 0.12, 0.12, 0.15, 0.15, 0.17, 0.22, 0.22, 0.27, 0.29, 0.41, 0.54, 0.55, 0.59, 0.62, 0.72, 1.1, 2.2, 2.3, 2.5, 2.8, 5.7, 5.9, 6.4, 9.5, 13 and 15 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues in wheat forage samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 0.68, 0.69, 0.85, 0.85, 0.89, 1.0, 1.1, 1.3, 1.7, 1.8, 2.1, 2.4, 2.6, 2.9, 3.4, 3.8, 4.9, 5.4, 12, 13, 14, 15, 21, 24, 25, 41, 51 and 68 mg/kg (HR 77 mg/kg).

Sorghum forage

Residue trials were conducted in sorghum (nine trials) in the USA according to the GAP in the USA for cereal grains (except rice) (two foliar applications at 205 g ai/ha, 7-day RTI, 7-day PHI for sorghum forage).

For the calculation of the livestock animal dietary burden, the ranked order of total residues (fresh weight) of flupyradifurone in sorghum forage from supervised trials according to the GAP in the USA was 2.3 (2), 2.5, 2.8 (2), 3.2, 3.4, 4.2 and 4.3 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues in sorghum forage samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 6.5 (2), 6.6, 6.8, 9.7, 10, 11 and 12 (2) mg/kg (HR 14 mg/kg).

Maize and sweet corn forage

Residue trials were conducted in maize (20 trials) and sweet corn (13 trials) in the USA according to the GAP in the USA for cereal grains (except rice) (two foliar applications at 205 g ai/ha, 7-day RTI, 7-day PHI for maize and sweet corn forage).

For the calculation of the livestock animal dietary burden, the ranked order of total residues (fresh weight) of flupyradifurone in maize and sweet corn forage from supervised trials according to the GAP in the USA was 0.79, 0.87, 1.0, 1.4, 1.5, 1.5, 1.6, 1.7, 1.7, 1.8, 1.8, 1.9, 1.9, 1.9, 2.0, 2.0, 2.3, 2.5, 2.5, 2.6, 2.6, 2.8, 2.9, 2.9, 3.1, 3.3, 3.3, 3.8, 4.0 and 9.4 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues in maize and sweet corn forage samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 3.7, 4.4, 4.5, 4.8, 4.8, 5.0, 5.5, 5.6, 5.8, 5.9, 6.0, 6.9, 6.9, 7.0, 7.6, 7.8, 8.0, 8.4, 8.5, 9.2, 10, 10, 11, 11, 12, 12, 14, 15, 25 and 58 (HR 65 mg/kg).

The Meeting noted that the GAPs for wheat, sorghum and maize and sweet corn forage were the same (2× 205 g ai/ha foliar applications, 7-day RTI and a 7-day PHI). The medians for the data sets differed by less than 5-fold and the data sets are statistically similar (Kruskal-Wallis). The Meeting agreed to combine the data sets for wheat, sorghum and maize and sweet corn forage for the purpose of estimating median and highest residue values.

Combined total residues data set for cereal forages (dry weight): 0.68, 0.69, 0.85, 0.85, 0.89, 1.0, 1.1, 1.3, 1.7, 1.8, 2.1, 2.4, 2.6, 2.9, 3.4, 3.7, 3.8, 4.4, 4.5, 4.8, 4.8, 4.9, 5.0, 5.4, 5.5, 5.6, 5.8, 5.9, 6.0, 6.5, 6.5, 6.6, 6.8, 6.9, 6.9, 7.0, 7.6, 7.8, 8.0, 8.4, 8.5, 9.2, 9.7, 10, 10, 10, 11, 11, 11, 12, 12, 12, 12, 12, 13, 14, 14, 15, 15, 21, 24, 25, 25, 41, 51, 58 and 68 mg/kg (HR 77 mg/kg).

The Meeting estimated a median residue and a highest residue of 6.9 and 77 mg/kg (dry weight) respectively for flupyradifurone in wheat, sorghum and maize forage, and agreed that these values could be extrapolated to all forages of cereal grains except rice.

Barley and wheat hay and straw, and sorghum and maize and sweet corn stovers

In field studies on succeeding crops, the overall mean, median and highest total residues in barley fodder (straw) were 0.22, < 0.12 and 0.78 mg/kg respectively. Assuming a dry matter content of 89%, dry weight mean, median and highest residues in barley straw are 0.25, 0.13 and 0.88 mg/kg. The Meeting concluded that residues in cereal fodders due to an additional uptake *via* the roots, are insignificant in comparison to residue levels following direct treatment.

Barley hay

Residue trials were conducted in barley (20 trials) in the USA and Canada according to the GAP in the USA for Crop Group 15 (except rice) (two foliar applications at 205 g ai/ha, 7-day RTI, and a 7-day PHI for barley hay).

For the calculation of the maximum residue level, the ranked order of residues (fresh weight) of flupyradifurone in barley hay from supervised trials according to the GAP in the USA was 0.33, 0.42, 0.73, 1.5, 1.6, 1.8, 2.0, 2.7, 3.3, 3.8, 5.2, 5.4, 5.8, 7.2, 8.8, 12, 17 and 24 mg/kg.

For the calculation of the maximum residue level, the ranked order of residues in barley hay samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 0.51, 1.1, 1.6, 2.1, 2.4, 2.9, 3.2, 4.4, 4.6, 4.8, 5.6, 6.4, 9.9, 11, 12, 19, 22 and 28 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of residues (fresh weight) of flupyradifurone in barley hay from supervised trials according to the GAP in the USA was 0.66, 0.89, 1.0, 1.9, 2.0, 2.2, 2.3, 2.4, 3.7, 4.4, 5.5, 6.1, 6.4, 7.5, 9.6, 14, 18 and 25 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of residues in barley hay samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 1.0, 2.2, 2.4, 2.9, 3.0, 3.2, 3.7, 3.8, 5.2, 5.4, 5.9, 7.2, 11, 11, 13, 22, 23 and 29 mg/kg (HR 31 mg/kg).

Barley straw

Residue trials were conducted in barley (20 trials) in the USA and Canada according to the GAP in the USA for Crop Group 15 (except rice) (two foliar applications at 205 g ai/ha, 7-day RTI, and a 21-day PHI for barley straw).

For the calculation of the maximum residue level, the ranked order of residues (fresh weight) of flupyradifurone in barley straw from supervised trials according to the GAP in the USA was 0.31, 0.33, 0.42, 0.42, 0.52, 0.61, 0.77, 0.92, 1.0, 1.3, 1.3, 1.4, 2.2, 2.5, 3.1, 3.8, 3.8, 4.0, 5.1 and 5.6 mg/kg.

For the calculation of the maximum residue level, the ranked order of residues in barley straw samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 0.77, 0.82, 0.94, 1.0, 1.1, 1.1, 1.2, 1.5, 1.7, 1.7, 2.8, 2.9, 3.5, 4.9, 5.7, 5.7, 6.6, 6.9, 7.3 and 12 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of residues (fresh weight) of flupyradifurone in barley straw from supervised trials according to the GAP in the USA was 0.41, 0.49, 0.58, 0.82, 0.83, 1.0, 1.1, 1.2, 1.3, 1.4, 1.4, 1.5, 2.5, 2.5, 3.5, 3.9, 4.1, 4.2, 5.2 and 5.6 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of residues in barley straw samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 1.2, 1.4, 1.4, 1.4, 1.5, 1.8, 1.8, 1.9, 1.9, 1.9, 3.2, 3.5, 4.0, 5.0, 5.9, 6.0, 6.7, 7.7, 7.8 and 12 mg/kg (HR 12 mg/kg).

Wheat hay

Residue trials were conducted in wheat (29 trials) in the USA and Canada according to the GAP in the USA for Crop Group 15 (except rice) (two foliar applications at 205 g ai/ha, 7-day RTI, and a 7-day PHI for wheat hay).

For the calculation of the maximum residue level, the ranked order of residues (fresh weight) of flupyradifurone in wheat hay from supervised trials according to the GAP in the USA was 2.5, 2.6, 3.5, 3.6, 4.3, 5.5, 5.7, 5.7, 5.9, 6.4, 6.6, 6.7, 7.2, 7.5, 7.8, 8.0, 8.1, 8.3, 8.4, 9.2, 9.9, 11, 11, 12, 13, 16, 16, 17 and 22 mg/kg.

For the calculation of the maximum residue level the ranked order of residues in wheat hay samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 3.2, 3.3, 4.2, 4.9, 5.3, 6.6, 6.6, 6.6, 6.7, 7.5, 8.0, 8.5, 8.7, 9.0, 9.0, 9.3, 9.3, 9.7, 10, 11, 11, 12, 13, 14, 16, 19, 19, 20 and 26 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of residues (fresh weight) of flupyradifurone in wheat hay from supervised trials according to the GAP in the USA was 3.5, 3.6, 3.9, 4.1, 5.6, 5.8, 5.9, 6.2, 6.4, 7.0, 7.3, 7.4, 7.7, 7.8, 8.2, 8.5, 8.9, 9.4, 9.9, 9.9, 10, 11, 12, 13, 13, 17, 18, 18 and 23 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of residues in wheat hay samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 4.4, 4.7, 4.7, 6.0, 6.4, 6.9, 7.0, 7.1, 7.2, 8.1, 8.3, 9.0, 9.1, 9.4, 9.6, 10, 10, 12, 12, 12, 12, 12, 15, 15, 17, 19, 20, 21 and 26 mg/kg (HR 28 mg/kg).

Wheat straw

Residue trials were conducted in wheat (29 trials) in the USA and Canada according to the GAP in the USA for Crop Group 15 (except rice) (two foliar applications at 205 g ai/ha, 7-day RTI, and a 21-day PHI for wheat straw).

For the calculation of the maximum residue level, the ranked order of residues (fresh weight) of flupyradifurone in wheat straw from supervised trials according to the GAP in the USA was 0.24, 0.44, 0.58, 0.66, 0.93, 1.0, 1.1, 2.0, 2.0, 2.1, 2.5, 3.5, 3.7, 3.8, 4.0, 4.6, 4.6, 5.0, 5.7, 6.1, 6.9, 7.0, 7.0, 7.7, 8.1, 11, 13 and 19 mg/kg.

For the calculation of the maximum residue level, the ranked order of residues in wheat straw samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 0.66, 1.0, 1.3, 1.5, 1.6, 1.7, 2.4, 2.5, 2.9, 3.9, 4.2, 4.4, 4.4, 5.8, 5.8, 6.2, 6.5, 6.9, 7.9, 8.5, 8.5, 8.9, 9.2, 9.3, 11, 13, 15 and 23 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of residues (fresh weight) of flupyradifurone in wheat straw from supervised trials according to the GAP in the USA was 0.60, 0.95, 1.4, 1.5, 1.5, 1.6, 2.0, 2.3, 2.7, 3.0, 3.6, 3.8, 3.9, 4.1, 4.6, 4.7, 5.2, 5.3, 6.1, 6.7, 6.9, 7.0, 7.1, 7.9, 8.1, 11, 13 and 19 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of residues in wheat straw samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 1.7, 2.2, 2.5, 2.8, 2.9, 3.0, 3.6, 3.8, 3.9, 4.6, 4.6, 5.2, 6.0, 6.3, 6.3, 6.5, 7.8, 8.0, 8.2, 8.6, 8.9, 9.2, 9.4, 9.4, 12, 13, 15 and 23 mg/kg (HR 23 mg/kg).

Sorghum stover

Residue trials were conducted in sorghum (nine trials) in the USA according to the GAP in the USA for Crop Group 15 (except rice) (two foliar applications at 205 g ai/ha, 7-day RTI, 21-day PHI for sorghum stover).

For the calculation of the maximum residue level, the ranked order of residues (fresh weight) of flupyradifurone in sorghum stover from supervised trials according to the GAP in the USA was 0.97, 1.2, 1.3, 1.7, 2.3, 2.4 (2), 2.7 and 5.0 mg/kg.

For the calculation of the maximum residue level, the ranked order of residues in sorghum stover samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 1.1, 1.4, 1.5, 1.9, 2.6, 2.7 (2), 3.1 and 5.7 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues (fresh weight) of flupyradifurone in sorghum stover from supervised trials according to the GAP in the USA was 1.1, 1.3, 1.4, 1.8, 2.4, 2.6, 2.7, 2.9 and 5.3 mg/kg.

For the calculation of the livestock animal dietary burden the ranked order of total residues in sorghum stover samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 1.3, 1.5, 1.6, 2.0, 2.7, 3.0, 3.1, 3.3 and 6.0 mg/kg (HR 6.9 mg/kg).

Maize and sweet corn stover

Residue trials were conducted in maize (20 trials) and sweet corn (13 trials) in the USA according to the GAP in the USA for Crop Group 15 (except rice) (two foliar applications at 205 g ai/ha, 7-day RTI, 21-day PHI for maize and sweet corn stover).

For the calculation of the maximum residue level, the ranked order of residues (fresh weight) of flupyradifurone in maize and sweet corn stover from supervised trials according to the GAP in the USA was 0.53, 0.90, 1.1, 1.1, 1.2, 1.2, 1.4, 1.4, 1.4, 1.5, 1.5, 1.6, 1.7, 1.7, 1.8, 1.9, 2.1, 2.2, 3.0, 3.1, 3.2, 3.2, 3.4, 3.5, 3.6, 3.6, 4.6, 5.1, 5.9 and 8.2 mg/kg.

For the calculation of the maximum residue level, the ranked order of residues in maize and sweet corn stover samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 1.6, 1.8, 2.2, 2.3, 2.3, 2.6, 2.9, 3.0, 3.2, 3.2, 3.2, 3.9, 4.2, 4.7, 5.0, 5.2, 5.3, 5.7, 5.9, 6.1, 6.2, 6.2, 6.6, 9.3, 9.9, 10, 11, 12, 12 and 27 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of residues (fresh weight) of flupyradifurone in maize and sweet corn stover from supervised trials according to the GAP in the USA was 0.67, 0.95, 1.1, 1.2, 1.3, 1.3, 1.5, 1.5, 1.5, 1.5, 1.7, 1.7, 1.8, 1.8, 1.9, 2.1, 2.2, 2.2, 3.0, 3.3, 3.3, 3.4, 3.6, 3.6, 3.8, 3.8, 4.7, 5.2, 5.9 and 8.5 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of residues in maize and sweet corn stover samples converted to a dry weight basis from supervised trials according to the GAP in the USA was 1.8, 1.9, 2.1, 2.4, 2.8, 2.9, 3.0, 3.3, 3.3, 3.7, 4.1, 4.3, 4.7, 5.3, 5.5, 5.6, 6.0, 6.0, 6.3, 6.6, 6.6, 6.7, 6.8, 9.7, 11, 11, 11, 12, 12 and 28 mg/kg (HR 32 mg/kg).

The Meeting noted that the GAPs for wheat and barley hay were the same (2× 205 g ai/ha foliar applications, with a 7-day RTI and a 7-day PHI) and the GAPs for wheat and barley straw and sorghum and maize stover were the same (2× 205 g ai/ha foliar applications, with a 7-day RTI and a 21-day PHI). The Meeting considered a group maximum residue level for straw and fodder of cereal grains. The Meeting noted that the median residues (for estimation of maximum residue levels) for the cereal straws and stovers differed by less than 5-fold, however the data sets were not statistically similar (Kruskal-Wallis). The Meeting similarly noted that the median residues for wheat and barley hay differed by less than 5-fold, although the data sets were not statistically similar (Mann-Whitney).

Based on the data for barley hay, the Meeting estimated a maximum residue level of 40 mg/kg for flupyradifurone in straw and fodder (dry) of cereal grains. The Meeting concluded that this maximum residue level was adequate to cover residues in hays and straws/stovers of cereals.

Based on the barley hay data, the Meeting estimated a highest residue value of 31 mg/kg (dry weight) for flupyradifurone in wheat and barley hay, and based on the wheat hay data, a median residue of 9.6 mg/kg (dry weight) was estimated. The Meeting agreed that these values could be extrapolated to oat, rye, millet and triticale hays.

Based on the wheat straw data, the Meeting estimated median and highest residues of 6.3 and 23 mg/kg (dry weight) for flupyradifurone in wheat and barley straw, and maize and sorghum stover. The Meeting agreed that these values could be extrapolated to oat, rye, millet and triticale straws.

Cotton gin by-products

Residue trials were conducted in cotton in the USA according to the critical foliar GAP in the USA for cotton (two foliar applications at 205 g ai/ha, 10-day RTI, 14-day PHI).

For the calculation of the livestock animal dietary burden, the ranked order of total residues of flupyradifurone in cotton gin by-products on a fresh weight basis from supervised trials according to GAP in the USA was 7.1, 8.3, 14, 19 and 21 mg/kg.

For the calculation of the livestock animal dietary burden, the ranked order of total residues of flupyradifurone in cotton gin by-products on a dry weight basis from supervised trials according to GAP in the USA was 9.6, 10, 15, 21 and 28 mg/kg (HR 29 mg/kg).

The Meeting estimated a median and highest residue of 15 and 29 mg/kg (dry weight) respectively for flupyradifurone in cotton gin by-products.

Fate of residues during processing

Data showed that flupyradifurone was not degraded during the simulation of pasteurisation (pH 4, 90 °C, 20 minutes), baking, boiling and brewing (pH 5, 100 °C, 60 minutes) and during sterilisation (pH 6, 120 °C, 20 minutes).

The Meeting also received processing studies for oranges, apples, grapes, cucumbers, tomatoes, soya beans, potatoes, barley, wheat, corn, cotton, peanuts, coffee and hops. The table below summarizes STMR-P values calculated on the determined processing factors (total residues). In addition, the following maximum residue levels were estimated.

Apples

Based on the flupyradifurone (parent only) processing factor of 2.0 for dried apples and the pome fruit MRL of 0.9 mg/kg, the calculated expected highest residues in dried apples are 1.8 mg/kg. The Meeting estimated an MRL for flupyradifurone in apples, dried of 2 mg/kg.

Grape

Based on the flupyradifurone (parent only) processing factor of 2.5 for raisin (mean of 2.1 and 2.9) and the grape MRL of 3 mg/kg, the calculated expected highest residues in raisins are 7.5 mg/kg. The Meeting estimated an MRL for flupyradifurone in dried grapes of 8 mg/kg.

Maize

Based on the flupyradifurone (parent only) processing factor of 2.8 for maize bran (mean of 1.8 and 3.8) and the maize MRL of 0.015 mg/kg, the calculated expected highest residues in maize bran are 0.042 mg/kg. The Meeting estimated an MRL for flupyradifurone in maize bran of 0.05 mg/kg.

Wheat

Based on the flupyradifurone (parent only) processing factor of 2.35 for wheat bran (mean of 2.3 and 2.4) and the cereal grain MRL of 3 mg/kg, the calculated expected highest residues in wheat bran are 7.05 mg/kg. The Meeting estimated an MRL for flupyradifurone in wheat bran of 8 mg/kg.

Based on the flupyradifurone (parent only) processing factor of 1.45 for wheat germ (median of 0.79, 1.3, 1.6 and 1.7) and the cereal grain MRL of 3 mg/kg, the calculated expected highest residues in wheat germ are 4.35 mg/kg. The Meeting estimated an MRL for flupyradifurone in wheat germ of 5 mg/kg.

Based on the flupyradifurone (parent only) processing factor of 1.4 for wheat whole meal (median of 1.1, 1.3, 1.5 and 1.6) and the cereal grain MRL of 3 mg/kg, the calculated expected highest residues in wheat whole meal are 4.2 mg/kg. The Meeting estimated an MRL for flupyradifurone in wheat whole meal of 5 mg/kg.

The processing (or transfer) factors derived from the processing studies and the resulting recommendations for STMR-Ps are summarized in the table below.

Processing Factors from the Processing of Raw Agricultural Commodities (RACs) with Field-Incurred Residues from Foliar Treatment with Flupyradifurone

RAC	Processed Commodity	Best Estimate Processing Factor (total residues)	RAC STMR	Processed Commodity STMR-P	RAC HR	Processed Commodity HR-P
Oranges	Peel, ripe unwashed	1.85	0.505	0.93	2.2	4.1
	Juice	0.135		0.068		0.30
	Oil	0.135		0.068		0.30
	Pulp	0.21		0.11		0.46
	Pomace, wet	1.3		0.66		2.9
	Pomace, dried	4.5		2.3		9.9
	Marmalade	0.155		0.078		0.34
Apple	Whole fruit, washed	1.1	0.23	0.25	0.62	0.68
	Sauce	0.80		0.18		0.50
	Pomace, dried	3.95		0.91		2.4
	Juice	0.60		0.14		0.37
	Fruit, dried	1.9		0.44		1.2
Grape	Berry	0.865	0.63	0.54	2.3	2.0
	Pomace, grape	1.75		1.1		4.0
	Must	0.70		0.44		1.6
	Juice, pasteurised	0.69		0.43		1.6
	Wine at first taste test	0.415		0.26		0.95
	Jelly	0.295		0.19		0.68
	Raisin	2.5		1.6		5.8
Tomatoes	Juice	0.67	0.71	0.48	2.79	1.9
	Puree	1.5		1.1		4.2
	Paste	1.9		1.3		5.3
	Peel	2.1		1.5		5.9
	Preserve	0.71		0.50		2.0
	Fruit, dried	2.45		1.7		6.8
Soya bean	Aspirated grain fractions	7.1	3.44	24.4		
seed	Meal	1.35		4.6		
	Hull	0.76		2.6		
	Oil, refined	0.038		0.13		
	Milk	0.21		0.72		
	Defatted flour	1.55		5.3		
Potato	Crisps	1.25	0.291	0.36	0.57	0.71
	Flakes	1.55		0.45		0.88
	Peel, wet	0.596		0.17		0.34
	Starch	0.546		0.16		0.31
	Tuber with peel, cooked	1.05		0.31		0.60
	Tuber, steamed, cooked	0.546		0.16		0.31
Barley	Malt sprouts	0.79	1.315	1.04		
	Brewer's malt	0.49		0.64		
	Brewer's grain	0.069		0.091		
	Hops draff	0.44		0.58		
	Brewer's yeast	0.13		0.17		
	Beer	0.075		0.099		
	Pearl barley	0.12		0.16		
	Pearl barley rub-off	2.93		3.85		
Wheat	White flour	0.445	1.315	0.59		
	White bread	0.32		0.42		
	Whole meal	1.25		1.64		

RAC	Processed Commodity	Best Estimate Processing Factor (total residues)	RAC STMR	Processed Commodity STMR-P	RAC HR	Processed Commodity HR-P
	Whole meal bread	0.795		1.05		
	Wheat germ	1.25		1.64		
	Aspirated grain fractions	10.5		13.8		
	Bran	1.55		2.0		
	Gluten	0.40		0.53		
	Pasta, cooked	0.135		0.18		
	Pasta, dried and cooked	0.175		0.23		
	Pasta, dry	0.645		0.85		
	Pasta, fresh	0.51		0.67		
	Shorts	0.945		1.2		
	Starch	0.026		0.034		
Corn	Aspirated grain fractions	6.6	0.49	3.2		
	Bran	1.55		0.76		
	Flour	0.89		0.44		1
	Germ, dry milling	1.035		0.51		
	Meal, dry milled	0.895		0.44		
	Oil, dry milled	0.89		0.44		
	Starch	0.89		0.44		
Cotton	Oil, refined	0.20	0.395	0.079		
	Meal	0.83		0.33		
	Hull	0.99		0.39		
Peanuts	Meal	1.2	0.225	0.27	0.35	0.42
	Oil, refined	0.56		0.13		0.20
	Peanut butter	0.75		0.17		0.26
	Peanut, roasted	0.75		0.17		0.26

Residues in animal commodities

Estimated maximum and mean dietary burdens of farm animals

Dietary burden calculations for beef cattle and dairy cattle and poultry are provided below. The dietary burdens were estimated using the OECD diets listed in Appendix IX of the 2009 edition of the FAO Manual.

Potential cattle feed items include: alfalfa forage and hay, clover forage and hay, apple pomace, grape pomace, citrus dried pulp, barley grain, forage, hay and straw, brewer's grain, barley grain, wheat grain, maize grain, millet grain, rye grain, sorghum grain, triticale grain, forage, hay and straw, wheat milled by-products, maize aspirated grain fractions, maize meal, maize grain fodder and forage, maize milled by-products, millet forage and hay, oat forage and hay, rye forage and straw, carrot culls, potato culls, potato process waste, barley bran, cabbage heads, cotton seed, cotton seed meal and hulls, cotton gin by-products, soya bean forage and fodder (hay), soya beans, soya bean meal and hulls, soya bean aspirated grain fractions, sorghum forage and stover, bean and pea seed, bean vines and pea vines and hay/ fodder, peanut hay and peanut meal.

Summary of livestock dietary burden for flupyradifurone (ppm of dry matter diet)

	US-Canada		EU		Australia		Japan	
	max	mean	max	mean	Max	Mean	max	mean
Beef cattle	22.7	7.63	71.8 [a]	16.6	71.8 [a]	23 [c]	8.18	5.38
Dairy cattle	46.0	9.36	66.6	12.1	71.8 [b]	18.0 [d]	50.25	8.2

[a] Highest maximum beef or dairy cattle dietary burden suitable for HR and MRL estimates for mammalian meat

[b] Highest maximum dairy cattle dietary burden suitable for HR and MRL estimates for mammalian milk

[c] Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian meat

[d] Highest mean dairy cattle dietary burden suitable for STMR estimates for mammalian milk

Potential poultry feed items include: apple pomace, carrot culls, cabbage heads, barley grain, forage, hay and straw, brewer's grain, wheat grain, forage, hay and straw, wheat milled by-products, maize grain fodder and forage, maize meal, maize milled by-products, potato culls and dried pulp (potato process waste), barley bran, cotton seed, cotton seed meal and hulls, cotton gin by-products, soya bean forage and fodder (hay), soya beans, soya bean meal and hulls, soya bean aspirated grain fractions, bean and pea seed, bean vines and pea vines and hay/ fodder.

Summary of poultry dietary burden for flupyradifurone (ppm of dry matter diet)

	US-Canada		EU		Australia		Japan	
	max	Mean	max	mean	Max	Mean	max	mean
Poultry Broiler	2.89	2.89	3.43	3.41	3.905	3.905	5.21	3.67
Poultry Layer	2.89	2.89	15.4 [a]	6.03 [b]	3.905	3.905	2.42	2.42

[a] Highest maximum poultry dietary burden suitable for HR and MRL estimates for poultry meat and eggs

[b] Highest maximum poultry dietary burden suitable for STMR estimates for poultry meat and eggs

Farm animal dietary burden

The Meeting received a lactating dairy cow feeding study which provided information on residues of flupyradifurone arising in tissues and milk when dairy cows were dosed for 29 days, at feeding levels equivalent to 0, 4.8, 23, 50 and 135 ppm flupyradifurone in the diet. Residues of parent, DFA, fpd-OH and fpd-AMCP were determined.

Total (parent + DFA) flupyradifurone residues in milk from the 135 ppm feed group reached plateau levels within two or four days of consecutive dosing. The residue in milk was primarily (60–90%) parent compound. Residues of parent + DFA did not concentrate in cream.

Residues of parent were observed in tissues (fat, kidney, liver and muscle) at every feeding level and it was the dominant residue. Total (parent + DFA) flupyradifurone residues at the 135 ppm feeding level, for example, were 1.37 mg/kg in fat, 5.28 mg/kg in kidney, 3.85 mg/kg in liver and 1.89 mg/kg in muscle with 71, 89, 89 and 80% of these residues respectively being parent.

The Meeting also received information on residues arising in tissues and eggs when laying hens were dosed with flupyradifurone for 28 days, at feeding levels equivalent to 0, 1.5, 6.5, 19.4 and 65.1 ppm in the diet. Residues of parent, DFA, fpd-OH and fpd-AMCP were determined.

Residues of all four analytes were present at the highest feeding level in eggs. DFA was the dominant residue. No quantifiable residues of parent were observed at the lower feeding levels (1.5 and 6.5 ppm). At the next highest feeding level residues of parent ranged from 0.01–0.026 mg/kg, while residues of DFA were approximately 0.45–0.56 mg/kg.

DFA was also the dominant residue in tissues (fat, liver and muscle). Total (parent + DFA) flupyradifurone residues at the 65.1 ppm feeding level, for example, were 1.20 mg/kg in fat, 3.35 mg/kg in liver and 2.31 mg/kg in muscle with 84, 99 and 98% of these residues respectively being DFA.

Animal commodity maximum residue levels

Cattle—STMR, HR and MRLs

For highest residue level estimation, the high residues in the cattle tissues were calculated by interpolating the maximum dietary burden for beef cattle (71.8 ppm) between the relevant feeding levels (49.6 and 135 ppm) in the dairy cow feeding study and using the highest tissue concentrations from individual animals within those feeding groups. For highest residue level estimation, the high residues in the cattle milk were calculated by interpolating the maximum dietary burden for dairy cattle (71.8 ppm) between the relevant feeding levels (49.6 and 135 ppm) in the dairy cow feeding study and using the highest mean milk concentrations from those feeding groups.

The STMR values for the tissues were taken from the 23.1 ppm feeding level from the dairy cow feeding study and using the mean tissue concentrations from that feeding group as the mean dietary burden for beef cattle was almost the same (23 ppm). The STMR values for the milk were calculated by interpolating the mean dietary burden for dairy cattle (18.0 ppm) with the 4.8 and 23.1 ppm feeding levels from the dairy cow feeding study and using the mean milk concentrations from those feeding groups.

Flupyradifurone Feeding Study	Feed Level (ppm) for milk residues	Residues (mg/kg) in milk	Feed Level (ppm) for tissue residues	Residues (mg/kg)			
				Muscle	Liver	Kidney	Fat
HR Determination (beef or dairy cattle)							
Feeding Study	49.6	0.308	49.6	0.910	2.17	2.37	0.489
	135	0.974	135	2.28	4.40	6.35	1.93
Dietary burden and estimate of highest residue	71.8	0.481	71.8	1.27	2.75	3.40	0.864
STMR Determination (beef or dairy cattle)							
Feeding Study	4.81	0.043	23.1	0.304	0.812	0.867	0.147
	23.1	0.129					
Dietary burden and estimate of highest residue	18.0	0.105	23	0.304	0.812	0.867	0.147

The Meeting estimated the following STMR values: milk 0.11 mg/kg; muscle 0.30 mg/kg; edible offal (based on kidney) 0.87 mg/kg and fat 0.15 mg/kg.

The Meeting estimated the following HR values: milk 0.48 mg/kg; muscle 1.27 mg/kg; edible offal (based on kidney) 3.40 mg/kg and fat 0.86 mg/kg.

The Meeting estimated the following maximum residue levels: milk 0.7 mg/kg; meat (mammalian except marine mammals) 1.5 mg/kg, edible offal (based on kidney) 4 mg/kg and mammalian fats (except milk fats) 1 mg/kg.

Poultry—STMR, HR and MRLs

For highest residue level estimation, the high residues in the hen tissues and eggs were calculated by interpolating the maximum dietary burden (15.4 ppm) with the 6.5 and 19.4 ppm feeding levels in the laying hen feeding study and using the highest tissue concentrations from individual animals within that feeding group and using the highest mean egg concentration from those feeding groups.

The STMR values for the tissues and eggs were calculated by extrapolating the mean dietary burden (6.03 ppm) with the 1.5 and 6.5 ppm feeding levels from the poultry feeding study and using the mean tissue and egg concentrations from those feeding groups.

Flupyradifurone Feeding Study	Feed Level (ppm) for egg residues	Residues (mg/kg) in egg	Feed Level (ppm) for tissue residues	Residues (mg/kg)		
				Muscle	Liver	Fat
HR Determination (poultry broiler or layer)						
Feeding Study	6.5 19.4	0.169 0.532	6.5 19.4	0.307 0.783	0.427 1.08	0.127 0.290
Dietary burden and estimate of highest residue	15.4	0.42	15.4	0.64	0.88	0.24
STMR Determination (poultry broiler or layer)						
Feeding Study	1.5 6.5	0.053 0.164	1.5 6.5	0.0868 0.294	0.107 0.419	0.0319 0.120

Flupyradifurone Feeding Study	Feed Level (ppm) for egg residues	Residues (mg/kg) in egg	Feed Level (ppm) for tissue residues	Residues (mg/kg)		
				Muscle	Liver	Fat
Dietary burden and estimate of highest residue	6.03	0.15	6.03	0.27	0.39	0.11

The Meeting estimated the following STMR values: egg 0.15 mg/kg; muscle 0.27 mg/kg; edible offal (based on liver) 0.39 mg/kg and fat 0.11 mg/kg.

The Meeting estimated the following HR values: egg 0.42 mg/kg; muscle 0.64 mg/kg; edible offal (based on liver) 0.88 mg/kg and fat 0.24 mg/kg.

The Meeting estimated the following maximum residue levels: eggs 0.7 mg/kg; poultry meat 0.8 mg/kg, poultry edible offal (based on liver) 1 mg/kg and poultry fats 0.3 mg/kg.

RECOMMENDATIONS

Definition of the residue (for compliance with the MRL for plant commodities): *Flupyradifurone.*

Definition of the residue (for estimation of dietary intake for plant commodities): Sum of flupyradifurone, difluoroacetic acid (DFA) and 6-chloropyridine-3-carboxylic acid (6-CNA), expressed as parent equivalents.

Definition of the residue (for compliance with the MRL and for estimation of dietary intake for animal commodities): Sum of flupyradifurone and difluoroacetic acid (DFA), expressed as parent equivalents.

The residue is not fat soluble.

The Meeting estimated the maximum residue levels and STMR values shown below.

Commodity		MRL, mg/kg	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name			
	Alfalfa hay (dry weight)	30	14	42
DF 0226	Apples, dried	2	0.44	1.2
VD 0071	Beans, dry	0.4	3.22	
VP 0062	Beans, shelled (succulent = immature seeds)	0.2	1.17	2.77
VP 0061	Beans, except broad bean and soya bean (green pods and immature seeds)	1.5	2.63	5.1
	Bean hay (dry weight)	30	5.7	17
VA 0036	Bulb vegetables, except Fennel, Bulb (RC only)	*0.01	0.18	0.39
FB 2006	Bush berries	4	0.725	2.6
VB 0041	Cabbage, head	1.5	0.79	1.71
VB 0404	Cauliflower	6	0.48	3.01
VS 0624	Celery	9 [a]	2.38	7.19
GC 0080	Cereal grains (except maize and rice)	3	1.315	
SO 0691	Cotton seed	0.8	0.395	
VC 0424	Cucumber	0.4		
DF 0269	Dried grapes	8	1.6	5.8
MO 0105	Edible offal (mammalian)	4	Kidney 0.87 Liver 0.81	Kidney 3.40 Liver 2.75
PE 0112	Eggs	0.7	0.15	0.42
FB 0269	Grapes	3	0.63	2.3
FC 0002	Lemons and limes (including citron)	1.5	0.32	0.73
VL 0482	Lettuce, head	4	1.3	2.4

Commodity		MRL, mg/kg	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name			
VL 0483	Lettuce, leaf	15[a]	2.6	8.0
FC 0003	Mandarins	1.5	0.44	0.99
MF 0100	Mammalian fats (except milk fats)	1	0.15	0.86
MM 0095	Meat (from mammals other than marine mammals)	1.5	Muscle 0.30 Fat 0.15	Muscle 1.27 Fat 0.86
GC 0645	Maize	0.015	0.49	
	Maize bran	0.05	0.76	
VC 0046	Melons, except watermelon	0.4	0.57	1.07
ML 0106	Milks	0.7	0.11	0.48
VL 0485	Mustard greens	40[a]	12	25
FC 0004	Oranges, Sweet, Sour	4	0.505	2.2
SO 0697	Peanut	0.04	0.225	0.35
	Peanut hay (dry weight)	30	11	20
VD 0072	Peas (dry)	3	3.605	
	Pea hay (dry weight)	50	19.5	36
VP 0063	Peas (pods and succulent = immature seeds)	3	2.68	5.5
VP 0064	Peas, shelled (succulent seeds)	3	2.78	5.7
TN 0672	Pecan	0.015	0.060	0.063
VO 0051	Peppers	0.9	0.68	2.39
HS 0444	Peppers Chilli, dried	9	6.8	23.9
FP 0009	Pome Fruit	0.9	0.45	0.69
VR 0589	Potato	0.05	0.291	0.57
PF 0111	Poultry fats	0.3	0.11	0.24
PM 0110	Poultry meat	0.8	Muscle 0.27 Fat 0.11	Muscle 0.64 Fat 0.24
PO 0111	Poultry, edible offal of	1	0.39	0.88
FC 0005	Pomelo and Grapefruits	0.7	0.21	0.32
VR 0075	Root and tuber vegetables (except potato)	0.7	0.29	1.37
VD 0541	Soya bean (dry)	1.5	3.44	
	Soya bean hay (dry weight)	40	15.5	41
VL 0502	Spinach	30[a]	8.5	19
AS 0081	Straw and fodder, dry of cereal grains (dry weight)	40	9.6 (hay) 6.3 (straw and stover)	31 (hay) 23 (straw and stover)
FB 0275	Strawberry	1.5	1.505	2.74
VC 0431	Squash, summer	0.2	0.655	2.19
VO 0447	Sweet corn (corn-on-the-cob)	0.05	0.56	1.59
VR 0508	Sweet Potato	0.05	0.291	0.57
VO 0448	Tomato	1	0.71	2.79
CM 0654	Wheat, bran	8	2.0	
CF 1210	Wheat, germ	5	1.64	
CF 1212	Wheat wholemeal	5	1.64	
	Alfalfa forage (dry weight)		20	51
	Apple juice		0.14	0.37
	Apple pomace, dry		0.91	
	Apple sauce		0.18	0.50
	Barley brewer’s grain		0.091	
	Barley forage (dry weight)		6.9	77
	Bean forage (dry weight)		12.5	21
	Beer		0.099	
VD 0523	Broad bean (dry)		2.49	
VD 0524	Chick pea (dry)		2.49	
JF 0001	Citrus juice		0.068	0.30
	Cotton gin by-products		15	29
	Cotton hulls		0.39	
	Cotton meal		0.33	

Commodity		MRL, mg/kg	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name			
	Cotton seed oil		0.079	
JF 0269	Grape juice		0.43	1.6
	Grape must		0.44	
	Grape pomace		1.1	4.0
VD 0533	Lentil (dry)		2.49	
VD 0545	Lupin (dry)		2.49	
	Maize aspirated grain fractions		3.2	
	Maize flour		0.44	
	Maize forage (dry weight)		6.9	77
	Maize germ		0.51	
	Maize meal		0.44	
	Maize oil		0.44	
	Maize starch		0.44	
	Millet forage (dry weight)		6.9	77
AF 0647	Oat forage (green) (dry weight)		6.9	77
	Orange marmalade		0.078	0.34
	Orange oil		0.068	0.30
	Pea forage (dry weight)		22.5	44
	Pearled barley		0.16	
	Peanut butter		0.17	0.26
	Peanut meal		0.27	0.42
	Peanuts, roasted		0.17	0.26
	Peanut oil, refined		0.13	0.20
VD 0537	Pigeon pea (dry)		2.49	
	Potato chips		0.36	0.71
	Potato flakes		0.45	0.88
	Potato starch		0.16	0.31
	Potato wet peel		0.17	0.34
	Rye forage (dry weight)		6.9	77
	Sorghum forage (dry weight)		6.9	77
	Soya bean aspirated grain fractions		24.4	
	Soya bean forage (dry weight)		23	46
	Soya bean hulls		2.6	
	Soya bean meal		4.6	
	Soya bean milk		0.72	
	Soy flour		5.3	
OR 0541	Soy oil, refined		0.13	
JF 0048	Tomato juice		0.48	1.9
VW 0448	Tomato paste		1.3	5.3
	Tomato puree		1.1	4.2
	Triticale forage (dry weight)		6.9	77
	Wheat aspirated grain fractions		13.8	
CF 1211	Wheat flour		0.59	
	Wheat forage (dry weight)		6.9	77
	Wheat gluten		0.53	
	Wheat starch		0.034	
	Wheat white bread		0.42	
	Wheat whole meal bread		1.05	
	Wine		0.26	0.95

[a] The information provided to the JMPR precludes an estimate that the dietary intake for celery, leaf lettuce, spinach and mustard greens would be below the ARfD

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) for flupyradifurone were calculated for the 17 GEMS/ Food cluster diets, based on STMRs and STMRPs estimated by the current Meeting. The results are shown in Annex 3 to the 2016 Report.

The ADI is 0–0.08 mg/kg bw and the estimated IEDIs were 6–20% of the maximum ADI of 0.08 mg/kg bw. The Meeting concluded that the long-term intake of residues of flupyradifurone resulting from the uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The 2015 JMPR established an ARfD of 0.2 mg/kg bw. The International Estimated Short-Term Intakes (IESTIs) for flupyradifurone were calculated for the food commodities for which STMRs and HRs were estimated and for which consumption data were available. The results are shown in Annex 4 to the 2016 Report.

For celery the IESTI represented 120% of the ARfD for children. For leaf lettuce the IESTI represented 250% of the ARfD for children. For mustard greens the IESTI represented 250% of the ARfD for the general population and 610% for children. For spinach the IESTI represented 130% of the ARfD for the general population and 420% for children. No alternative GAP for celery, leaf lettuce, mustard greens and spinach was available. On the basis of the information provided to the JMPR, the Meeting concluded that the short-term intake of flupyradifurone from the consumption of celery, leaf lettuce, mustard greens and spinach may present a public health concern.

Estimates of intakes for the other commodities considered by the 2016 JMPR varied from 0–90% of the ARfD (0.2 mg/kg bw). The Meeting concluded that apart from celery, leaf lettuce, mustard greens and spinach, the short-term intake of residues of flupyradifurone, from uses considered by the current Meeting, is unlikely to present a public health concern.

REFERENCES

Code	Authors	Year	Title, Institute, Report reference
M-481362-01-1	Anon.	2013	BYI 02960; SL 200; coffee; Brazil; BBA Bayer CropScience Bayer CropScience, Report No.: M-481362-01-1, Date: 2013-07-30 unpublished
M-435020-02-1	Anon.	2014	BYI 02960 480 FS Bayer CropScience LP, RTP, NC, USA Bayer CropScience, Report No.: M-435020-02-1, Date: 2014-12-01 unpublished
RARVY012	Beedle, E & Niczyporowicz, LM	2012	BYI 02960 200 SL—Magnitude of the residue in/on citrus (crop group 10) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY012, Date: 2012-06-27 unpublished
RARVY023	Beedle, E	2012	BYI 02960 200 SL—Magnitude of the residue in head and stem Brassica (crop subgroup 5A) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY023, Date: 2012-05-15 unpublished
RARVY021	Beedle, EC & Harbin, AM	2012	BYI 02960 200 SL—Magnitude of the residue in/on cucurbit vegetables (crop group 9) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY021, Date: 2012-06-15 unpublished
AF09/042	Bogdoll, B & Eyrich, U	2011	Flupyradifurone (BYI 02960): Calculation of the Henry´s law constant Bayer CropScience, Report No.: AF09/042, Date: 2011-08-15 unpublished
PA09/006	Bogdoll, B & Strunk, B	2011a	Flupyradifurone (BYI 02960), pure substance: Relative density Bayer CropScience AG, Report No.: PA09/006, Date: 2011-08-18 Unpublished

Code	Authors	Year	Title, Institute, Report reference
PA09/008	Bogdoll, B & Strunk, B	2011b	Flupyradifurone (BYI 02960), pure substance—Physical characteristics colour, physical state and odour Bayer CropScience, Report No.: PA09/008, Date: 2011-08-18 unpublished
PA09/004	Bogdoll, B & Strunk, B	2011c	Flupyradifurone (BYI 02960), pure substance: Partition coefficient 1-octanol / water at pH 4, pH 7 and pH 9 (HPLC-method) Bayer CropScience AG, Report No.: PA09/004, Date: 2011-09-26 unpublished
MEF-11/269	Bongartz, R & Koester, J	2011a	[Pyridinylmethyl-^{14}C]BYI 02960: Metabolism in the lactating goat Bayer CropScience, Report No.: MEF-11/269, Date: 2011-12-07 unpublished
MEF11/268	Bongartz, R & Koester, J	2011b	[Furanone-4-^{14}C]BYI 02960: Metabolism in the lactating goat Bayer CropScience, Report No.: MEF11/268, Date: 2011-12-16 unpublished
MEF-11/199	Bongartz, R & Koester, J	2012a	[Pyridinylmethyl-^{14}C]BYI 02960: Metabolism in the laying hen Bayer CropScience, Report No.: MEF-11/199, Date: 2012-01-10 unpublished
MEF-11/200	Bongartz, R & Koester, J	2012b	[Furanone-4-^{14}C]BYI 02960: Metabolism in the laying hen Bayer CropScience, Report No.: MEF-11/200, Date: 2012-01-10 unpublished
MEF-10/892	Breuer-Rehm, M	2011	Metabolism of [pyridinylmethyl-^{14}C]BYI 02960 in confined rotational crops Bayer CropScience, Report No.: MEF-10/892, Date: 2011-12-02.Amended: 2012-02-22 unpublished
MR-07/337	Brumhard, B & Reineke, A	2009	Analytical method 01074 for the determination of BYI 02960 in soil using LC/MS/MS Bayer CropScience, Report No.: 01074, Edition Number: M-337752-01-2 Method Report No.: MR-07/337 Date: 2009-02-24 unpublished
EBRVP109	Bruns, E	2012	Fate of BYI 02960 (tech.) in outdoor microcosm ponds simulating actual exposure conditions in agricultural use Bayer CropScience, Report No.: EBRVP109, Date: 2012-03-20 unpublished
RARVY017	Dacus, SC & Lenz, C	2012	BYI 02960 200 SL—Magnitude of the residue in alfalfa Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY017, Date: 2012-05-15 unpublished
RARVY007	Dallstream, KA	2012a	BYI 02960 200 SL—Magnitude of the residue in/on small fruit vine climbing (except Fuzzy kiwifruit) Crop Subgroup 13-07F Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY007, Date: 2012-06-04 unpublished
RARVY006	Dallstream, KA	2012b	BYI 02960 200 SL—Magnitude of the residue in/on low growing berry (crop subgroup 13-07G)—strawberry Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY006, Date: 2012-03-16 unpublished
2301-BS120-342-11	de Souza, TJT	2012	BYI 02960—Rate of degradation of [Pyridine-2,6-^{14}C]-BYI 02960 in Brazilian soils Bioensaios Análises e Consultoria Ambiental, Viamao, Brazil Bayer CropScience, Report No.: 2301-BS120-342-11, Date: 2012-06-04 unpublished
IR-4 PR No. 10747	Dorschner, KW	2012a	BYI 02960: Magnitude of the residue on clover Rutgers, The State University of New Jersey, Princeton, NJ, USA TF- BCS-IR4, Report No.: IR-4 PR No. 10747, Date: 2012-07-15 unpublished
IR-4 PR No. 10637	Dorschner, KW	2012b	BYI 02960: Magnitude of the residue on blueberry Rutgers, The State University of New Jersey, Princeton, NJ, USA TF- BCS-IR4, Report No.: IR-4 PR No. 10637, Date: 2012-07-17 unpublished
IR-4 PR NO. 10722	Dorschner, KW	2012c	BYI 02960: Magnitude of the residue on prickly pear cactus U.S. Department of Agriculture, Salinas, CA, USATF- BCS-IR4, Report No.: IR-4 PR NO. 10722, Date: 1996-10-24 unpublished
PA11/062	Eyrich, U & Bogdoll, B	2011a	Flupyradifurone (BYI 02960), technical substance: Physical characteristics colour, physical state and odour Bayer CropScience AG, Report No.: PA11/062, Date: 2011-09-16 unpublished
PA11/063	Eyrich, U & Bogdoll, B	2011b	Flupyradifurone (BYI 02960), technical substance: Relative density Bayer CropScience, Report No.: PA11/063, Date: 2011-09-16 GLP/GEP: yes, unpublished
PA09/005	Eyrich, U & Bogdoll, B	2011c	Flupyradifurone (BYI 02960): Solubility in organic solvents Bayer CropScience AG, Report No.: PA09/005, Date: 2011-09-16 unpublished
PA11/065	Eyrich, U & Bogdoll, B	2011d	Flupyradifurone (BYI 02960), technical substance: Determination of the surface tension Bayer CropScience AG, Report No.: PA11/065, Date: 2011-09-16 unpublished
PA09/007	Eyrich, U & Bogdoll, B	2011e	Flupyradifurone (BYI 02960), pure substance: Determination of the pH-value in distilled water Bayer CropScience, Report No.: PA09/007, Date: 2011-08-09 unpublished

Code	Authors	Year	Title, Institute, Report reference
PA11/064	Eyrich, U & Bogdoll, B	2011f	Flupyradifurone (BYI 02960), technical substance: Determination of the pH-value in distilled water Bayer CropScience, Report No.: PA11/064, Date: 2011-09-16 unpublished
PA11/066	Eyrich, U & Bogdoll, B	2011g	Flupyradifurone (BYI 02960), technical substance: The oxidation or reduction properties Bayer CropScience AG, Report No.: PA11/066, Date: 2011-09-16 unpublished
01213	Fargeix, G & Rosati, D	2012	Analytical method no 01213 for the determination of residues of BYI 02960 in drinking and surface water by HPLC-MS/MS Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 01213, Date: 2012-03-29 unpublished
RARVP048	Fenn, L	2012	Independent laboratory validation of Bayer method—RV-005-W12-01 Analytical method for the determination of residues of BYI 02960 and its metabolites difluoroacetic acid, BYI 02960-succinamide, BYI 02960-azabicycclosuccinamide in water using LC/MS/MS JRF America, Audubon, PA, USA Bayer CropScience, Report No.: RARVP048, Date: 2012-06-28 unpublished
RARVY025	Fisher, DR	2012a	BYI 02960 200 SL—Magnitude of the residue in/on bulb vegetables (crop group 3) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY025, Date: 2012-06-18 unpublished
RARVY002	Fisher, DR	2012b	BYI 02960 200 SL and BYI 02960 480 FS—Magnitude of the residue in/on corn Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY002, Date: 2012-06-18 unpublished
RARVY027	Fisher, DR	2012c	BYI 02960 200 SL—Magnitude of the residue in/on succulent, shelled pea and bean (crop subgroup 6B) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY027, Date: 2012-06-18 unpublished
RARVY003	Fischer, DR & Niczyporowicz, LM	2012	BYI 02960 200 SL and BYI 02960 480 FS—Magnitude of the residue in/on wheat Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY003, Date: 2012-06-27 unpublished
MERVP042-1	Hall, LR	2011a	Phototransformation of [^{14}C]BYI 02960 in Aqueous pH 7 Buffer—Amended Report Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: MERVP042-1, Date: 2011-11-28. Amended: 2012-03-05 unpublished
MERVP020	Hall, LR	2011b	Phototransformation of [^{14}C]BYI 02960 in natural water Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: MERVP020, Date: 2011-08-16 unpublished
MERVP055	Harbin, AM	2012	Terrestrial field dissipation of BYI 02960 200 SL in three Canadian soils Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: MERVP055, Date: 2012-06-07 unpublished
MEF-11/554	Heinemann, O	2011a	BYI 02960: Determination of the Quantum Yield and Assessment of the Environmental Half-life of the Direct Photo-Degradation in Water Bayer CropScience, Report No.: MEF-11/554, Date: 2011-09-26 unpublished
09-2702	Heinemann, O	2011b	Determination of the residues of BYI 02960 in/on soil after spraying of BYI 02960 SL 200 in the field in Germany, Italy, Spain and the United Kingdom Bayer CropScience, Report No.: 09-2702, Date: 2011-09-13 unpublished
MEF-10/896	Hellpointner; E	2010	BYI 02960: Calculation of the Chemical Half-Life in the Troposphere Bayer CropScience AG, Report No.: MEF-10/896, Date: 2010-12-27 unpublished
M-422371-01-1	Hellpointner, E & Unold, M	2012a	[1-^{14}C]BYI 02960-DFA (BCS-AB60481): Aerobic aquatic degradation Bayer CropScience, Report No.: M-422371-01-1, Date: 2012-01-12 unpublished
MEF-11/907	Hellpointner, E & Unold, M	2012b	[Pyridine-2,6-^{14}C]BYI 02960: Aerobic aquatic metabolism Bayer CropScience, Report No.: MEF-11/907, Date: 2012-01-12 unpublished
RARVY028	Hoag, RE, Arthur, EL & Woodard, DL	2012	BYI 02960 200 SL—Magnitude of the residue in/on dried, shelled pea and bean (except soya bean), foliage of legume vegetables (except soya bean); (CG 6C and 7A) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY028, Date: 2012-06-26 unpublished
RARVY001	Hoag, RE	2012a	BYI 02960 200 SL and BYI 02960 480 FS—Magnitude of the residue in/on barley Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY001, Date: 2012-04-19 unpublished

Code	Authors	Year	Title, Institute, Report reference
RARVP074	Hoag, RE	2012b	BYI 02960 200 SL—Magnitude of the residue in/on coffee; U.S., Canada and E.U. import tolerance Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP074, Date: 2012-06-27 unpublished
RARVP075	Hoag, RE	2012cc	BYI 02960 200 SL—Magnitude of the residue in/on processed commodities for coffee; U.S., Canada and E.U. import tolerance Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP075, Date: 2012-06-26 unpublished
MEF-11/016	Justus, K	2011a	Metabolism of [furanone-4-^{14}C]BYI 02960 in tomatoes Bayer CropScience, Report No.: MEF-11/016, Date: 2011-07-25 unpublished
MEF-11/182	Justus, K	2011b	Metabolism of [pyridinylmethyl-^{14}C]BYI 02960 in tomatoes Bayer CropScience, Report No.: MEF-11/182, Date: 2011-07-26 unpublished
MEF-10/769	Justus, K	2011c	Metabolism of [furanone-4-^{14}C]BYI 02960 in potatoes Bayer CropScience, Report No.: MEF-10/769, Date: 2011-09-14 unpublished
MEF-10/710	Justus, K	2011d	Metabolism of [pyridinylmethyl-^{14}C]BYI 02960 in potatoes Bayer CropScience, Report No.: MEF-10/710, Date: 2011-09-14 unpublished
MEF-11/499	Justus, K	2011e	Metabolism of [furanone-4-^{14}C]BYI 02960 in apples Bayer CropScience, Report No.: MEF-11/499, Date: 2011-12-29 unpublished
MEF-11/198	Justus, K	2011f	Metabolism of [pyridinylmethyl-^{14}C]BYI 02960 in apples Bayer CropScience, Report No.: MEF-11/198, Date: 2011-09-09 unpublished
MEF-11/793	Justus, K	2011g	Extraction efficiency testing of the residue analytical method RV-001-P10-02 for the determination of BYI 02960, 6-chloronicotinic acid, difluoroacetic acid and difluoroethyl-amino-furanone in plant matrices using aged radioactive residues Bayer CropScience, Report No.: MEF-11/793, Date: 2011-12-01 unpublished
MEF-11/365	Klempner, A	2011	Metabolism of [furanone-4-^{14}C]BYI 02960 in confined rotational crops Bayer CropScience, Report No.: MEF-11/365, Date: 2011-12-16 unpublished
MEF-11/747	Klempner, A	2012	[Pyridinylmethyl-^{14}C]BYI 02960—Absorption, distribution, excretion, and metabolism in the rat Bayer CropScience, Report No.: MEF-11/747, Date: 2012-01-12 unpublished
MEF-11/275	Koester, J	2011a	Quantitative whole body autoradiography of [furanone-4-^{14}C]BYI 02960 in male and female rats: Distribution of total radioactivity and elimination from blood, organs and tissues after single oral administration including determination of radioactivity in the excreta and exhaled $^{14}CO_2$ Bayer CropScience, Report No.: MEF-11/275, Date: 2011-05-30 unpublished
MEF-11/270	Koester, J	2011b	[Ethyl-1-^{14}C]BYI 02960—Metabolism in organs and tissues of male and female rats (3 time-points) Bayer CropScience, Report No.: MEF-11/270, Date: 2011-09-29. Amended: 2012-02-02 unpublished
MEF-11/276	Koester, J & Weber, E	2011	Quantitative whole body autoradiography of [pyridinylmethyl-^{14}C]BYI 02960 in male and female rats: Distribution of total radioactivity and elimination from blood, organs and tissues after single oral administration including determination of radioactivity in the excreta and exhaled 14CO2 Bayer CropScience, Report No.: MEF-11/276, Date: 2011-05-30 unpublished
2011/0134/01	Konrad, S	2012a	Independent lab validation of BCS method 01330 for the determination of residues of BYI 02960 and its metabolite difluoroacetic acid in/on plant matrices by HPLC-MS/MS Currenta GmbH & Co. OHG, Leverkusen, Germany Bayer CropScience, Report No.: 2011/0134/01, Date: 2012-02-28 unpublished
2011/0164/01	Konrad, S	2012b	Independent lab validation of BCS method 01214 for the determination of residues of BYI 02960 and its metabolite difluoroacetic acid in/on animal matrices by HPLC-MS/MS Currenta GmbH & Co. OHG, Leverkusen, Germany Bayer CropScience, Report No.: 2011/0164/01, Date: 2012-02-28 unpublished
2012/0082/01	Konrad, S	2012c	Independent lab validation of BCS method 01330/M001 for the determination of residues of BYI 02960 and its metabolite difluoroacetic acid in/on plant matrices by HPLC-MS/MS Currenta GmbH & Co. OHG, Leverkusen, Germany Bayer CropScience, Report No.: 01330/M001, Date: 2012-10-15 unpublished
RARVY008	Krolski, ME	2012	BYI 02960 200 SL—Magnitude of the residue in/on hops Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY008, Date: 2012-06-12 unpublished

Code	Authors	Year	Title, Institute, Report reference
RARVY004	Krolski, ME & Dallstream, KA	2012	BYI 02960 200 SL and BYI 02960 480 FS—Magnitude of the residue in/on sorghum Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY004, Date: 2012-03-14 unpublished
RARVY010	Krolski, ME & Harbin, AH	2012a	BYI 02960 200 SL—Magnitude of the residue in peanut Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY010, Date: 2012-01-30 unpublished
RARVY015	Krolski, ME & Harbin, AM	2012b	BYI 02960 200 SL—Magnitude of the residue in potato—Tuberous and corm vegetables (Crop Subgroup 1C) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY015, Date: 2012-05-04 unpublished
RARVY013	Lam, C	2012	BYI 02960 200 SL—Magnitude of the residue in/on pome fruits (crop group 11) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY013, Date: 2012-06-18 unpublished
RARVY029	Lenz, C	2012a	BYI 02960 200 SL—Magnitude of the residue in/on soya bean processed commodities Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY029, Date: 2012-04-04 unpublished
RARVY030	Lenz, C	2012b	BYI 02960 200 SL—Magnitude of the residue in/on field corn processed commodities Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY030, Date: 2012-02-09 unpublished
RARVY032	Lenz, C	2012c	BYI 02960 200 SL—Magnitude of the residue in/on peanut processed commodities Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY032, Date: 2012-05-04 unpublished
RARVY035	Lenz, C	2012d	BYI 02960 200 SL—Magnitude of the residue in/on orange processed commodities Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY035, Date: 2012-06-01 unpublished
RARVY038	Lenz, C	2012e	BYI 02960 200 SL—Magnitude of the residue in/on potato processed commodities Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY038, Date: 2012-05-07 unpublished
RARVY033	Lenz, C & Beedle, E	2012	BYI 02960 200 SL—Magnitude of the residue in cotton processed commodities (crop subgroup 20C) Bayer Corporation, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY033, Date: 2012-06-22 unpublished
RARVY031	Lenz, C & Fischer, DR	2012	BYI 02960 200 SL—Magnitude of the residue in/on wheat processed commodities Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY031, Date: 2012-03-13 unpublished
MERVY001	Lenz, M	2012a	Terrestrial field dissipation of BYI 02960 in California soil Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: MERVY001, Date: 2012-05-30 unpublished
MERVY002	Lenz, M	2012b	Terrestrial field dissipation of BYI 02960 in Florida soil Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: MERVY002, Date: 2012-06-08 unpublished
MERVP028	Lenz, M	2012c	Terrestrial field dissipation of BYI 02960 in Idaho soil Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: MERVP028, Date: 2012-05-31 unpublished
RARVP013	Li, Y & Schöning, R	2011	Amendment No. 1—Validation of Bayer CropScience method RV-001-P10-02—An analytical method for the determination of residues of BYI 02960, 6-chloronicotinic acid, difluoroacetic acid, and difluoroethyl-amino-furanone in plant matrices using LC/MS/MS Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP013, Date: 2011-10-12...Amended: 2012-01-11 unpublished
11257	Lowden, P, Oddy, AM & Jones, MK	1997	NI-25 Rate of Degradation of the Acid Metabolite, [^{14}C]-IC-O in Three Soils Rhône-Poulenc Agriculture Ltd., Ongar, Essex, United Kingdom Bayer CropScience, Report No.: 11257, Date: 1997-08-14 unpublished
MEF-07/334	Menke, U	2011	[Pyridinylmethyl-^{14}C]BYI 02960: Aerobic soil Metabolism/Degradation and Time-dependent Sorption in Soils, Bayer CropScience AG, Report No.: MEF-07/334, Date: 2011-08-05 unpublished
MEF-10/351	Menke, U & Unold, M	2011a	[Pyridinylmethyl-^{14}C]BYI 02960 and [Furanone-4-^{14}C]BYI 02960: Phototransformation on Soil Bayer CropScience AG, Report No.: MEF-10/351, Date: 2011-03-24 unpublished
MEF-10/804	Menke, U & Unold, M	2011b	[Furanone-4-^{14}C]BYI 02960: Aerobic Soil Metabolism/Degradation Bayer CropScience AG, Report No.: MEF-10/804, Date: 2011-07-28 unpublished

Code	Authors	Year	Title, Institute, Report reference
MEF-10/858	Menke, U & Unold, M	2011c	[Ethyl-1-^{14}C]BYI 02960: Aerobic Soil Metabolism Bayer CropScience AG, Report No.: MEF-10/858, Date: 2011-09-08 unpublished
MEF-10/880	Menke, U.; Unold, M.	2011d	[Pyridine-2,6-^{14}C]BYI 02960: Aerobic soil metabolism Bayer CropScience AG, Report No.: MEF-10/880, Date: 2011-07-28 unpublished
MEF-11/514	Menke, U & Unold, M	2012	[Furanone-4-^{14}C] and [Ethyl-1-^{14}C] and [Pyridine-2,6-^{14}C]BYI 02960: Aerobic Soil Metabolism, Bayer CropScience AG, Report No.: MEF-11/514, Date: 2012-01-03 unpublished
RARVY022	Miller, A & Helfrich, KK	2012	BYI 02960 200 SL—Magnitude of the residue in/on fruiting vegetables (CG 8); US / Canada import tolerance Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY022, Date: 2012-06-22 unpublished
MERVP019	Mislankar, S & Woodard, D	2011	BYI-02960: Hydrolytic Degradation Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: MERVP019, Date: 2011-01-07 unpublished
MERVP094	Mislankar, SG & Woodard, D	2012	[Pyridine-2,6^{14}C]BYI 02960: Anaerobic Soil Metabolism, Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: MERVP094, Date: 2012-01-10 unpublished
RARVP050-1	Moore, SM & Harbin, AM	2012	BYI 02960—Magnitude of the residue in dairy cows—Amended report Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP050-1, Date: 2012-04-03. Amended: 2012-05-31 unpublished
RARVY039	Murphy, IM	2012	BYI 02960 200 SL—Magnitude of the residue on root vegetables except sugar beets (CG 1B) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY039, Date: 2012-06-14 unpublished
RV-002-S10-01	Netzband, D	2010	An analytical method for the determination of residues of BYI 02960, 6-chloronicotinic acid (6-CNA) and difluoroacetic acid (DFA) in soil and sediment using LC/MS/MS Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RV-002-S10-01, Date: 2010-10-21 unpublished
RV-002-S10-02	Netzband, D	2011	Bayer method RV-002-S10-02—An analytical method for the determination of residues of BYI 02960, 6-chloronicotinic acid (6-CNA) and difluoroacetic acid (DFA) in soil and sediment using LC/MS/MS Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RV-002-S10-02, Date: 2011-12-06 unpublished
RV-005-W12-01	Netzband, D	2012	Bayer method RV-005-W12-01—Analytical method for the determination of residues of BYI 02960 and its metabolites Difluoroacetic acid, BYI 2960-Succinamide and BYI 2960-Azabicyclosuccinamide in water using LC/MS/MS Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RV-005-W12-01, Date: 2012-05-04 unpublished
RARVP010	Netzband, DJ	2012	In house laboratory validation of an analytical method for the determination of residues of BYI 02960 and its metabolites difluoroacetic acid, BYI 02960-succinamide and BYI 02960-azabicyclosuccinamide in water using LC/MS/MS Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP010, Date: 2012-06-25 unpublished
RARVY042	Netzband, D & Helfrich, KK	2012	BYI 02960 200 SL—Magnitude of the residue in leafy Brassica greens (Crop Subgroup 5B) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY042, Date: 2012-06-25 unpublished
RARVP070	Netzband, DJ & Stoughton, SM	2012	Stability of BYI 02960 and its metabolites 6-chloronicotinic acid (6-CNA) and difluoroacetic acid (DFA) in soil during frozen storage, 2010 (reported through 381 days of storage) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP070, Date: 2012-04-03 unpublished
RARVP008	Netzband, DJ & Stoughton, SM	2012	In house laboratory validation of an analytical method for the determination of residues of BYI 02960 and its metabolites 6-chloronicotinic acid (6-CNA) and difluoroacetic acid (DFA) in soil and sediment using LC/MS/MS Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP008, Date: 2012-04-03 unpublished
RARVY005	Netzband, D & Niczyporowicz, LM	2012	BYI 02960 SL 200—Magnitude of the residue in/on leafy vegetables (Crop Group 4) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY005, Date: 2012-06-27 unpublished

Code	Authors	Year	Title, Institute, Report reference
RARVP046-1	Netzband, D, Timberlake, BC & Harbin, AM	2012	Storage stability of BYI 02960, difluoroacetic acid, and difluoroethyl-amino-furanone in plant matrices (18-month data) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP046-1, Date: 2012-04-03. Amended: 2012-11-01 unpublished
RARVY016	Niczyporowicz, LM & Netzband, D	2012	BYI 02960 200 SL—Magnitude of the residue in/on tree nuts (Crop Group 14) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY016, Date: 2012-06-27 unpublished
10-3406	Noss, G, Bauer, J & Ruhl, S	2011	Determination of the residues of BYI 02960 in/on grape and the processed fractions (pomace, grape; must; wine at bottling; wine at first taste test, juice, pasteurised; jelly; washings; raisin waste; raisin) after spraying of BYI 02960 SL 200 in the field in Germany Bayer CropScience, Report No.: 10-3406, Date: 2011-09-07 unpublished
RARVP049	Pennell, MF	2012	Independent laboratory validation of Bayer method RV-002-S10-02 for the determination of BYI 02960 and its two acid metabolites in soil and sediment EN-CAS Analytical Laboratories, Winston-Salem, NC, USA Bayer CropScience, Report No.: RARVP049, Date: 2012-05-16 unpublished
15-600-2439	Peters; S	2009	Spectral data set of BYI 02960 ai.—Reference material Bayer CropScience, Report No.: 15-600-2439, Date: 2009-04-07 unpublished
B 015/2011	Petrovic, P	2011	Flupyradifurone (BYI 02960), technical substance: Complex formation ability in water Allessa Chemie GmbH, Frankfurt am Main, Germany Bayer CropScience, Report No.: B 015/2011, Date: 2011-09-21 unpublished
M-427469-03-2	Resende, G	2012	Amendment 01 to the final report—Determination of residues of BYI 02960 and its metabolites, in coffee after drench application at the base of the plants, followed by foliar application of BYI 02960 (200 SL) in field trials in Brazil Departamento de Registro Bayer CropScience, São Paulo, Brazil Bayer CropScience, Report No.: M-427469-03-2, Date: 2012-03-09. Amended: 2012-07-02 unpublished
MR-10/174	Rosati, D	2012	Analytical method no 01212 for the determination of residues of BYI 02960 and its metabolites BCS-AA56716 (DFA), AE F161089 (6CNA) and BCS-CC98193 (furanone) in/on plant material by HPLC-MS/MS Bayer S.A.S., Bayer CropScience, Lyon, France Bayer CropScience, Report No.: 01212, Date: 2012-03-27 unpublished
MERVP037-2	Ripperger, RJ	2011	[Furanone-4-^{14}C]BYI 02960: Aerobic Soil Metabolism in Two US Soils Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: MERVP037-2, Date: 2011-04-14...Amended: 2012-01-05 unpublished
2011/0073/01	Schloesser, G & Mans, HC	2011	BYI 02960—Determination of metallic impurities Currenta GmbH & Co. OHG, Leverkusen, Germany Bayer CropScience, Report No.: 2011/0073/01, Date: 2011-08-10 unpublished confidential
MEF-11/392	Schmeling, S & Weber, E	2011a	Metabolism of [furanone-4-^{14}C]BYI 02960 in cotton after spray application Bayer CropScience, Report No.: MEF-11/392, Date: 2011-12-12 unpublished
MEF-11/393	Schmeling, S & Weber, E	2011b	Metabolism of [pyridinylmethyl-^{14}C]BYI 02960 in cotton after spray application Bayer CropScience, Report No.: MEF-11/393, Date: 2011-12-12, unpublished
MEF-11/058	Schmeling, S & Weber, E	2011c	Metabolism of [furanone-4-^{14}C]BYI 02960 in paddy rice Bayer CropScience, Report No.: MEF-11/058, Date: 2011-09-09 unpublished
MEF-11/059	Schmeling, S & Weber, E	2011d	Metabolism of [pyridinylmethyl-^{14}C]BYI 02960 in paddy rice Bayer CropScience, Report No.: MEF-11/059, Date: 2011-09-09 unpublished
10-2503	Schoening, R & Bauer, J	2012	Determination of the residues of BYI 02960 in/on the field rotational crops barley, carrot, lettuce and turnip after spray application of BYI 02960 SL 200 on lettuce and soil in the field in Germany, the Netherlands, France (South) and Spain Bayer CropScience, Report No.: 10-2503, Date: 2012-04-11 unpublished
MR-11/050	Schoening, R & Ruhl, S	2012	Determination of residues of difluoroacetic acid in extracts of samples from plant metabolism and confined rotational crops studies after application of BYI 02960 Bayer CropScience, Report No.: MR-11/050, Date: 2012-01-17 unpublished
11-2550	Schulte, G	2012a	Determination of the residues of BYI 02960 in/on the field rotational crop potato after spray application of BYI 02960 SL 200 to bare soil in the field in northern France, the Netherlands, Spain and Italy—Limited rotational crop study Bayer CropScience, Report No.: 11-2550, Date: 2012-09-10 unpublished

Code	Authors	Year	Title, Institute, Report reference
11-2551	Schulte, G	2012b	Determination of the residues of BYI 02960 in/on the field rotational crop leek after spray application of BYI 02960 SL 200 to bare soil in the field in northern France, Germany, Spain and Italy—Limited rotational crop study Bayer CropScience, Report No.: 11-2551, Date: 2012-09-13 unpublished
11-2552	Schulte, G	2012c	Determination of the residues of BYI 02960 in/on the field rotational crop cucumber after spray application of BYI 02960 SL 200 to bare soil in the field in northern France, Germany, Spain and Italy—Limited rotational crop study Bayer CropScience, Report No.: 11-2552, Edition Number: M-438343-01-1 Date: 2012-09-13 unpublished
11-2553	Schulte, G	2012d	Determination of the residues of BYI 02960 in/on the field rotational crop onion after spray application of BYI 02960 SL 200 on bare soil in the field in France (North), Germany, Italy and Spain—Limited rotational crop study Bayer CropScience, Report No.: 11-2553, Date: 2012-09-14 unpublished
11-2555	Schulte, G	2012e	Determination of the residues of BYI 02960 in/on the field rotational crop French bean after spray application of BYI 02960 SL 200 to bare soil in the field in northern France, Germany, Italy and Spain—Limited rotational crop study Bayer CropScience, Report No.: 11-2555, Date: 2012-09-14 unpublished
11-2556	Schulte, G	2012f	Determination of the residues of BYI 02960 in/on the field rotational crop pea after spray application of BYI 02960 SL 200 on bare soil in the field in France (North), Germany, Italy and Spain—Limited rotational crop study Bayer CropScience, Report No.: 11-2556, Date: 2012-09-17 unpublished
11-2554	Schulte, G	2012g	Determination of the residues of BYI 02960 in/on the field rotational crop rape after spray application of BYI 02960 SL 200 on bare soil in the field in northern France, Germany, Italy and Spain Bayer CropScience, Report No.: 11-2554, Date: 2012-12-07 unpublished
10-3172	Schulte, G & Ballmann, C	2012a	Determination of the residues of BYI 02960 in/on apple and the processed fractions (whole fruit, washed; washings; raw sauce; strain rest; sauce; pomace, wet; pomace, dried; raw juice; retentate; and juice) after spray application of BYI 02960 SL 200 in the field in Italy and Spain Bayer CropScience, Report No.: 10-3172, Edition Number: M-434006-01-2 Date: 2012-07-05 unpublished
10-3186	Schulte, G & Ballmann, C	2012b	Amendment No. 2—Determination of the residues of BYI 02960 in/on tomato and the processed fractions (whole fruit, washed; washings; strain rest; raw juice; juice; raw puree; puree; paste; peel; peeling water; fruit peeled; preserve and tomato, dried) after spraying of BYI 02960 SL 200 in the field in southern France, Italy, Spain and Portugal Bayer CropScience, Report No.: 10-3186, Edition Number: M-427003-03-1 Date: 2012-03-08...Amended: 2012-09-28 unpublished
MR-011/096	Schulte, G & Bauer, J	2012a	Analytical method 01330 for the determination of residues of BYI 02960 and its metabolite difluoroacetic acid in/on plant matrix by HPLC-MS/MS—Enforcement method plant Bayer CropScience, Report No.: 01330, Date: 2012-02-22 unpublished
MR-011/144	Schulte, G & Bauer, J	2012b	Analytical method 01214 for the determination of residues of BYI 02960 and its metabolite difluoroacetic acid in/on animal matrices by HPLC-MS/MS—Enforcement method animal Bayer CropScience, Report No.: 01214, Date: 2012-02-22 unpublished
10-3171	Schulte, G & Bauer, J	2012c	Amendment No. 1—Determination of the residues of BYI 02960 in/on apple and the processed fractions (whole fruit, washed; washings; raw sauce; strain rest; sauce; pomace, wet; pomace, dried; raw juice; juice, retentate; peel; fruit peeled; fruit, dried) after spraying of BYI 02960 SL 200 in the field in Germany and Belgium Bayer CropScience, Report No.: 10-3171, Date: 2012-07-05. Amended: 2012-09-28 unpublished
10-3407	Schulte, G & Bauer, J	2012d	Determination of the residues of BYI 02960 in/on hop (cone, green and cone, kiln-dried) and the processed fractions (hops draff, brewer's yeast and beer) after spraying of BYI 02960 SL 200 in the field in Germany Bayer CropScience, Report No.: 10-3407, Date: 2012-02-13 unpublished

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10-3409	Schulte, G & Bauer, J	2012e	Processing study—Determination of the residues of BYI 02960 in/on wheat and the processed fractions (semolina; semolina bran; white flour bran; white flour; white bread; whole meal; wholemeal bread and wheat germ) after spraying of BYI 02960 SL 200 in the field in Germany Bayer CropScience, Report No.: 10-3409, Date: 2012-10-30 unpublished
10-3184	Schulte, G & Ruhl, S	2012a	Determination of the residues of BYI 02960 in/on cucumber and the processed fractions (washings; whole fruit, washed; preserve; brine; and fruit, fermented) after spraying of BYI 02960 SL 200 in the field in France (south)—Processing study Bayer CropScience, Report No.: 10-3184, Date: 2012-09-06 unpublished
10-3410	Schulte, G & Ruhl, S	2012b	Processing study—Determination of the residues of BYI 02960 in/on spring barley and the processed fractions (malt sprouts; brewer's malt; brewer's grain, hops draff; brewer's yeast; beer; pearl barley; pearl barley rub off) after spraying of BYI 02960 SL 200 in the field in Germany Bayer CropScience, Report No.: 10-3410, Date: 2012-10-16 unpublished
MR-12/054	Schulte, G & Teubner, L	2012a	Modification M001 of the analytical method 01330 for the determination of residues of BYI 02960 and its metabolite difluoroacetic acid in/on plant matrix by HPLC-MS/MS- Enforcement method plant Bayer CropScience, Report No.: MR-12/054, Date: 2012-09-10 unpublished
10-3405	Schulte, G & Teubner, L	2012b	Processing Study—Determination of the residues of BYI 02960 in/on orange (fruit and pulp) and the processed fractions (fruit, stored; whole fruit, washed; washings; raw juice; pomace, wet; pomace, dried; juice; marmalade; pulp; peel; peel washed; peel without oil; oil) after spraying of BYI 02960 SL 200 in the field in Spain Bayer CropScience, Report No.: 10-3405, Date: 2012-10-01 unpublished
MERVP038-1	Shepherd, JJ	2011	[Pyridine-2,6-^{14}C]BYI 02960: Aerobic soil metabolism in two US soils Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: MERVP038-1, Date: 2011-09-06. Amended: 2012-01-05 unpublished
20080615.01	Smeykal, H	2008	BYI 02960, pure substance: Vapour pressure—Final report Siemens AG, Frankfurt am Main, Germany Bayer CropScience, Report No.: 20080615.01, Date: 2008-10-10 unpublished
20090051.01	Smeykal, H	2010	BYI 02960, pure substance: Melting point, boiling point, thermal stability Siemens AG, Frankfurt am Main, Germany Bayer CropScience, Report No.: 20090051.01, Date: 2010-03-25 Unpublished
20110197.01	Smeykal, H	2011a	Flupyradifurone (BYl 02960), technical substance: Melting point, boiling point, thermal stability Siemens AG, Frankfurt am Main, Germany Bayer CropScience, Report No.: 20110197.01, Date: 2011-09-16 GLP/GEP: yes, unpublished
20110197.03	Smeykal, H	2011b	Flupyradifurone (BYl 02960), technical substance: flammability (solids) Siemens AG, Frankfurt am Main, Germany Bayer CropScience, Report No.: 20110197.03, Date: 2011-09-16 unpublished
20110197.05	Smeykal, H	2011c	Flupyradifurone (BYl 02960), technical substance: auto-flammability Siemens AG, Frankfurt am Main, Germany Bayer CropScience, Report No.: 20110197.05, Date: 2011-09-16 unpublished
20110197.07	Smeykal, H	2011d	Flupyradifurone (BYl 02960), technical substance: autoflammability (UN Bowes Cameron Cage Test) Siemens AG, Frankfurt am Main, Germany Bayer CropScience, Report No.: 20110197.07, Date: 2011-09-16 unpublished
20110197.04	Smeykal, H	2011e	Flupyradifurone (BYl 02960), technical substance: explosive properties Siemens AG, Frankfurt am Main, Germany Bayer CropScience, Report No.: 20110197.04, Date: 2011-09-16 unpublished
20110197.06	Smeykal, H	2011f	Flupyradifurone (BYl 02960), technical substance: oxidizing properties Siemens AG, Frankfurt am Main, Germany Bayer CropScience, Report No.: 20110197.06, Date: 2011-09-16 unpublished
20110197.02	Smeykal, H	2011g	Flupyradifurone (BYl 02960), technical substance: Particle size distribution Siemens AG, Frankfurt am Main, Germany Bayer CropScience, Report No.: 20110197.02, Date: 2011-09-16 unpublished
RARVY011	Sturdivant, DW	2012	BYI 02960 200 SL and BYI 02960 480 FS—Magnitude of the residue in/on soya beans Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY011, Date: 2012-05-15 unpublished

Code	Authors	Year	Title, Institute, Report reference
MEF-11/619	Sur, R & Dorn, S	2012a	Supplement to Report MEF-07/334—Kinetic Evaluation of the Aerobic Metabolism of BYI 02960 in four Soils for the Determination of Modelling Endpoints Bayer CropScience, Report No.: MEF-11/619, Date: 2012-01-19 unpublished
MEF-11/620	Sur, R.; Dorn, S.	2012b	Supplement to Report MEF-10/804—Kinetic Evaluation of the Aerobic Metabolism of BYI 02960 in four Soils for the Determination of Modelling Endpoints Bayer CropScience, Report No.: MEF-11/620, Date: 2012-01-19 unpublished
MEF-11/855	Sur, R.; Dorn, S.	2012c	Kinetic Evaluation of the Degradation Rate of [Ethyl-1-^{14}C] BYI 02960 and its Metabolite Difluoroacetic Acid (BYI 02960-DFA) for the Determination of Trigger and Modelling Endpoints Bayer CropScience, Report No.: MEF-11/855, Date: 2012-01-18 unpublished
MEF-11/838	Sur, R & Dorn, S	2012d	Kinetic Evaluation of the Degradation Rate of (Pyridine-2, 6-^{14}C] BYI 02960 and its metabolite 6-chloronicotinic acid (6-CNA) for the Determination of Trigger and Modelling Endpoints Bayer CropScience, Report No.: MEF-11/838, Date: 2012-01-18 unpublished
MEF-11/837	Sur, R & Dorn, S	2012e	Kinetic Evaluation of the Degradation Rate of 6-chloronicotinic acid (6-CNA) for the Determination of Trigger and Modelling Endpoints Bayer CropScience, Report No.: MEF-11/837, Date: 2012-01-18 unpublished
RARVY009	Timberlake, BC & Harbin, AM	2012	BYI 02960 200 SL and BYI 02960 480 FS—Magnitude of the residue in/on cotton (Crop Subgroup 20C) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVY009, Date: 2012-06-01 unpublished
MEF-11/498	Unold, M & Justus, K	2011	Metabolism of [ethyl-1-^{14}C]BYI 02960 in tomatoes Bayer CropScience, Report No.: MEF-11/498, Date: 2011-09-05 unpublished
MEF-10/730	Unold, M & Menke, U	2012	[Furanone-4-^{14}C] and [ethyl-1-^{14}C]BYI 02960: Aerobic aquatic metabolism Bayer CropScience, Report No.: MEF-10/730, Date: 2012-02-16 unpublished
RARVP041	Wade, JM & Netzband, DJ	2012	BYI 02960—Magnitude of the residue in laying hens Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP041, Date: 2012-04-05 unpublished
15-155-2438	Wagner, S	2011a	Chemical storage stability of BYI 02960—Flupyradifurone (BYI 02960) Bayer CropScience, Report No.: 15-155-2438, Date: 2011-07-19 unpublished
15-160-2527	Wagner, S	2011b	Stability to normal and elevated temperature, metals, and metal ions and corrosion characteristics to plastic containers of flupyradifurone (BYI 02960) according to OPPTS 830.6313 and 830.6320 Bayer CropScience, Report No.: 15-160-2527, Date: 2011-09-12 unpublished
MEF-10/856	Weber, E	2011a	Nature of the residues of [pyridinylmethyl-^{14}C]BYI02960 in processed commodities—High temperature hydrolysis Bayer CropScience, Report No.: MEF-10/856, Date: 2011-02-09 unpublished
MEF-11/555	Weber, E	2011b	[Ethyl-1-^{14}C]BYI 02960—Absorption, distribution, excretion, and metabolism in male rats Bayer CropScience, Report No.: MEF-11/555, Date: 2011-10-10 unpublished
MEF-11/556	Weber, E	2011c	[Furanone-4-^{14}C]BYI 02960—Absorption, distribution, excretion, and metabolism in the rat Bayer CropScience, Report No.: MEF-11/556, Date: 2011-12-22 unpublished
PA09/003	Wiche, A & Bogdoll, B	2011a	BYI 02960, pure substance: Solubility in distilled water (pH 7), at pH 4 and pH 9 (flask method) Bayer CropScience, Report No.: PA09/003, Date: 2011-06-17 unpublished
PA10/048	Wiche, A & Bogdoll, B	2011b	Flupyradifurone (BYI 02960), pure substance: Dissociation constant in water Bayer CropScience, Report No.: PA10/048, Date: 2011-09-16 unpublished
MERVL006	Woodard, D	2012	[Pyridine-2,6^{14}C]BYI 02960: Anaerobic Soil Metabolism in Springfield, Nebraska (USA) Soil Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: MERVL006, Date: 2012-02-14 unpublished
RARVP064	Woodard, DL	2012	BYI 02960 200 SL—Magnitude of the residue in/on mandarin orange (CG 10) Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RARVP064, Date: 2012-06-05 unpublished
MERVP027	Xu, T	2012	[Pyridine-2,6-^{14}C]BYI 02960: Anaerobic aquatic metabolism in two water/sediment systems Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: MERVP027, Date: 2012-01-17 unpublished

Code	Authors	Year	Title, Institute, Report reference
M-417799-01-1	Ziemer, F	2011a	Flupyradifurone (BYI 02960), technical substance: Statement on the viscosity according to OPPTS 830.7100 Bayer CropScience, Report No.: AF11/027, Edition Number: M-417799-01-1 Date: 2011-11-17 unpublished
AF11/026	Ziemer, F	2011b	Flupyradifurone (BYI 02960), technical substance: Statement on the miscibility according to OPPTS 830.6319 Bayer CropScience, Report No.: AF11/026, Date: 2011-11-17 unpublished
AF11/028	Ziemer, F	2011c	Flupyradifurone (BYI 02960), technical substance: Statement on the dielectric breakdown voltage according to OPPTS 830.6321 Bayer CropScience, Report No.: AF11/028, Date: 2011-11-17 unpublished.