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Pesticide residues in food – 2006

Joint FAO/WHO Meeting on Pesticide Residues

EVALUATIONS 2006

PART I – RESIDUES

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World Health Organization

Food and Agriculture Organization of the United Nations

Pesticide residues in food 2006

Evaluations
Part I – Residues

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Sponsored jointly by FAO and WHO

Joint meeting of the
FAO Panel of Experts on Pesticide Residues
in Food and the Environment
and the
WHO Core Assessment Group
Rome, Italy, 3–12 October 2006

WORLD HEALTH ORGANIZATION
FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS
Rome, 2007

Monographs containing summaries or residue data and toxicological data considered at the 2006 JMPR, together with recommendations, are available upon request from FAO or WHO under the title:

Pesticide residues in food 2006
Evaluations
Part I: Residues
FAO Plant Production and Protection Paper
and
Part II: Toxicology
WHO

This report contains the collective views of two international groups of experts and does not necessarily represent the decisions or the stated policy of the Food and Agriculture Organization of the United Nations or of the World Health Organization.

INTERNATIONAL PROGRAMME ON CHEMICAL SAFETY

The preparatory work for the toxicological evaluation of pesticide residues carried out by the WHO Expert Group on Pesticide Residues for consideration by the FAO/WHO Joint meeting on Pesticide Residues in Food and the Environment is actively supported by the International Programme on Chemical Safety (IPCS).
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ABBREVIATIONS

(Well-known abbreviations in general use are not included. Specific abbreviations for pesticide degradation products, etc., may be used in the monographs and these are either identified where first used or in a table within the monograph. Two-letter codes for pesticide formulations are given in the Manual on development and use of FAO and WHO specifications for pesticides, 1st Ed., FAO Plant Production and Protection Paper 173, FAO, Rome, 2002.)

AChE	anti-acetylcholinesterase
ACN	acetonitrile
ADI	acceptable daily intake
AFID	alkali flame-ionization detection or detector (equivalent to TSD, forerunner of NPD)
ai	active ingredient
AR	Applied radioactivity
ARfD	acute reference dose
AUC	area under the curve for concentration–time
BBCH	Biologische Bundesanstalt, Bundessortenamt and Chemical industry.
$BMDL_{10}$	benchmark-dose lower 95% confidence level
bw	body weight
CA	Chemical Abstracts
CAC	Codex Alimentarius Commission
CAS	Chemical Abstracts Services
CCN	Codex classification number (for compounds or commodities)
CCPR	Codex Committee on Pesticide Residues
CCRVDF	Codex Committee on Residue of Veterinary Drugs in Food
CEC	cation exchange capacity
CI	chemical ionization
CV	coefficient of variation (RSD)
CXL	Codex Maximum Residue Limit (Codex MRL). See MRL
d	days
DAT	days after (last) treatment
DCM	dichloromethane
DFG	Deutsche Forschungsgemeinschaft
DT_{50}	time for 50% decomposition (i.e. half-life)
DT_{90}	time for 90% decomposition
2D-TLC	two dimensional thin layer chromatography
dw	dry weight
ECD	electron capture detection or detector
EI	electron-impact (ionization), now more usually electron ionization
EPA	Environmental Protection Agency (usually US EPA)
eq	residue expressed as ai equivalent
F_1	first filial generation
F_2	second filial generation
FAO	Food and Agriculture Organization of the United Nations
FID	flame-ionization detection or detector
FPD	flame-photometric detection or detector
GAP	good agricultural practice(s)

GC	gas chromatography; the detector system used is usually also abbreviated as a suffix
GEMS/Food	Global Environment Monitoring System–Food Contamination Monitoring and Assessment Programme
GLP	good laboratory practice (i.e. the defined system, not in the general sense)
GPC	gel-permeation chromatography
GSH	glutathione
HPLC	high-performance liquid chromatography
HPLC-MS	high-performance liquid chromatography – mass spectrometry
HPLC-UV	high-performance liquid chromatography with UV absorption detection
hr	hour
HR	highest residue in the edible portion of a commodity found in trials used to estimate a maximum residue level in the commodity
HR-P	highest residue in a processed commodity calculated by multiplying the HR of the raw commodity by the corresponding processing factor
IARC	International Agency for Research on Cancer
IEDI	international estimated daily intake
IESTI	international estimate of short-term dietary intake
IPCS	International Programme on Chemical Safety
IR	infrared spectroscopy
ITD	ion-trap detector or detection
IUPAC	International Union of Pure and Applied Chemistry
JECFA	Joint Expert Committee on Food Additives
JMPR	Joint Meeting on Pesticide Residues
JMPS	Joint FAO/WHO Meeting on Pesticide Specifications
LC	liquid chromatography
LC-MS	liquid chromatography – mass spectrometry
LC_{50}	median lethal concentration
LD_{50}	median lethal dose
LOAEL	lowest-observed-adverse-effect level
LOAEC	lowest-observed-adverse-effect concentration
LOD	limit of detection
LOQ	limit of quantification
LSC	liquid scintillation counting or counter
M	molar = mole/L
MID	multiple ion detection (mass spectrometric)
MRL	Maximum Residue Limit. MRLs include draft MRLs and Codex MRLs (CXLs). The MRLs recommended by the JMPR on the basis of its estimates of maximum residue levels enter the Codex procedure as draft MRLs. They become Codex MRLs when they have passed through the procedure and have been adopted by the Codex Alimentarius Commission.
MS	mass spectrometry or mass spectrometric detector (suffix to GC- or LC-)
MSD	mass-selective detection or detector
MS/MS	tandem mass spectrometry
NOAEL	no-observed-adverse-effect level
NMR	nuclear magnetic resonance
NPD	nitrogen/phosphorus detector
OECD	Organization for Economic Co-operation and Development
om	amount of organic matter in soil

PES	post extracted solids
PF	processing factor
PHI	pre-harvest interval
ppm	parts per million (used only with reference to the concentration of a pesticide in a diet, in all other contexts the terms mg/kg or mg/l are used)
P_{ow}	octanol–water partition coefficient
RAC	raw agricultural commodity
r.d.	relative density (formerly called specific gravity)
RfD	reference dose (usually in phrase “acute RfD”)
RSD	precision under repeatability conditions (measurements within one day or one run) expressed as relative standard deviation (= coefficient of variation)
SD	standard deviation
SPE	solid-phase extraction (may also describe a post-extraction clean-up process)
STMR	supervised trials median residue
STMR-P	supervised trials median residue in a processed commodity calculated by multiplying the STMR of the raw commodity by the corresponding processing factor
t	tonne (metric ton)
TAR	total applied (or administered) radioactivity
TLC	thin-layer chromatography
TRR	total radioactive residue
TMDI	theoretical maximum daily intake
TSD	thermionic specific detection or detector (equivalent to AFID, forerunners of NPD)
USDA	US Department of Agriculture
US FDA	US Food and Drug Administration
UV	ultraviolet (radiation)
W	the previous recommendation is withdrawn, or withdrawal of the existing Codex or draft MRL is recommended
WHO	World Health Organization

USE OF JMPR REPORTS AND EVALUATIONS BY REGISTRATION AUTHORITIES

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

INTRODUCTION

The Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Core Assessment Group (JMPR), held in Rome, 3-12 October 2006, contains a summary of the evaluations of residues in foods of the various pesticides considered, as well as information on the general principles followed by the Meeting (JMPR, 2006). The present document contains summaries of the residues data considered, together with the recommendations made.

The Evaluations are issued in two parts:
Part I: Residues (by FAO);
Part II: Toxicology (by WHO).

For those interested in both aspects of pesticide evaluation, both parts and the Report containing summaries of residues and toxicological considerations are available.

Some of the compounds considered at the Meeting were previously evaluated and reported on in earlier publications. In general, only new information is summarized in the relevant monographs but reference is made to previously published evaluations, which should also be consulted. In the case of older compounds which are re-evaluated as part of the periodic review programme of the CCPR, a review of all available data, including data which may have previously been submitted, is carried out. Compounds evaluated for the first time and those evaluated in the CCPR periodic review programme are identified in the Table of Contents.

Summaries of recommended MRLs, STMR and HR levels and assessments of dietary intake, are published as Annexes 1, 3 and 4 in the Report, and reference is made to this report.

The name of the compound appearing as the title of each monograph is followed by its Codex Classification Number in parentheses.

References to previous Reports and Evaluations of Joint Meetings are listed in Annex I.

Acknowledgements

The monographs in these Evaluations were prepared by the following participants in the 2006 JMPR, for the FAO Panel of Experts on Pesticide Residues in Food and the Environment:

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Note. Any comment on pesticide residues in food and their evaluation should be addressed to the:

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JMPR, 2006. Pesticide residues in Food – 2006. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Core Assessment Group on Pesticide Residues, Rome, Italy, 3-12 October 2006. WHO and FAO, Rome, 2006.

FENPROPATHRIN (185)

First draft prepared by Dr. Yukiko Yamada, National Food Research Institute, Tsukuba, Japan.

EXPLANATION

Fenpropathrin, an insecticide/acaricide, was first evaluated by JMPR in 1993 as a new compound. At that time the Meeting allocated an ADI of 0-0.03 mg/kg and recommended 14 MRLs, later adopted by the Codex Alimentarius Commission in 1995 or 1997 as Codex MRLs. The residue definition is fenpropathrin (residue is fat soluble).

At the 38th Session of the CCPR in 2006, the Delegation of India requested the elaboration of an MRL for tea. Fenpropathrin was added to the agenda of the current Meeting for the evaluation its use on tea, pending availability of data. The Meeting received the current label in India, results of supervised residue trials, processing and plant metabolism studies and methods of analysis.

Plant metabolism

The 1993 JMPR reviewed plant metabolism studies on apple, beans, cotton and tomato and concluded that fenpropathrin itself was the primary component of the residues in the fruits of plants but degradation products constituted the greater part of the residues present in the leaves. In addition, the 1993 JMPR concluded that any uptake of residues from the soil is too slow for detectable residues to occur in succeeding crops, especially in view of the comparatively short persistence of the compound in soils.

The current Meeting received studies conducted to determine the metabolism of fenpropathrin in leaves.

Cabbage

The metabolism of radio-labelled fenpropathrin was investigated in cabbages grown and treated in a greenhouse (Mikami *et al*., 1983; Report No. FM-30-0009). Fenpropathrin labelled at either the cyano group (referred to as ^{14}CN), or the C1 position of the cyclopropyl ring (cyclopropyl-^{14}C), or the benzylphenyl ring (benzyl-^{14}C) was dissolved in methanol and evenly applied to the upper surface of two 3rd–4th leaves of cabbage seedlings at a rate of 22 µg per leaf. The cabbages were sampled immediately after application and at 3, 7, 14, 21, 28, 35 and 42 days after application.

The cabbage samples were separated into treated leaves and un-treated shoot portions. The treated leaves were rinsed twice with methanol and the radioactivity in the wash, leaves and untreated shoots determined. The leaves and the untreated shoots were separately homogenized and extracted with a solution of methanol:chloroform:distilled water (4:2:1). Metabolites in cabbages treated with ^{14}C-fenpropathrin were identified by thin layer chromatography (TLC). Metabolites present on TLC from three labelled preparations were compared to distinguish products retaining the ester linkage from hydrolysis products. The extractable components in cabbages harvested 28 and 42 days after application are shown in Table 1.

Table 1. Extractable components in cabbage samples harvested 28 and 42 days after application. (Mikami *et al*., 1983; Report No. FM-30-0009).

	% of the applied ^{14}C					
	Cyclopropyl-^{14}C		^{14}CN		Benzyl-^{14}C	
	28 days	42 days	28 days	42 days	28 days	42 days
Surface Wash:						
Fenpropathrin	0.6	0.3	1.0	0.6	1.7	0.4
Others	0.3	0.1	0.4	0.3	0.3	0.1
Surface Wash Total	0.9	0.4	1.4	0.9	2.0	0.5

	% of the applied ^{14}C					
	Cyclopropyl-^{14}C		^{14}CN		Benzyl-^{14}C	
	28 days	42 days	28 days	42 days	28 days	42 days
methanol:chloroform:distilled water (4:2:1) Extracts:						
Fenpropathrin	15.8	11.7	16.9	6.0	12.9	11.3
$CONH_2$-fenpropathrin	0.7	< 0.1	0.3	< 0.1	0.9	< 0.1
COOH-fenpropathrin	0.4	0.4	0.6	0.3	0.7	< 0.1
2'-OH-fenpropathrin	0.1	< 0.1	0.4	< 0.1	0.4	< 0.1
Fenpropathrin-CH_2OH	0.4	0.3	0.5	0.5	0.6	0.2
TMPA-lactone	0.1	< 0.1	-	-	-	-
TMPA-CH_2OH-lactone	0.8	0.9	-	-	-	-
COOH-fenpropathrin-conjugate	0.4	0.2	0.6	0.4	0.6	0.7
2'-OH-fenpropathrin-conjugate	0.2	0.1	0.1	0.1	0.1	0.2
4'-OH-fenpropathrin-conjugate	1.3	0.7	1.6	1.0	0.6	0.8
Fenpropathrin-CH_2OH-conjugate	3.5	4.0	3.4	4.3	4.2	4.0
2'-OH-fenpropathrin-CH_2OH-conjugate 4'-OH-fenpropathrin-CH_2OH-conjugate	4.8	4.5	4.6	4.5	5.9	6.2
2'-OH-fenpropathrin-$(CH_2OH)_2$-conjugate 4'-OH-fenpropathrin-$(CH_2OH)_2$-conjugate	20.3	22.0	18.6	20.7	19.4	21.6
TMPA-conjugate	0.9	0.8	-	-	-	-
TMPA-CH_2OH-conjugate	1.1	1.0	-	-	-	-
TMPA-COOH-conjugate	3.7	4.2	-	-	-	-
TMPA-CH_2OH-lactone-conjugate	11.3	11.1	-	-	-	-
Pbalc-conjugate	-	-	-	-	0.1	0.1
Pbacid-conjugate	-	-	-	-	0.8	1.1
2'-OH-Pbacid-conjugate	-	-	-	-	6.9	7.4
4'-OH-Pbacid-conjugate	-	-	-	-	4.5	4.6
Others	5.2	5.8	4.2	5.8	9.6	9.1
Extracts Total	71.0	67.7	51.8	43.6	68.2	67.3
Unextractable ^{14}C Total	2.6	5.1	6.7	11.3	4.0	7.5
Treated Leaves Total	74.5	73.2	59.9	55.8	74.2	75.3
Untreated Shoots	0.9	1.2	0.6	0.7	0.4	0.4
Overall Total	75.4	74.4	60.5	56.5	74.6	75.7

The study demonstrated that after foliar application of ^{14}C-fenpropathrin to cabbages the radioactive carbon remaining on the surface of treated leaves decreased, as ^{14}C in the leaves increased. Most of the recovered radioactivity was in the treated leaves and less than 1.2% of the applied radioactivity was found in the untreated shoots indicating that there is little translocation of fenpropathrin and its metabolites from the application site to other parts of the plant.

TLC showed that, in all cases, the predominant radioactive component in the surface washes was the parent compound. Fenpropathrin underwent ester cleavage, hydrolysis of the –CN group to the $–CONH_2$ and the –COOH groups, hydroxylation at either or both of the gem-dimethyl group with subsequent oxidation to carboxylic acid, and hydroxylation at the 2'- or 4'-position of the phenoxy group. Most of the resultant carboxylic acids and alcohols occurred as glycoside conjugates in plants.

Abscised leaves of apple, cabbage, kidney bean, mandarin orange, tomato and vine

Mikami *et al*., (1983; Report No. FM-30-0009) also conducted a study on the fate of HCN and 2,2,3,3-tetramethylcyclopropanecarboxylic acid (TMPA) in abscised leaves of apple, cabbage, kidney bean, mandarin orange, tomato and vine. TMPA labelled at the C1 position of the cyclopropyl ring (^{14}C-TMPA) was prepared. Two abscised leaves from each plant were placed in 100 mL distilled water containing ^{14}C-TMPA at a concentration of 1.0 ppm. After cultivation for five days, the leaves were extracted with methanol:chloroform:water (4:2:1). In a separate experiment, abscised leaves of cabbage and bean plants were placed in a 100 ppm solution of ^{14}C-TMPA in order to obtain large quantities of metabolites for characterization. The extracts were partitioned between ethyl ether and distilled water. After acidification the aqueous layer was partitioned with ethyl acetate. The extractable components in abscised leaves of various plants over a 5 day period are shown in Table 2.

Table 2. Extractable components in abscised leaves of various plants over a 5 day period. (Mikami *et al*., 1983; Report No. FM-30-0009)

	% of the applied ^{14}C					
	Apple	Bean	Cabbage	Orange	Tomato	Vine
Extracts:						
TMPA	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
TMPA-Gu	21.5	14.7	3.2	3.0	2.0	6.6
CH_2OH-TMPA-Gu	5.2	3.2	3.4	0.2	0.2	0.4
TMPA-CH_2OH-Gu	1.3	-	-	-	-	0.1
TMPA-Gu-Gu	5.4	2.8	-	-	12.8	0.7
TMPA-malonyl-Gu	0.7	56.2	70.5	3.1	4.3	0.1
CH_2OH-TMPA-malonyl-Gu	-	-	1.5	0.4	-	-
Others	1.5	3.0	0.2	0.5	1.1	0.2
Extracts Total	35.6	79.9	78.8	7.2	20.4	8.1
Unextractable ^{14}C Total	0.3	0.2	0.2	0.4	0.2	0.3
Abscised Leaves Total	35.9	80.1	79.0	7.6	20.6	8.4
Aqueous Solution:						
TMPA	57.1	24.5	13.2	63.8	76.1	83.0
Others	1.2	0.8	0.9	0.7	1.6	0.5
Aqueous Solution Total	58.3	25.3	14.1	64.5	77.7	83.5
Overall Total	94.2	105.4	93.1	72.1	98.3	91.9

TMPA was readily converted in plants to more polar products. In orange, cabbage and bean plants, the malonylglucoside was mainly formed. In tomato, the gentiobioside was predominant.

Further work was carried out using $K^{14}CN$. Two abscised cabbage leaves were treated for four hours with distilled water containing $K^{14}CN$ and then transferred to $K^{14}CN$-free distilled water. The treated leaves were extracted at specific intervals after dosing with $K^{14}CN$, and the extracts were subject to TLC.

There was a gradual increase in the amount of volatile ^{14}C trapped in NaOH, and most of the radioactivity was considered to be $^{14}CO_2$. At least six ^{14}C metabolites were present in the extracts of the abscised leaves treated with $K^{14}CN$. The extractable components in abscised leaves of cabbage over a 2 day period are shown in Table 3.

Table 3. Extractable components in abscised leaves of cabbage over a 2 day period following treatment with $K^{14}CN$. (Mikami *et al*., 1983; Report No. FM-30-0009).

	% of the applied ^{14}C			
	2 h	4 h	8 h	48 h
Extracts:				
β-Cyanoalanine	0.6	0.9	0.9	0.6
Asparagine	1.8	2.4	2.8	1.1
Aspartic acid	0.7	1.0	1.1	1.3
γ-Glutamyl-β-cyanoalanine	3.8	5.4	5.1	2.4
Others	0.7	1.2	1.1	1.9
Extracts Total	**7.6**	**10.9**	**11.0**	**7.3**
Unextractable ^{14}C Total	**2.9**	**3.7**	**4.5**	**7.7**
Treated Leaves Total	**10.5**	**14.6**	**15.5**	**15.0**
Aqueous Solution	**86.9**	**79.6**	**83.1**	**63.2**
Overall Total	**97.4**	**94.2**	**98.6**	**78.2**

This study demonstrates that $H^{14}CN$ liberated on ester hydrolysis of fenpropathrin and its derivatives would be rapidly incorporated into β-cyanoalanine, asparagine, aspartic acid and γ-glutamyl-β-cyanoalanine, with ultimate formation of $^{14}CO_2$ and unextractable ^{14}C residues.

RESIDUE ANALYSIS

Analytical methods

The Meeting received information on a method of analysis developed and used in the supervised trials on black tea conducted in India (Lavakumar, S., *et al*., 2003; Report No. 11861 and Lavakumar, S., *et al*., 2004; Report No.14246). The method uses the same principle as that of the methods developed by the manufacturer and reviewed by the 1999 JMPR. The method involves extraction of fenpropathrin from black tea with an acetonitrile-water mixture (65:35). The extracts were filtered under suction and the filtrate was concentrated to approximately 150 mL. This extract was then diluted with 225 mL of 5 percent aqueous sodium chloride solution and extracted with hexane-ether mixture (8:2). The combined extracts were filtered through sodium sulphate and evaporated to near dryness before being dissolved in 5 mL of hexane. Clean-up was performed and the eluate evaporated and dissolved in acetone. Fenpropathrin was quantified by gas chromatography using an electron capture detector (ECD).

Recovery tests were conducted at a range of 0.05–2.0 mg/kg. Only the summaries of results were provided as shown in Table 4. The procedural recovery ranged between 88 and 96%. The limit of quantification (LOQ) was 0.05 mg/kg.

Table 4. Procedural recovery of fenpropathrin from fortified black tea (Lavakumar, S., *et al.*, 2004; Report No.14246)

Fortification levels (mg/kg)	Green tea		Made tea	
	Recovery %	RSD %	Recovery %	RSD %
0.05	88	2.2	88	1.6
	88		86	
	92		88	
	90		88	
	92		90	
0.5	90	1.6	94	2.7
	88		88	
	90		92	
	90		92	
	92		94	
1.0	88	1.7	89	2.3
	91		92	
	89		93	
2.0	93	0.6	96	2.3
	93		93	
	92		92	
Mean	90		91	

The Meeting also received information from the Government of India on the method described in the Journal of AOAC (1999) and used in some of supervised trials conducted in India. The method uses the same principle as that of the methods developed by the manufacturer and reviewed by the 1999 JMPR. Twenty grams of black tea sample was extracted with 150 mL of acetonitrile:water (2:1, v/v) for two hours. The contents were filtered and 200 mL of 4% NaCl and 60 mL of hexane were added to the filtrate. After partitioning, the hexane layer was passed through an anhydrous sodium sulfate layer. The extract was evaporated to dryness and the residue was dissolved in 10 mL of hexane. About 30 mL of hexane-saturated acetonitrile was added and the acetonitrile layer was drained onto anhydrous sodium sulfate. The acetonitrile extract was then evaporated to dryness at 60°C. The concentrated residue was dissolved in 5 mL of hexane and cleaned up using 10 g of 5% deactivated Florisil and 150 mL of 6% diethyl ether in hexane as the eluting solvent. Prior to elution the column was washed with 50 mL of hexane. The eluate collected was concentrated at about 60°C to dryness and diluted with 10 mL of hexane and quantified by gas chromatography using ECD detector.

The LOQ was claimed to be 0.05 mg/kg. Method validation was attempted by analysis of fortified black tea samples at 0.283 mg/kg, much higher than the claimed LOQ. No recovery test was reported at 0.05 mg/kg. Only the summary of results was provided as shown in Table 5.

Table 5. Procedural recovery of fenpropathrin from fortified tea leaves.

Fortification levels (mg/kg)	Tea leaves (green)	
	Recovery %	RSD %
0.283	89	
	90	
	83	
	95	
	103	
Mean	92	8.1

USE PATTERN

Fenpropathrin is registered for the control of mites in tea. The label from India was provided and the Indian GAP is summarized in Table 6.

Table 6. Registered use of fenpropathrin on tea.

Crop	Country	For G	Formulation	Application					PHI days
				Method	Rate kg ai/ha	Spray conc. kg ai/hLl	Water L/ha	No.	
Tea	India	F	300 EC	Foliar	0.05 – 0.06	0.01 – 0.015	400 – 500	1	7

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information and results of supervised trials conducted in India. The application rate and method, information on varieties, plot size and sampling were provided. The residues in control plots were all below the LOQ and therefore not recorded in the following tables. Residue data are not adjusted for recovery. When residues were not detected they are shown as below the LOQ. Residues, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure.

Data according to GAP are double-underlined. Data from trials not in accordance with GAP but used for the estimation of maximum residue level are single-underlined. No procedural recovery information was available for the analysis of samples from supervised trials.

Black Tea

All supervised trials on black tea were conducted in India. Matured tea leaves were collected from each plot and processed[1]. In the trials conducted in 2004, and reported in Report No. 14246, leaves were processed by machine drying.

[1] The good manufacturing practice of black tea in India is as follows (Submission from the Government of India):
Withering: Harvested tea shoots were spread at a thickness of 2.5 cm and allowed to wither under ambient conditions for a period of 16-20 hours.
CTC: The withered leaves were passed into a rolling machine and rolled for 30 minutes. The rolled leaves were taken out and passed thrice through the CTC (Crush, Tear & Curl) machine, to give three cuts.
Oxidation (Fermentation): The rolled CTC tea, was spread over fermenting trays at a thickness of 1.3 - 1.8 cm for a period of one hour. Humidity was maintained at 90-95%.
Drying: The fermented 'dhool' (ground, fermented green tea shoot) was put on drying chamber. Hot air was blown over the tea with an inlet temperature of about 95-115° C. After 30 minutes of drying, dried tea was obtained with 2-3% moisture.

The results of these trials are summarized in Table 7.

Table 7. Fenpropathrin residues in black tea from supervised trials in India.

country, month/year, season (variety)	Application				PHI days	Residues[1] mg/kg	Reference
	Form	kg ai/ha	Water L/ha	No.			
GAP	300 EC	0.05– 0.06	400 –500	1	7		
Valparai, India January 2002 Season I (UPASI-9)	300 EC	0.06	400	1	0 1 3 5 7 10 14	2.74 1.75 1.17 0.61 0.17 < 0.05 < 0.05	Lavakumar, S., *et al.*, 2003 (Report No. 11861) also in Submission of the Government of India
Valparai, India September 2002 Season II (UPASI-9)	300 EC	0.06	400	1	0 1 3 5 7 10 14	2.69 1.69 1.10 0.61 0.18 < 0.05 < 0.05	Lavakumar, S., *et al.*, 2003 (Report No. 11861) also in Submission of the Government of India
Valparai, India May 2003 Season III (UPASI-9)	300 EC	0.06	400	1	0 1 3 5 7 10 14	2.22 1.45 0.91 0.49 0.14 < 0.05 < 0.05	Lavakumar, S., *et al.*, 2003 (Report No. 11861)
Valparai, India January 2002 Season I (UPASI-9)	300 EC	0.12	400	1	0 1 3 5 7 10 14	5.47 3.13 2.24 1.04 0.37 < 0.05 < 0.05	Lavakumar, S., *et al.*, 2003 (Report No. 11861)
Valparai, India September 2002 Season II (UPASI-9)	300 EC	0.12	400	1	0 1 3 5 7 10 14	5.24 3.02 2.22 1.08 0.36 < 0.05 < 0.05	Lavakumar, S., *et al.*, 2003 (Report No. 11861)
Valparai, India May 2003 Season III (UPASI-9)	300 EC	0.12	400	1	0 1 3 5 7 10 14	4.40 2.57 1.89 0.86 0.30 < 0.05 < 0.05	Lavakumar, S., *et al.*, 2003 (Report No. 11861)
Valparai, India January 2004 Fourth Season (UPASI-9)	300 EC	0.06	400	1	0 1 3 5 7 10 14	0.85 0.50 0.17 < 0.05 ≤ 0.05 < 0.05 < 0.05	Lavakumar, S., *et al.*, 2004 (Report No. 14246) also in Submission of the Government of India
Valparai, India January 2004 Fourth Season (UPASI-9)	300 EC	0.12	400	1	0 1 3 5 7 10 14	1.62 0.93 0.30 < 0.05 ≤ 0.05 < 0.05 < 0.05	Lavakumar, S., *et al.*, 2004 (Report No. 14246)

country, month/year, season (variety)	Application				PHI days	Residues[1] mg/kg	Reference
	Form	kg ai/ha	Water L/ha	No.			
Gudalur, India June 2004 (Mixed clones)	300 EC	0.06	450	1	0 7 10 14	2.22 <u>0.14</u> < 0.05 < 0.05	Submission of the Government of India
Tocklai, India November 2005 (Mixed clones)	300 EC	0.03	400	1	0 7 10	12.0 <u>1.38</u> 0.12	Submission of the Government of India

[1]Average of three replications.

Green Tea

All supervised trials on green tea were conducted in India. Samples leaves were air dried after harvest. The results of these trials are summarized in Table 8. No procedural recovery information was available for the analysis of samples from supervised trials.

Table 8. Fenpropathrin residues in green tea from supervised trials in India

country, month/year, season (variety)	Application				PHI days	Residues[1] mg/kg	Reference
	Form	kg ai/ha	Water Ll/ha	No.			
GAP	300 EC	0.05–0.06	400–500	1	7		
Valparai, India January 2004 Fourth Season (UPASI-9)	300 EC	0.06	400	1	0 1 3 5 7 10 14	1.96 1.32 0.83 0.45 <u>0.13</u> < 0.05 < 0.05	Lavakumar, S., *et al.*, 2004 (Report No. 14246)
Valparai, India January 2004 Fourth Season (UPASI-9)	300 EC	0.12	400	1	0 1 3 5 7 10 14	4.20 2.43 1.55 0.90 0.29 < 0.05 < 0.05	Lavakumar, S., *et al.*, 2004 (Report No. 14246)

[1]Average of three replicates.

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

Processing tea into tea decoctions

Processing studies were conducted in India to determine the residues in tea decoctions from leaf tea samples treated with fenpropathrin (Lavakumar, S., *et al.*, 2004; Report No. 14246).

The trials consisted of application of an EC formulation containing 300 g ai/L of fenpropathrin at three treatment regimes involving different use rates: (i) a single application at a rate of 0.06 kg ai/ha; (ii) a single application at a rate of 0.12 kg ai/ha; (iii) untreated. Samples were collected at 0, 1, 3, 5, 7, 10 and 14 days after application. Fifty grams of leaf tea sample was collected and boiled in 100 mL of water for 5 minutes in a 500 mL conical flask. This was then filtered and concentrated to 10mL. Partitioning and clean-up were then carried out as described in the method of analysis for black tea. Residues of fenpropathrin in the tea decoctions were determined by gas chromatography with electron capture detection (ECD). No information was available on procedural recoveries for the analysis of samples

A transfer factor was used to indicate the amount of fenpropathrin transferred from tea leaves to water (decoction) during brewing.

The transfer factor was calculated by dividing the total residue in mg in the decoction (concentrated to 10 mL) by the total residue in mg in tea leaves (50 g) assuming that the specific gravity of the decoction is the same as that of water. A precise processing factor could not be estimated because the residue levels in decoction before concentration were too low to quantify.

The results are summarized in Tables 9 and 10.

Table 9. Fenpropathrin residues in black tea decoctions from supervised trials in India (Lavakumar, S., *et al*., 2004; Report No. 14246).

Country, month/year, season (variety)	PHI (days)	Residues[1] (mg/kg)		Transfer factor	Reference
		Black tea	Tea decoction (10 ml)		
Valparai, India January 2004 Fourth Season (UPASI-9)	0 1 3 5 7 10 14	0.85 0.50 0.17 < 0.05 <u>< 0.05</u> < 0.05 < 0.05	0.13 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.03 < 0.02 < 0.06	Lavakumar, S., *et al.*, 2004 (Report No. 14246)
Valparai, India January 2004 Fourth Season (UPASI-9)	0 1 3 5 7 10 14	1.62 0.93 0.30 < 0.05 <u>< 0.05</u> < 0.05 < 0.05	0.18 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.02 < 0.01 < 0.03	Lavakumar, S., *et al.*, 2004 (Report No. 14246)

[1]Average of three replicates.

Table 10. Fenpropathrin residues in green tea decoctions from supervised trials in India (Lavakumar, S., *et al.*, 2004; Report No. 14246).

Country, month/year, season (variety)	PHI (days)	Residues[1] (mg/kg)		Transfer factor	Reference
		Green tea	Tea decoction (10 ml)		
Valparai, India January 2004 Fourth Season (UPASI-9)	0 1 3 5 7 10 14	1.96 1.32 0.83 0.45 <u>0.13</u> < 0.05 < 0.05	0.11 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.01 < 0.008 < 0.01 < 0.02 < 0.08	Lavakumar, S., *et al.*, 2004 (Report No. 14246)
Valparai, India January 2004 Fourth Season (UPASI-9)	0 1 3 5 7 10 14	4.20 2.43 1.55 0.90 0.29 < 0.05 < 0.05	0.25 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.01 < 0.004 < 0.006 < 0.01 < 0.03	Lavakumar, S., *et al.*, 2004 (Report No. 14246)

[1]Average of three replicates.

APPRAISAL

Fenpropathrin, an insecticide/acaricide, was first evaluated by the JMPR in 1993 as a new compound. The JMPR allocated an ADI of 0-0.03 mg/kg and recommended 14 MRLs, later adopted by the Codex Alimentarius Commission as Codex MRLs. The residue definition is fenpropathrin (the residue is fat soluble).

At the 38th Session of the CCPR in 2006, the Delegation of India requested the elaboration of an MRL for tea. Fenpropathrin was added to the agenda of the current Meeting for evaluation pending

availability of trial data on tea. The Meeting received the current label from India, results of supervised trials, a processing and plant metabolism study and methods of analysis.

Metabolism

Plant metabolism

The Meeting received studies conducted to determine metabolism of fenpropathrin in leaves.

The metabolism of radio-labelled fenpropathrin was investigated in cabbages grown and treated in a greenhouse. After foliar application of ^{14}C -fenpropathrin to cabbages the radioactive carbon on the surface of treated leaves decreased as ^{14}C in the leaves increased. Most of the recovered radiocarbon was in the treated leaves and less than 1.2% of the applied radioactive carbon was found in the untreated shoots. This indicates that fenpropathrin and its metabolites only slightly translocate from the site of application to other parts of the plant. The predominant radioactive component in the surface washes was the parent compound, fenpropathrin. The major radioactive components in leaves were fenpropathrin and the conjugates of metabolites with a –CH2OH group.

The fate of HCN and 2,2,3,3-tetramethylcyclopropanecarboxylic acid (TMPA) in abscised leaves of apple, cabbage, kidney bean, mandarin orange, tomato and vine was investigated. TMPA was readily converted in plants to more polar products. In orange, cabbages and bean plants, the malonyl glucoside was mainly formed. In tomato, the gentiobioside was predominant. Further work was carried out using $K^{14}CN$. There was a gradual increase in the amount of volatile ^{14}C trapped in NaOH solution, most of the radioactive carbon was considered to be $^{14}CO_2$. The study demonstrates that $H^{14}CN$ liberated on ester hydrolysis of fenpropathrin and its derivatives would be rapidly incorporated into β-cyanoalanine, asparagine, aspartic acid and γ-glutamyl-β-cyanoalanine, with ultimate formation of $^{14}CO_2$ and unextractable ^{14}C residues.

The Meeting confirmed that the residue definition of fenpropathrin is appropriate for leafy vegetables as well as for tea.

Methods of residue analysis

The Meeting received descriptions and validation data for methods of analysis used in the supervised trials on tea conducted in India.

Both methods use the same principle as that of the methods developed by the manufacturer and reviewed by the 1999 JMPR and involve extraction of fenpropathrin, partitioning, clean-up and analysis using GC-ECD. For one method, recovery test were conducted at a range of 0.05–2 mg/kg and procedural recoveries in this range were 88–96%. The limit of quantification was 0.05 mg/kg. For the second method, a recovery test was conducted at 0.283 mg/kg resulting in procedural recovery around 90%. No details for the procedural recovery tests were reported for either method.

Results of supervised trials on crops

The Meeting received information and results from a total of 12 supervised trials conducted in India on tea. The current product label from India was provided. No information was available for procedural recoveries in the analysis of samples from supervised trials.

Tea

Fenpropathrin (300 g ai/kg EC) is registered in India for use on tea at 0.05–0.06 kg ai/ha with a PHI of 7 days.

In ten trials, collected tea leaves were processed into black tea. In six trials this was achieved through withering, crush/tear/curl process, oxidation and drying while in another two trials by machine drying. In trials with conditions matching the registered use, residues of fenpropathrin were in rank order: < 0.05, 0.14, 0.14, 0.17 and 0.18 mg/kg. In one trial conducted with double rates, the residues in

the black tea from the sample taken 7 days after treatment were < 0.05 mg/kg. In another trial conducted with half rates, the residues in black tea from the sample taken 7 days after treatment were 1.38 mg/kg. No information on the possible cause of the high residue concentration was available. Although the used rate was half of the Indian GAP rate, the Meeting decided to include this value for estimating the maximum residue level.

In two additional trials, collected tea leaves were air-dried to prepare green tea. In trials where conditions matching the registered use pattern, residues of fenpropathrin were 0.13 mg/kg.

Since growing conditions and application rate/method for black tea and green tea are equivalent with the only difference being in processing methods, the Meeting estimated a maximum residue level for tea, green, black on the basis of combined residue results: < 0.05 (2), 0.13, 0.14, 0.14, 0.17, 0.18 and 1.38 mg/kg.

The Meeting estimated a maximum residue level, STMR and HR at 2 mg/kg, 0.14 mg/kg and 1.38 mg/kg respectively.

Fate of residues during processing

The Meeting received information on the fate of fenpropathrin during the brewing of tea.

Black tea (50 g) from field trials at the maximum rate or green tea (50 g) from trials at the maximum rate or double rates was brewed by boiling in 100 mL of water in a flask. Tea samples and concentrated decoctions were analyzed. However, no information was available on procedural recoveries for the analysis of samples.

A transfer factor was used to indicate the amount of fenpropathrin transferred from tea leaves to water (decoction) during brewing. The transfer factor was tentatively calculated by dividing the total residue (mg) in the decoction (concentrated to 10 mL) by the total residue (mg) in tea leaves (50 g) assuming that the specific gravity of the decoction is the same as that of water. No estimation of the processing factor was possible as the residue levels in the decoction before concentration were too low to quantify. The results indicate that only a small amount of fenpropathrin was transferred into the decoction as predicted from the highly fat-soluble nature of the compound. Table 3 shows the calculated transfer factor.

Table 11. Transfer factor from tea to decoction.

Process	Transfer factor	Best estimate
Black tea - decoction	0.03, < 0.02, < 0.06 0.02, < 0.01, < 0.03	0.03
Green tea - decoction	0.01, < 0.008, < 0.01, < 0.02, < 0.08 0.01, < 0.004, < 0.006, < 0.01, < 0.03	0.01

Where the residues in black or green tea were below the LOQ, the transfer factor was not calculated.

RECOMMENDATIONS

On the basis of the data from supervised trials on tea, the Meeting concluded that the residue concentration below is suitable for establishing an MRL and for assessing dietary intakes.

Definition of the residue: *fenpropathrin*

The residue is fat soluble.

Table 12. Summary of recommendations.

Commodity		Recommended MRL mg/kg		STMR/ STMR-P mg/kg	HR/HR-P mg/kg
CCN	Name	New	Previous		
DT 1114	Tea, Green, Black	2	-	0.14	1.38

DIETARY RISK ASSESSMENT

Long-term intake

The long-term dietary intakes were estimated for the 13 cluster diets using maximum residue levels for fenpropathrin recommended by the 1999 Meeting and an STMR for tea estimated by the current. The maximum ADI is 0.03 mg/kg and the calculated intakes were 3–80% of the maximum ADI. The Meeting concluded that the long-term intake of residues of fenpropathrin resulting from the uses considered by the Meeting was unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intakes (IESTIs) of fenpropathrin by general population and by children were calculated for tea, green, black, for which an HR was estimated by the current Meeting. As it is not known if it is necessary to establish an ARfD, no the short-term intake assessment could be determined.

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Anonymous 2005. (March) Submission of the Government of India: Data information required for CCPR/JMPR evaluations for fixation of MRL of fenpropathrin in tea (Group 24 – TEAS, Codex Classification Group 066: Teas)

FLUDIOXONIL (211)

First draft prepared by Dr. Katerina Mastovska, Agricultural Research Service, United States Department of Agriculture, Wyndmoor PA,USA.

EXPLANATION

Fludioxonil was first evaluated at the 2004 JMPR Meeting. The 2004 Meeting concluded that the residue definition for plant commodities for compliance with MRLs and for estimation of dietary intake was fludioxonil only. No MRL was recommended for pome fruit due to an insufficient number of post-harvest trials at the critical GAP.

Additional supervised trials to support the use pattern were carried out in 2005. The present Meeting received information on the post-harvest use pattern, residue analysis, and post-harvest trials on apples and pears performed in 2000, 2001, and 2005. Results of an apple processing study were also reported.

RESIDUE ANALYSIS

Analytical methods

Two single-residue methods (REM 133.04 and AG-597) and one multiresidue method (DFG S19) for the determination of fludioxonil residues in plant materials were reported to the JMPR meeting in 2004. The 2004 Meeting concluded that these analytical methods were adequate for gathering data in supervised trials, processing studies and for monitoring and enforcing MRLs in samples of plant origin.

The present Meeting received information on the residue analysis of fludioxonil in treated apples and pears and in samples resulting from an apple processing study. Analytical method AG-597B (Campbell, 1996; Williams, 1998) was employed for the determination of fludioxonil residues resulting from post-harvest application to apples and pears in the USA. In brief, the AG-597B method for apples and pears involved the following steps: (i) homogenization of the sample with acetonitrile-water (9:1, v/v), (ii) filtration, (iii) evaporation of an aliquot to remove acetonitrile, (iv) dilution of the concentrated extract with saturated NaCl solution and partitioning with methyl *tert*-butyl ether (MTBE), (v) addition of toluene to the organic phase and evaporation of MTBE, (vi) addition of hexane, (vii) clean-up of the extract using a silica SPE cartridge eluted with dichloromethane-toluene (1:1, v/v), (viii) evaporation of the eluent and reconstitution of the residue in methanol, (ix) addition of water, (x) clean-up of the extract using a phenyl SPE cartridge eluted with acetone, (xi) evaporation of acetone and reconstitution of the residue in HPLC mobile phase consisting of hexane-methanol-isopropanol (90:5:5, v/v), (xii) normal-phase HPLC analysis using an amino column and UV detection at 268 nm.

The LOQ of the method was 0.02 mg/kg. Recoveries of fortified blank (untreated) samples obtained during method validation and concurrently with the analysis of treated samples are shown in Table 1. The overall average recovery was 97% with average relative standard deviation of 9.8%.

Table 1. Recoveries by analytical method AG-597B for determination of fludioxonil in apples and pears in supervised trials.

Sample	Fortification level (mg/kg)	Recovery (%)		RSD (%)	No.	Author(s) Date Study No. Syn. Archive No.
		Mean	Values			
Apples	0.021	107	100, 103, 107, 107, 109, 112	3.4	6	Thompson, Ediger 2003 IR4 07568/1751-02
	0.21	96	95, 96, 97	1.0	3	
	0.525	91	86, 92, 95	5.0	3	

Sample	Fortification level (mg/kg)	Recovery (%)		RSD (%)	No.	Author(s) Date Study No. Syn. Archive No.
		Mean	Values			
	1.05	104	97, 99, 99, 101, 102, 112	6.7	6	CGA173506/6074
	5.25	103	98, 103, 108	4.9	3	
Pears	0.02	108	97, 119		2	Starner 2003 IR4 07569/556-00 CGA173506/5896
	0.1	127	93, 102, 187	41	3	
	1.0	85	76, 83, 84, 95	9.3	4	
	2.0	92	92		1	
	5.0	82	80, 83		2	
Apples	0.02	91	91		1	Ediger 2005 T005045-05 CGA173506/6756
	10	97	97		1	
Pears	0.02	72	72		1	
	10	102	102		1	

Analytical method REM 133.04 (Mair, 1993) was employed for the determination of fludioxonil residues in samples resulting from an apple processing study (Solé, 2004). In brief, slightly modified REM 133.04 method for whole and processed apple samples involved the following steps: (i) homogenization of the sample with methanol, (ii) filtration of an aliquot and dilution with water, (iii) clean-up of the extract using a phenyl SPE cartridge eluted with acetone, (iv) dilution with saturated NaCl solution and partitioning with hexane-diethyl ether (8:2, v/v), (v) evaporation of the organic phase to dryness and reconstitution of the residue in hexane-isopropanol (9:1, v/v), (vi) normal-phase HPLC analysis using an amino column and fluorescence detection (excitation wavelength 265 nm, emission wavelength 312 nm).

The LOQ of the method was 0.02 mg/kg. Recoveries of fortified blank (untreated) samples obtained during method validation and concurrently with the analysis of treated samples are shown in Table 2. The overall average recovery was 81% (range 70–113%) with relative standard deviation of 14%.

Multiresidue methods, such as previously reported DFG S19 method (Specht *et al*., 1995; Pelz, 2001) or recently developed QuEChERS method (Anastassiades *et al*., 2003; Lehotay *et al.,* 2005a), are more suitable for routine monitoring analysis than the single-residue methods AG-597B and REM 133.04. The DFG S19 method (European standard method DIN EN 12393) involves the extraction of the sample with acetone, followed by partition with ethyl acetate-cyclohexane (1:1, v/v) and gel permeation chromatography clean-up. The final extract is analysed by capillary gas chromatography (GC), typically with a mass spectrometric (MS) detection. The QuEChERS method involves the extraction of the sample with acetonitrile and simultaneous liquid-liquid partitioning by adding NaCl and anhydrous $MgSO_4$ (a buffered version of the QuEChERS method uses acetonitrile with 1% acetic acid and sodium acetate instead of NaCl).

After centrifugation, an aliquot is transferred to a mini centrifuge tube for dispersive SPE clean-up using primary secondary amine sorbent and anhydrous $MgSO_4$. After centrifugation, the extract is ready for analysis by either GC-MS or LC-MS/MS. Recoveries in the range of 90–110% were reported for fludioxonil in fruits and vegetables (Lehotay *et al.*, 2005b).

Table 2. Recoveries by analytical method REM 133.04 for determination of fludioxonil in samples resulting from an apple processing study (Solé, 2004).

Sample	Fortification level (mg/kg)	Recovery (%)		No.
		Mean	Values	
Whole and washed apples	0.02	81		1
	0.2	76		1
	0.4	70		1
Washing water	0.02	99		1
	0.2	78		1
Wet pomace	0.02	71		1
	0.2	70		1

Sample	Fortification level (mg/kg)	Recovery (%)		No.
		Mean	Values	
	0.5	79	77, 80	2
Dry pomace	0.02	78		1
	0.2	70		1
	5	71		1
Raw juice	0.02	100		1
	0.2	77		1
Pasteurised juice	0.02	96	79, 113	2
	0.2	83		1
Sieved purée	0.02	85		1
	0.2	77		1
Purée	0.02	86		1
	0.2	76		1

Stability of pesticide residues in stored analytical samples

Fludioxonil residues were previously shown to be stable in apples (Tribolet, 2000) and a number of other commodities for at least 24 months under deep-frozen conditions (< -18°C). In the supervised trial and processing studies reported to the present Meeting, apple and pear samples were stored frozen for a maximum of 177 days (5.8 months).

USE PATTERN

Fludioxonil is registered globally as a fungicide for seed treatment, foliar treatment, and post-harvest application on a variety of crops. The Meeting received a copy of the official label providing information on registered post-harvest use of fludioxonil on pome fruit in the USA relevant to the supervised trial data. This information is summarized in Table 3. For maximum control, it is recommended to treat the fruit once before and once after storage.

Table 3. Registered post-harvest uses of fludioxonil on pome fruit in the USA.

Formulation, ai %	Application		
	Method	Dip or spray concentration (kg ai/hL)	Number
WP, 50	dip (for 30 s)/drench	0.06	2
	spray - low volume (concentrate)[1]	0.86	
	spray - high volume (dilute)	0.24	

[1]Application of 0.5 kg ai/200,000 kg fruit (2.5 mg ai/kg).

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on fludioxonil supervised post-harvest trials on apples and pears, which is summarized in Tables 4 and 5 respectively.

Table 4. Fludioxonil residues resulting from post-harvest application to apples in the USA.

Location Year (Variety)	Form	Method	Fludioxonil residue, mg/kg[6]	Author(s), Date Study No. Syngenta Archive No.
Idaho[1] 2001 (Red Spur Delicious)	WP 50	Dip treatment[2]	0.75, 0.59 (0.67)	Thompson, Ediger, 2003 IR4 07568/1751-02 CGA173506/6074
Michigan[1] 2001 (Red Delicious)	WP 50	Dip treatment[2]	0.52, 0.35 (0.44)	Thompson, Ediger, 2003 IR4 07568/1751-02 CGA173506/6074
New Jersey[1] 2001 (McIntosh)	WP 50	Dip treatment[2]	0.56, 0.50 (0.53)	Thompson, Ediger, 2003 IR4 07568/1751-02 CGA173506/6074

Location Year (Variety)	Form	Method	Fludioxonil residue, mg/kg[6]	Author(s), Date Study No. Syngenta Archive No.
California[1] 2001 (Fuji)	WP 50	Dip treatment[2]	1.1, 0.76 (0.93)	Thompson, Ediger, 2003 IR4 07568/1751-02 CGA173506/6074
	WP 50	Packing line spray[3]	1.7, 1.3 (1.5)	
	WP 50	Dip treatment followed by packing line spray[4]	2.4, 2.1 (2.2)	
Washington[1] 2001 (Red Delicious)	WP 50	Dip treatment[2]	1.1, 0.72 (0.91)	Thompson, Ediger, 2003 IR4 07568/1751-02 CGA173506/6074
	WP 50	Packing line spray[3]	0.68, 0.57 (0.62)	
	WP 50	Dip treatment followed by packing line spray[4]	2.2, 1.8 (2.0)	
Visalia, California 2005 (Golden Delicious)	WP 50	Dip treatment followed by packing line spray[5]	2.3, 2.6 (2.5)	Ediger, 2005 T005045-05 CGA173506/6756
Parlier, California 2005 (Golden Delicious)	WP 50	Dip treatment followed by packing line spray[5]	2.3, 2.4 (2.4)	Ediger, 2005 T005045-05 CGA173506/6756

[1]Study originally submitted to the 2004 JMPR.

[2]Post-harvest dip: 0.06 kg ai/hL (dip solution included carnuba packing wax), fruit dipped for 2 min (± 10 s)

[3]Packing line spray: 0.5 kg ai in low pressure/low volume post-harvest packing line spray (0.30 - 0.37 kg ai/hL in water with carnuba fruit wax) per 200,000 kg fruit

[4]Post-harvest dip: 0.06 kg ai/hL (without carnuba packing wax), fruit dipped for 2 min (± 10 s); 3 hours drying followed by packing line spray: 0.5 kg ai in low pressure/low volume post-harvest packing line spray (0.30 - 0.37 kg ai/hL in water with carnuba fruit wax) per 200,000 kg fruit

[5]Post-harvest dip: 0.06 kg ai/hL (without fruit wax), fruit dipped for 30 s; approx. 30 min drying followed by packing line spray: 0.5 kg ai in low pressure/low volume post-harvest packing line spray (water with fruit wax) per 200,000 kg fruit

[6]Results from replicate samples are on same line; average values are in parentheses; underlined values were selected for estimation of STMR and MRL

Table 5. Fludioxonil residues resulting from post-harvest application to pears in the USA.

PEARS Location Year, (Variety)	Form	Method	Fludioxonil residue, mg/kg[8]	Author(s), Date Study No. Syngenta Archive No.
Idaho[1] 2000 (D'Anjou)	WP 50	Drench treatment[2]	3.5, 2.2 (2.9)	Starner, 2003 IR4 07569/556-00 CGA173506/5896
	WP 50	Dip treatment[3]	1.4, 0.93 (1.2)	
New Jersey[1] 2000 (Bartlett)	WP 50	Drench treatment[4]	0.76, 0.71 (0.74)	Starner, 2003 IR4 07569/556-00 CGA173506/5896
	WP 50	Dip treatment[3]	1.2, 0.79 (1.0)	
California[1] 2000 (Shinko)	WP 50	Drench treatment[2]	1.6, 1.3 (1.5)	Starner, 2003 IR4 07569/556-00 CGA173506/5896
	WP 50	Dip treatment[3]	2.7, 1.6 (2.2)	
	WP 50	Packing line spray[5]	2.5, 1.4 (2.0)	
	WP 50	Drench treatment[2] followed by packing line spray[5]	2.8, 2.7 (2.8)	
Washington[1] 2000 (Anjou)	WP 50	Drench treatment[2]	1.3, 1.1 (1.2)	Starner, 2003 IR4 07569/556-00 CGA173506/5896
	WP 50	Dip treatment[3]	0.68, 0.67 (0.68)	
	WP 50	Packing line spray[6]	1.6, 1.3 (1.5)	
	WP 50	Drench treatment[2] followed by packing line spray[6]	1.6, 1.5 (1.6)	

PEARS Location Year, (Variety)	Form	Method	Fludioxonil residue, mg/kg[8]	Author(s), Date Study No. Syngenta Archive No.
Visalia, California 2005 (Bartlett)	WP 50	Dip treatment followed by packing line spray[7]	1.1, 1.1 (1.1)	Ediger, 2005 T005045-05 CGA173506/6756
Parlier, California 2005 (Bartlett)	WP 50	Dip treatment followed by packing line spray[7]	1.2, 1.1 (1.2)	Ediger, 2005 T005045-05 CGA173506/6756

[1]Study originally submitted to the 2004 JMPR.

[2]Post-harvest drench: 0.06 kg ai/hL water

[3]Post-harvest dip: 0.06 kg ai/hL water + carnuba fruit wax, fruit dipped for 30 s

[4]Post-harvest drench: 0.048 kg ai/hL water

[5]Packing line spray: 0.5 kg ai in low pressure/low volume post-harvest packing line spray (0.60 kg ai/hL in undiluted carnuba fruit wax) per 200,000 kg fruit

[6]Packing line spray: 0.57-0.58 kg ai in low pressure/low volume post-harvest packing line spray (0.34-0.35 kg ai/hL in water with carnuba fruit wax) per 200,000 kg fruit

[7]post-harvest dip: 0.06 kg ai/hL (without fruit wax), fruit dipped for 30 s; approx. 30 min drying followed by packing line spray: 0.5 kg ai in low pressure/low volume post-harvest packing line spray (water with fruit wax) per 200,000 kg fruit

[8]Results from replicate samples are on same line; average values are in parentheses; underlined values were selected for estimation of STMR and MRL.

FATE OF RESIDUES IN STORAGE AND PROCESSING

Processing

The Meeting received information on the fate of incurred residues of fludioxonil during the processing of apples into juice and purée. The sponsor noted that this information has been included for completeness because post-harvest treatment of fruit is normally reserved for high value commodities and it is therefore unlikely that treated crops will be processed.

Fludioxonil, formulated as WG 62.5 (containing 25% fludioxonil and 37.5% cyprodinil), was applied to apple trees (variety Golden Delicious) three times as a foliar treatment at an application rate of approximately 250 g ai/ha at a test location in Switzerland. The application interval was 7–8 days and the fruit was harvested seven days after the final application. The fruit was harvested by hand and washed by spraying with water. Sub-samples were taken for processing into juice and purée (see Figure 1 for a processing flow chart).

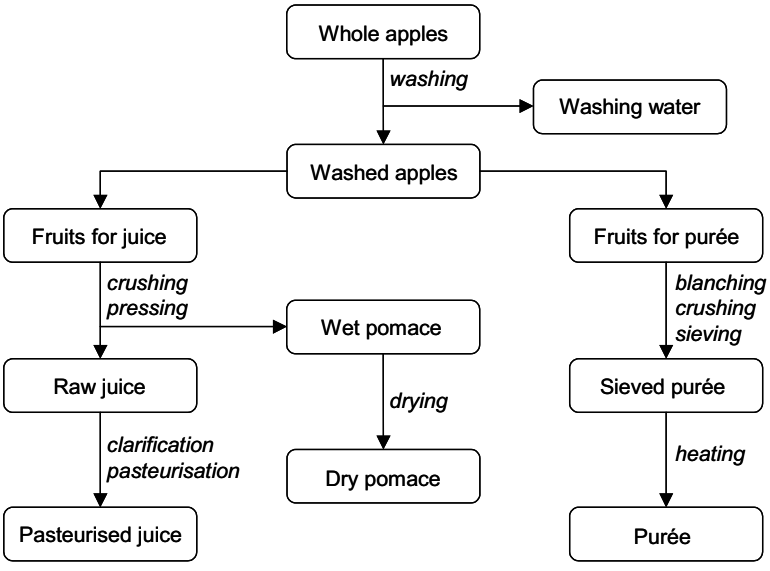

Figure 1. Flow chart for apple processing.

In apple juice processing, washed apples were crushed and pressed, generating raw juice and wet pomace. Wet pomace was dried in an oven at 60°C to yield dry pomace. Pectolytic enzymes were added to apple juice and the mixture was allowed to settle. The clear juice was racked and pasteurised by heating to 85°C for one minute.

In apple purée processing, washed apples were blanched in boiling water (2 L/kg apple) for two minutes to avoid enzymatic browning. The blanched fruits were crushed and sieved to obtain purée. After addition of sugar, the purée was reduced by heating in a double jacket saucepan to obtain a Brix degree of 24%.

Residues of fludioxonil were determined in the harvested and washed apples, wet and dry pomace, raw juice, pasteurised juice, sieved purée and purée. Results are shown in Table 6.

Table 6. Fludioxonil residues in apples and processed commodities.

APPLES Location Year (Variety)	Application[1]			PHI days	Commodity	Fludioxonil mg/kg or mg/L[2]	Processing factor	Author(s) Date Study No. Syn. Arch. No.
	Form	No.	kg ai/ha					
Switzerland 2003 (Golden Delicious)	WG 62.5	3	0.26 0.25 0.25	7	Whole apple	0.25		Solé 2004 03-0801 CGA173506/ 6057
					Washed apple	0.21	0.84	
					Washing water	0.12		
					Wet pomace	0.34	1.4	
					Dry pomace	1.25, 1.39, 1.33, 1.33 (1.33)	5.3	
					Raw juice	0.05	0.20	
					Pasteurised juice	<0.02, <0.02, <0.02, 0.02 (0.02)	0.08	
					Sieved purée	0.02, 0.02, 0.02, 0.02 (0.02)	0.08	
					Purée	0.03, 0.03, 0.03, 0.03 (0.03)	0.12	

1 Three foliar treatments: the first application at the BBCH growth stage 85, followed by the second treatment after 7 days (at BBCH 86), and then the third treatment after further 8 days (at BBCH 86-87). The fruit was harvested 7 days after the third treatment (at BBCH 87).

2 Average values are in parentheses.

APPRAISAL

Fludioxonil was first evaluated by the 2004 JMPR Meeting. The 2004 Meeting estimated an MRL of 0.7 mg/kg for foliar uses on pears, but did not recommend an MRL for pome fruit based on post-harvest use due to an insufficient number of trials performed at the maximum GAP. The present Meeting received information on the post-harvest use pattern, residue analysis, and post-harvest trials on apples and pears. Results of an apple processing study were also reported.

Methods of residue analysis

The Meeting concluded that adequate multi- and single-residue methods exist for both gathering data in supervised trials and processing studies and for the monitoring and enforcement of fludioxonil MRLs in commodities of plant origin.

Two single-residue methods (AG-597 and REM 133.04) were used for the analysis of fludioxonil in treated apples and pears and in samples resulting from an apple processing study. The LOQ of both methods was 0.02 mg/kg. In the case of method AG-597B, the overall average recovery was 97% with an average relative standard deviation of 9.8%. The analytical method REM 133.04 gave an overall average recovery of 81% with an average relative standard deviation of 14%.

Multiresidue methods, such as the previously reported method DFG S19 or recently developed QuEChERS method, are more suitable for routine monitoring of residues than the two single-residue methods AG-597B and REM 133.04.

Stability of pesticide residues in stored analytical samples

The JMPR 2004 Meeting concluded that fludioxonil residues are stable in apples and many other commodities for at least 24 months under deep freeze conditions (<-18°C). In the supervised trial and processing studies reported to the present Meeting, apple and pear samples were stored frozen for a maximum of 177 days (5.8 months).

Results of supervised trials on crops

The Meeting received supervised trial data for post-harvest treatments of pome fruit (apples and pears) conducted in the USA. Apples and pears were treated by post-harvest dip, drench, or spray using a 50% wettable powder formulation of fludioxonil. GAP for pome fruit specifies a maximum of two treatments, one on entering storage and a second on exit from storage for market distribution, at a single application rate of 0.5 kg ai/200,000 kg fruit (2.5 mg ai/kg fruit) for spray treatment (0.86 kg ai/hL for droplet-type applications using a low-volume concentrate; 0.24 kg ai/hL for high-volume jet-type sprays) and 0.06 kg ai/hL for dip/drench treatments.

Seventeen trials (seven on apples and ten on pears) were conducted as a single application at approximately the GAP rate. Eight trials (four on apples and four on pears) were conducted at the GAP rate with two sequential applications, involving 0.06 kg ai/hL dip/drench treatment followed by packing-line spray at 2.5 mg ai/kg fruit (2.85 mg ai/kg fruit, *i.e.* 114% GAP, was used in one trial on pears).

As GAP specifies two treatments, the Meeting regarded the eight trials with two sequential applications as an approximation of the maximum GAP. The residue levels on apples, in ranked order were: 2.0, 2.2, 2.4, and 2.5 mg/kg. The residue levels on pears, in ranked order were: 1.1, 1.2, 1.6, and 2.8 mg/kg (note: 1.6 mg/kg resulted from a dip treatment at 100% GAP followed by the spray treatment at 114% GAP). The Meeting decided to combine the data, thus the residue levels on pome fruit, in ranked order, were: 1.1, 1.2, 1.6, 2.0, 2.2, 2.4, 2.5, and 2.8 mg/kg. The Meeting estimated a maximum residue level for pome fruit of 5 mg/kg and an STMR of 2.1 mg/kg, and withdrew its previous recommendation for a maximum residue level of 0.7 mg/kg for pears

Fate of residues during processing

The Meeting received information on the fate of incurred residues of fludioxonil during commercial-type processing of apples into juice and purée. The processing factors and STMR-P values, based on an STMR of 2.1 mg/kg for pome fruits, are summarized in the table below.

Raw agricultural commodity	Processed commodity		
	Commodity	Processing factor	STMR-P (mg/kg)
Apple	Washed fruit	0.84	
	Juice, pasteurised	0.08	0.17
	Pomace, wet	1.4	2.9
	Pomace, dry	5.3	11
	Purée	0.12	0.25

The Meeting estimated a maximum residue level of 20 mg/kg for apple pomace, dry, based on the highest residue of 2.8 mg/kg in the pome fruit post harvest trials and the processing factor of 5.3.

Farm animal dietary burden

The Meeting estimated the maximum dietary burden of fludioxonil residues for farm animals (beef cattle, dairy cows, and poultry) using previously recommended MRLs and STMR-Ps for possible feed commodities and STMR-P for wet apple pomace estimated by the present Meeting. The table below shows the basis for the dietary intake calculation.

Commodity	Group	Maximum or highest residue level (mg/kg)	STMR or STMR-P	Dry matter (%)	Residue on dry wt (mg/kg)	*Dietary content (%)*			Residue contribution (mg/kg)		
						Beef cattle	*Dairy cows*	*Poultry*	Beef cattle	Dairy cows	Poultry
Apple pomace (wet)	AB		2.9	40	7.3	*40*	*20*		2.9	1.5	
Wheat forage	AF	0.05		25	0.20	*25*	*60*		0.05	0.12	
Rape forage	AM	0.05		30	0.17	*30*	*20*		0.05	0.03	
Maize grain	GC	0.05		88	0.06			*80*			0.05
Pea seed	VD	0.07		90	0.08	*5*		*20*	0.004		0.02
Total						***100***	***100***	***100***	**3.0**	**1.7**	**0.07**

The maximum dietary burdens of fludioxonil in beef cattle, dairy cows, and poultry (on the basis of diets listed in Appendix IX of the *FAO Manual*) are 3.0, 1.7, and 0.07 mg/kg, respectively. For comparison, the previously calculated dietary burdens were 0.07, 0.06, and 0.07 mg/kg, respectively (JMPR Report 2004).

Farm animal feeding studies

The 2004 Meeting received information on a ruminant feeding study, the results of which are summarized in the tables below. No study was available on poultry feeding.

Residues of fludioxonil and its metabolites (converted via oxidation to 2,2-difluoro-1,3-benzodioxole-4-carboxylic acid), found in milk were:

Animal number	Dose level in diet	Residues (mg/kg) at dosing (day)						
		0 (pre-dosing)	1	3	7	14	21	26
2A	1x	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
2B	0.55 mg/kg	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
2C		< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
3A	3x	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
3B	1.6 mg/kg	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
3C		< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
4A	10x	< 0.01	< 0.01	< 0.01	< 0.01	**0.019**	**0.012**	< 0.01
4B	5.5 mg/kg	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
4C		< 0.01	< 0.01	**0.016**	**0.011**	**0.010**	**0.014**	< 0.01

Residues of fludioxonil and its metabolites (converted via oxidation to 2,2-difluoro-1,3-benzodioxole-4-carboxylic acid) found in ruminant tissues were:

Animal number	Dose level in diet	Residues (mg/kg) at dosing (day)					
		Round muscle	Tenderloin muscle	Liver	Kidney	Perirenal fat	Omental fat
2A	1x	na	na	na	na	na	na
2B	0.55 mg/kg	na	na	na	na	na	na
2C		na	na	na	na	na	na

Animal number	Dose level in diet	Residues (mg/kg) at dosing (day)					
		Round muscle	Tenderloin muscle	Liver	Kidney	Perirenal fat	Omental fat
3A	3x	na	na	na	na	na	na
3B	1.6 mg/kg	na	na	na	na	na	na
3C		na	na	na	na	na	na
4A	10x	< 0.01	< 0.01	< 0.05	< 0.05	< 0.05	< 0.05
4B	5.5 mg/kg	< 0.01	< 0.01	< 0.05	< 0.05	< 0.05	< 0.05
4C		< 0.01	< 0.01	< 0.05	< 0.05	< 0.05	< 0.05

Animal commodity maximum residue levels

The addition of wet apple pomace to the list of possible feed items resulted in the estimated maximum dietary burden of 3.0, 1.7, and 0.07 mg/kg for beef cattle, dairy cows, and poultry, respectively.

Based on the information in Appendix IX of the *FAO Manual*, apple pomace is not a significant part of a poultry diet, thus the addition of this feed item did not change the previous estimation of maximum dietary burden and MRLs. The 2004 Meeting recommended MRLs of 0.01 (*) mg/kg for poultry meat and 0.05 (*) mg/kg for eggs and poultry offal. STMR values of 0 mg/kg were estimated for eggs, poultry meat, and poultry offal.

In the feeding study reported to the 2004 Meeting, no quantifiable residue of fludioxonil was found in the tissues of ruminants at the 5.5 mg/kg feeding level, which corresponds to 3.2-fold and 1.7-fold higher levels than the estimated maximum dietary burdens for dairy cows and beef cattle, respectively. Thus, the addition of wet apple pomace to the list of possible feed items did not change the recommendation of the 2004 Meeting.

The present Meeting confirmed the previous recommendations for a maximum residue level of 0.05* for edible offal and 0.01 (*) mg/kg for muscle and the STMR values of 0 mg/kg for both edible offal and muscle.

In milk, the highest residue level found was 0.019 mg/kg at the 5.5 mg/kg feeding level. Using this information and extrapolating to a 1.7 mg/kg feeding level (corresponding to the maximum dietary burden for dairy cows), the highest residues expected in milk would be below the reported LOQ of 0.01 mg/kg. This estimation is also supported by the results of the 1.6 mg/kg feeding study (a close approximation of the maximum dietary burden for dairy cows), which led to no quantifiable fludioxonil residues (< 0.01 mg/kg) in milk. This reaffirms the 2004 JMPR recommendation of the MRL of fludioxonil residue at the LOQ, 0.01 (*) mg/kg, and the STMR value for milk of 0 mg/kg.

Definition of the residue for compliance with MRLs and estimation of dietary intake in plant commodities: fludioxonil.

Definition of the residue for compliance with MRLs and estimation of dietary intake in livestock commodities: fludioxonil and metabolites determined as 2,2-difluoro-1,3-benzodioxole-4-carboxylic acid and calculated as fludioxonil. Fludioxonil is fat-soluble.

RECOMMENDATIONS

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

Definition of the residue for compliance with MRLs and estimation of dietary intake in plant commodities: fludioxonil.

Definition of the residue for compliance with MRLs and estimation of dietary intake in livestock commodities: fludioxonil and metabolites determined as 2,2-difluoro-1,3-benzodioxole-4-carboxylic acid and calculated as fludioxonil. Fludioxonil is fat-soluble.

Commodity		MRL (mg/kg)		STMR or STMR-P (mg/kg)
CCN	Name	New	Previous	
JF 226	Apple juice			0.17
AB 226	Apple pomace, dry	20		11
	Apple purée			0.25
FP 0230	Pear	W	0.7	
FP 0009	Pome fruits	5 Po		2.1

DIETARY RISK ASSESSMENT

Long-term intake

The IEDIs of fludioxonil based on STMR and STMR-P values estimated for 47 commodities for the thirteen GEMS/Food regional diets were 0–2% of the ADI (Annex 3 of the 2006 Report). A similar result was obtained in 2004, when the Meeting concluded that the long-term dietary intake of fludioxonil residues is unlikely to present a public health concern.

Short-term intake

The 2004 Meeting decided that an ARfD for fludioxonil is unnecessary and concluded that the short-term dietary intake of fludioxonil residues is unlikely to present a public health concern.

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Williams, R.K.	CGA173506/1123	1998

IMIDACLOPRID (206)

First draft was prepared by Arpad Ambrus, Hungary

EXPLANATION

Imidacloprid was evaluated by the JMPR in 2001 and 2002 when an ADI of 0-0.06 mg/kg bw/day and an ARfD of 0.4 mg/kg bw/day were established, and a number of maximum residue levels were estimated, respectively.

The cranberry industry performed a number of supervised trials within the Interregional Research Project No. 4 to provide data for the establishment of US tolerances for imidacloprid residues in cranberry. The relevant labels and reports of supervised trials were submitted for evaluation by the 2006 JMPR.

RESIDUE ANALYSIS

Analytical methods

Samples were analyzed for combined residues of imidacloprid and metabolites M09 (WAK 4140), M06 (WAK 3745), M01 (WAK 4103), and M14 (6-CNA) (Bayer Method 00200). The limit of quantitation (LOQ) was 0.05 mg/kg. No quantifiable residues were observed in the control samples. The concurrent recovery obtained during the analysis of samples was 102% with a CV of 7% and range of 89–114% (n=15).

Stability of pesticide residues in stored analytical samples

The maximum storage interval for field-treated samples in this study was 683 days. To evaluate storage stability, control samples were fortified with imidacloprid, WAK 4140, WAK 3745, WAK 4103, and 6-CNA to a level equivalent to 0.50 mg/kg imidacloprid and analyzed after 655 or 656 days of frozen storage. The results, shown in Table 1, indicate that the residues were stable during the long storage period.

Table 1. Storage stability study performed in conjunction with field trial samples.

Commodity	Cranberry
Duration	655 days (imidacloprid and WAK 4140) 656 days (WAK 3745, WAK 4103, and 6-CNA)
Temperature	-20 ± 5 °C
Percentage of residues measured in stored samples	83, 90, and 92% of 0.50 mg/kg imidacloprid applied 87, 93, and 90% of 0.50 mg/kg WAK 4140 applied[1] 97, 86, and 88% of 0.50 mg/kg WAK 3745 applied[1] 93, 99, and 95% of 0.50 mg/kg WAK 4103 applied[1] 89, 98, and 97% of 0.50 mg/kg 6-CNA applied[1]

Note: 1: Expressed in parent imidacloprid equivalent

USE PATTERN

In the USA, Admire 2 Fl, containing 21.4% active substance, can be applied to protect cranberry as shown in Table 2:

Table 2. Registered uses of imidacloprid

Application					PHI
Method	No	Interval, days	Water L/ha	Rate kg ai/ha	day
Ground[1]	Max 2	NS[3]	Min 187	0.28	30
Chemigation	Max 2	NS[3]	5600-9350	0.28-0.56[2]	30

1. As a soil spray directed to the root or crown area using a minimum of 187 litre water/ha.
2. Maximum dose per season
3. NS: not specified

RESIDUES RESULTING FROM SUPERVISED TRIALS

Field trials were conducted in four geographical regions of the USA (Massachusetts, New Jersey, Wisconsin, and Oregon) (Dorschner, 2000). Each of the five field trial sites consisted of one untreated control plot and two treated plots. Each treated plot received one foliar application of the test substance at a rate of approximately 0.56 kg ai/ha immediately followed by irrigation to move the test substance into the soil. Mature cranberries were harvested from one of the treated plots at each site approximately 30 days following the application. Samples from the other treated plot at each site were harvested approximately 45 days following the application.

Samples of about 1 kg were taken in duplicate from each site. They were shipped deep-frozen to the testing laboratory.

Cranberries were analyzed for combined residues of imidacloprid and metabolites WAK 4140, WAK 3745, WAK 4103, and 6-chloronicotinic acid (6-CNA). Table 3 summarizes the trial conditions and the residues detected.

No quantifiable residues (> 0.05 mg/kg) were observed in any of the samples (Table 3).

Table 3. Summary of combined imidacloprid residues in/on cranberry following a single ground treatment with maximum dosage rate of 0.56 kg ai/ha.

Field Trial Location (City, State)	Spray Volume (GPA)	PHI (days)	Residue (mg/kg)
East Wareham, MA	744	30	< 0.05
(97-MA02)	763	46	< 0.05
Tabernacle, NJ	482	28	< 0.05
(97-NJ27)	618	43	< 0.05
Wisconsin Rapids, WI	225	28	< 0.05
(97-WI16)	226	43	< 0.05
Biron, WI	225	28	< 0.05
(97-WI17)	226	43	< 0.05
Bandon, OR	674	32	< 0.05[1]
(97-OR18)	713	45	< 0.05

1. Trace amounts of residues below the LOQ were detected in one of the samples

APPRAISAL

Imidacloprid was evaluated by the JMPR in 2001 and 2002 when an ADI of 0-0.06 mg/kg bw/day and an ARfD of 0.4 mg/kg bw/day were established, and a number of maximum residue levels were estimated. The residues were defined as the sum of imidacloprid and its metabolites containing the 6-chloropyridinyl moiety for both regulatory and dietary intake assessment purposes.

[1] Trace amounts below the LOQ were detected in one of the treated samples.

Results of supervised trials carried out on cranberry according the US registered uses, were submitted for evaluation.

Results of supervised trials on crops

Five field trials were conducted with foliar applications at the maximum recommended rate (0.56 kg ai/ha). Mature cranberries were harvested at the recommended PHI (30 days) and 45 days post treatment.

The individual residue components were found to be stable under deep freeze conditions (< -20 ^{o}C) for 655−656 days. The longest storage period of samples corresponded with the test period and confirmed the validity of residue data obtained.

Samples were analyzed for combined residues of imidacloprid and metabolites M09 (WAK 4140), M06 (WAK 3745), M01 (WAK 4103), and 6-CNA with a total residue method with average concurrent recovery of 102% obtained during the analysis of samples. The limit of quantification (LOQ) was 0.05 mg/kg. No quantifiable residues were observed in the samples (< 0.05 mg/kg).

Based on the results of these trials < 0.05 (5) mg/kg the Meeting estimated a maximum residue level of 0.05* mg/kg and values for STMR and HR of 0.05 mg/kg.

RECOMMENDATION

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

Summary of recommendations for MRLs, STMRs and HRs for imidacloprid

CCN	Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR/P mg/kg
		New	Previous		
FB 0265	Cranberry	0.05*		0.05	0.05

DIETARY RISK ASSESSMENT

Long-term intake

The GEMS/Food regional diets specify the following long-term cranberry consumption (g/day/person) for various cluster diets: A (0.1); D (0.3); F (0.6); M (2.5). The cranberry consumption in the other regions is nil.

The highest IEDI in the 13 GEMS/Food regional diets, based on estimated STMR was < 0.01% of the maximum ADI (0.06 mg/kg bw).

The Meeting concluded that the long-term dietary intake of imidacloprid residues from use on cranberry will add only marginally to the intake of residues from other uses considered by an earlier JMPR.

Short-term intake

The GEMS/Food regional diet specifies large portion sizes of cranberry as 3.53 g/kg bw for adults and 6.78 g/kg bw for children (both are from the USA).

The IESTIs of imidacloprid calculated on the basis of the large portion size and the estimated HR of 0.05 mg/kg are 0.04% and 0.1% of the ARfD for adults and children, respectively.

The Meeting concluded that the short-term intake of residues of imidacloprid resulting, from the use on cranberry that have been considered by the JMPR, is unlikely to present a public health concern.

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METHOXYFENOZIDE (209)

First draft was prepared by Arpad Ambrus, Hungary

EXPLANATION

Methoxyfenozide was evaluated by the JMPR in 2003 when an ADI of 0-0.1 mg/kg bw and ARfD of 0.9 mg/kg bw were established, and a number of maximum residue levels were estimated.

The cranberry industry performed a number of supervised trials within the Interregional Research Project No. 4 to provide data for the establishment of US tolerances for methoxyfenozide residues in cranberry. The relevant labels and reports of supervised trials were submitted for evaluation by the 2006 JMPR.

RESIDUE ANALYSIS

Analytical methods

The harvested fruit samples were analyzed by the method detecting the parent compound (Stein and Wu 1998, Report No. 34-98-87). The limit of quantification for methoxyfenozide in cranberries was 0.01 mg/kg.

No quantifiable residues were observed in the control samples. The recoveries obtained during the analysis of samples fortified between 0.01 and 1 mg/kg ranged from 76% to 109% with an average of 87% (n=17) and coefficient of variation of 15%.

Stability of pesticide residues in stored analytical samples

The maximum storage interval for field-treated samples was 115 days. To evaluate storage stability, control samples were fortified with 1.0 mg/kg methoxyfenozide, stored at < -15°C and analyzed after 109 days of frozen storage. The average residues that survived in fortified samples (79%, n=3) were not significantly different (α= 0.01) from the analytical average recovery (87%).

The 2003 JMPR reported the results of numerous studies on various plant commodities that indicated that methoxyfenozide residues were stable for more than 6 months (FAO 2003).

USE PATTERN

The Intrepid 2F containing 22.6% methoxyfenozide is registered to control various moths and worms in cranberry.

It can be applied to protect cranberry as shown in Table 1.

Table 1. Use pattern of Methoxyfenozide on cranberry.

Application					PHI
Method	No	Interval, days	Water l/ha	Rate kg ai/ha	day
Ground, aerial	Max 4	10-18	Min 40[1]-80	0.18-0.28[1]	14
Chemigation	Max 2	NS	Max 3400	0.28-0.56[2]	30

1 For aerial application and young or small plants.

2 Do not exceed 1.12 kg ai/ha/season.

3 NS: not specified

RESIDUES RESULTING FROM SUPERVISED TRIALS

During the 1999 growing season six trials were conducted on cranberries in four geographical regions of the USA and British Columbia, Canada (Dorschner, 2002).

The cranberry crops were grown and maintained according to typical agricultural practices for each geographical region.

Each treated plot received four foliar broadcast applications of the test substance at a rate of approximately 0.28 kg ai/ha each, for a total of approximately 1.12 kg ai/ha. All applications were made 7 to 11 days apart.

Two replicate samples of mature or nearly mature cranberry fruit were collected by hand at 13 to 15 days following the last application. The sampling positions were selected randomly within the control and treated plots, and the fruits were placed in plastic bags.

The residue results from the supervised field trials are presented in Table 2.

Table 2. Residues of methoxyfenozide in/on cranberry fruits following four foliar broadcast applications.

Field Trial Location (City, State)	Spray Volume (l/ha)	Days between Appl.	Dosage kg ai/ha	PHI (days)	Residue mg/kg[1]	
					Replicate samples	Average
East Wareham, MA (99-MA01)	703–768	10–11	0.26–0.28[2]	14	0.028–<u>0.035</u>	0.03
Tabernacle, NJ (99-NJ14)	323	9–11	0.28–0.49[3]	14	0.206–0.262	0.23
Wisconsin Rapids, WI (99-WI09)	284-318	9–10	0.28 (1X)	13	0.088–<u>0.104</u>	0.10
Biron, WI (99-WI10)	285–306	9–10	0.28 (1X)	13	0.064–<u>0.076</u>	0.07
Bandon, OR (99-OR13)	319–348	9–11	0.28 (1X)	15	<u>0.374</u>–0.407	0.39
Delta, BC (99-BC04)	365–382	7–11	0.28 (1X)	14	0.138–<u>0.160</u>	0.15

1 The LOQ was 0.01 mg/kg for cranberries.

2 The third application was under-applied by approximately 7%. The total amount of test substance applied, 1.09 kg ai/ha, was within -5 to +10% of the protocol rate of 1.12 kg ai/ha.

3 The fourth application was over-applied by approximately 75%, thus the seasonal maximum rate was exceeded by 20%.

APPRAISAL

Methoxyfenozide was evaluated by the JMPR in 2003 and an ADI of 0-0.1 mg/kg bw/day and an ARfD of 0.9 mg/kg bw/day were established, and a number of maximum residue levels were estimated. The JMPR defined the residues as parent compound for compliance with MRLs and for dietary intake estimations.

Results of supervised trials carried out on cranberry according the US registered uses were submitted for evaluation.

Results of supervised residue trials on crops

Six trials were conducted on cranberries in four geographical regions of the USA and British Columbia, Canada. Each treated plot received four foliar broadcast applications of the test substance approximately according to GAP. Two replicate samples of mature or nearly mature cranberry fruit were collected around the registered PHI.

The harvested fruit samples were analyzed by a method determining the parent compound alone. The method was validated at 0.01 mg/kg level. The average recovery of 87% was obtained in

the residue range of 0.01 and 1 mg/kg. Stability of residues (< -15°C) was tested in three samples indicating that the residues were stable during the storage period.

The residues of parent methoxyfenozide found in fruit treated according to GAP in rank order were: 0.03, 0.03, 0.07, 0.10, 0.15 and 0.39 mg/kg.

The Meeting estimated a maximum residue level of 0.7 mg/kg, HR of 0.39 mg/kg and STMR of 0.085 mg/kg.

RECOMMENDATION

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

Summary of recommendations for MRLs, STMRs and HRs for methoxyfenozide

CCN	Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR/P mg/kg
		New	Previous		
FB 0265	Cranberry	0.7		0.085	0.39

DIETARY RISK ASSESSMENT

Long-term intake

The GEMS/Food Consumption Cluster Diets specifies the following long-term cranberry consumption (g/day/person) for various cluster diets: A (0.1); D (0.3); F (0.6); M (2.5). The cranberry consumption in the other regions is nil.

The highest IEDI in the 13 GEMS/Food regional diets based on estimated STMR was < 0.01% of the maximum ADI (0.1 mg/kg bw).

The Meeting concluded that the long-term intake of residues of methoxyfenozide use on cranberry will not practically increase the intake of residues from other uses considered earlier by the JMPR.

Short-term intake

The GEMS/Food regional diet specifies the large portion size for cranberry of 3.53 g/kg bw for adults and 6.78 g/kg bw for children (both are from the USA).

The IESTIs of methoxyfenozide calculated on the basis of the large portion size and the estimated HR of 0.39 mg/kg are 0.15% and 0.3% of the ARfD for adults and children, respectively.

The Meeting concluded that the short-term intake of residues of methoxyfenozide resulting from the use on cranberry that has been considered by the JMPR is unlikely to present a public health concern.

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R. Stein and S. Wu. Tolerance enforcement method for parent RH-2485 in pome fruit, Rohm and Haas Co. TR-34-98-87

PIRIMICARB (101)

First draft prepared by T. van der Velde-Koerts and B.C. Ossendorp , The Netherlands and David Lunn, New Zealand

EXPLANATION

Residue and analytical aspects of pirimicarb were evaluated by the JMPR in 1976, 1978, 1979, 1981 and 1985. The compound was listed in the Periodic Re-Evaluation Program at the 37th Session of the CCPR for periodic review by 2006 JMPR. The toxicological review was conducted in 2004, when an ADI of 0-0.02 mg/kg bw and an ARfD of 0.1 mg/kg bw were established.

The company submitted a full data package including residue trial data on: pome fruit, peach, apricot and nectarine, plum, barley, cherries, red currants, black currants, gooseberries, and other small fruits and berries, raspberries and blackberries, strawberries, carrots, parsley roots, salsify, parsnip, horseradish, sugar beet, beetroot, swedes, turnip, fodder beet, bulb onion, garlic, shallots, tomatoes, aubergines, peppers, cucurbits, sweet corn , brassica , lettuce, legumes, pulses, artichoke (globe), asparagus, oil seeds, grains, sorghum, triticale and potato.

Residue and GAP information was also submitted by Australia.

IDENTITY

ISO common name: pirimicarb

Chemical name

IUPAC: 2-dimethylamino-5,6-dimethylpyrimidin-4-yl dimethylcarbamate

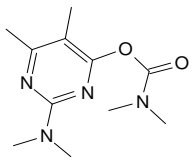
CA: 2-(dimethylamino)-5,6-dimethyl-4-pirimidinyl dimethylcarbamate

CAS Registry No: 23103-98-2

CIPAC No: 231

Synonyms and trade names: dimethyl-carbamic acid 2-dimethylamino-5,6-dimethyl-pyrimidin-4-yl ester
carbamic acid, dimethyl-, 2-(dimethylamino)-5,6-dimethyl-4-pyrimidinyl ester
PP062 (formerly ASF549)

Structural formula: confirmed by UV-VIS, IR, MS (EI), ^{1}H-NMR and ^{13}C-NMR (Wollerton, 1994: PP62/0020)



Molecular formula: $C_{11}H_{18}N_4O_2$

Molecular weight: 238.3

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient:

Property	Result	References	Guidelines/method
Minimum purity:	98%	Simmons, 2006	-
Appearance:	At 25 °C, purity 98.8% (w/w): white powdery solid, no characteristic odour	Wollerton and Husband, 1994: PP62/0020	none
Vapour pressure:	Purity 98.8% (w/w): 0.43 mPa at 20 °C (extrapolated)	Wollerton and Husband, 1994:	OECD 104 gas saturation

	0.94 mPa at 25 °C (interpolated)	PP62/0020	method
Melting/freezing point:	Purity 98.8% (w/w): 91.6 °C	Wollerton and Husband, 1994: PP62/0020	OECD 102 EC method A1 capillary method
Octanol/water partition coefficient:	At 20 °C, purity 98.2% (w/w): log K_{ow} = 1.1 at pH 3.9 log K_{ow} = 1.7 at pH 7.1 log K_{ow} = 1.7 at pH 10 .	Wollerton and Husband, 1994: PP62/0020 Simmons, 2006	OECD 107 EC method A8 shake flask method
Solubility in water:	At 20 °C, purity 98.2% (w/w): 3.0 mg/L in water 3.6 mg/L at pH 5.2 (buffer not specified) 3.1 mg/L at pH 7.4 (buffer not specified) 3.1 mg/L at pH 9.3 (buffer not specified) Validation data for GC-FID analytical method are not available	Wollerton and Husband, 1994: PP62/0020	OECD 105 EC method A6 flask method
Solubility in organic solvents:	At 20 °C: purity 97.3% (technical material) > 200 g/L in acetone > 200 g/L in dichloromethane > 200 g/L in ethyl acetate > 200 g/L in acetonitrile > 200 g/L in methanol 75 g/L in octanol 6 g/L in heptane	Wollerton and Husband, 1994: PP62/0020	OECD 105 flask method
Relative density:	At 25 °C, purity 98.8% (w/w): 1.18 g/cm^3	Wollerton and Husband, 1994: PP62/0020	OECD 109 EC method A3 pycnometer method
Hydrolysis	1.0 mg/L solution in buffered water hydrolytically stable (< 5% degradation) at pH 5, 7, 9 at 25 °C for up to 30 d	Huynh and Mathis: PP62/0915	-
Photolysis	1.0 mg/L solution in buffered water DT_{50} = 3.20 hrs at pH 5 at 25 °C DT_{50} = 2.28 hrs at pH 7 at 25 °C	Hamlet, 1997: PP62/0916	EPA, N 161-3 SETAC, section 2
Dissociation constant:	pK_a for pirimicarb-H^+ = 4.44 at 20 °C: pK_b for pirimicarb = 9.56 at 20 °C:	Wollerton and Husband, 1994: PP62/0020	OECD 112 spectrophotometric method
			-

Technical material:

PROPERTY	Result	References	Guidelines/methods
Minimum purity	97%	Simmons, 2006	-
Main impurities:	no data available	-	-
Appearance:	At 25 °C, purity 97.3% (w/w): cream powdery solid, no characteristic odour	Wollerton and Husband, 1994: PP62/0019	none
Relative density:	At 25 °C, purity 97.3% (w/w): 1.21 g/cm^3	Wollerton and Husband, 1994: PP62/0019	OECD 109 EC method A3 pycnometer method
Melting range:	Purity 97.3% (w/w): 87.3-90.7 °C	Wollerton and Husband, 1994: PP62/0019	OECD 102 EC method A1 capillary method
Stability:			
	Purity 97.3% (w/w): Chemically stable for 14 d at 54 °C and at least 22 months at ambient temperature (15-25 °C).	Wollerton and Husband, 1994: PP62/0019	CIPAC MT 46

FOMULATIONS

Pirimicarb is available as water dispersible granule formulations with active ingredient content of 17.5, 25.0 or 50% (w/w) or in a smoke generator formulation with active ingredient content of 10% (w/w). Pirimicarb is also available as an emulsifiable concentrate formulation in combination with lambda-cyhalothrin, containing 100 g/L pirimicarb and 5 g/L lambda-cyhalothrin.

FAO specifications for technical and formulated pirimicarb are not available.

Table 1. List of reference compounds used in various study reports.

Codes	Number	Trivial and systematic chemical names	Found in
Carbamates			
062/01 I	R32062	parent, pirimicarb 2-dimethylamino-5,6-dimethylpyrimidin-4-yl dimethylcarbamate; 5,6-dimethyl-2-dimethylamino-pyrimidin-4-yl dimethylcarbamate	A L P W g⁻ h⁻ R
062/03 IV	R35140	2-amino-5,6-dimethylpyrimidin-4-yl dimethylcarbamate	A L p⁻ w⁻ g⁻ h⁻ R
062/04 II	R34885	desmethyl formamido pyrimicarb 5,6-dimethyl-2-methylformamidopyrimidin-4-yl dimethylcarbamate	A L P W g⁻ h⁻ R
062/05 XV	R238359	2-dimethylamino-5-hydroxymethyl-6-methylpyrimidin-4-yl dimethylcarbamate	A l⁻ p⁻ w⁻ g⁻ h⁻
062/08 XVI	R238177	2-dimethylamino-6-hydroxymethyl-5-methylpyrimidin-4-yl dimethylcarbamate	A L p⁻ W g⁻ h⁻
836/01 III	R34836	desmethyl pirimicarb 5,6-dimethyl-2-methylaminopyrimidin-4-yl dimethylcarbamate	A L P W g⁻ h⁻ R
Hydroxypyrimidines			
062/06 V	R31805	5,6-dimethyl-2-dimethylamino-4-hydroxypyrimidine	A L P W G H R
062/07 VI	R34865	5,6-dimethyl-2-methylamino-4-hydroxypyrimidine; 5,6-dimethyl-4-hydroxy-2-methylaminopyrimidine	A L p⁻ w⁻ G H R
062/14 VII	R31680	2-amino-5,6-dimethyl-4-hydroxypyrimidine	a⁻ (L) p⁻ w⁻ G H R
062/15 XVII	R404094	5-hydroxymethyl-2-dimethylamino-6-methylpyrimidin-4-ol; 2-dimethylamino-5-hydroxymethyl-6-methyl-4-hydroxypyrimidin	A (L) p⁻ w⁻ g⁻ h⁻
062/16 XVIII	R404137	6-hydroxymethyl-2-dimethylamino-5-methylpyrimidin-4-ol; 2-dimethylamino-6-hydroxymethyl-5-methyl-4-hydroxypyrimidin	A l⁻ p⁻ w⁻ g⁻ h⁻
062/17	R407392	2-(N-methyl-formamido)-5,6-dimethylpyrimidin-4-ol	a⁻ (L) p⁻
062/18 XIX	R406405	5-hydroxymethyl-2-methylamino-6-methylpyrimidin-4-ol; 5-hydroxymethyl-6-methyl-2-methylamino-4-hydroxypyrimidin	a⁻ (L) p⁻ w⁻ G h⁻
062/19 XX	R407135	2-amino-5-hydroxymethyl-6-methylpyrimidin-4-ol; 2-amino-5-hydroxymethyl-6-methyl-4-hydroxypyrimidin	a⁻ p⁻ w⁻ g⁻ h⁻
062/20 XXII	R409239	2-amino-6-hydroxymethyl-5-methylpyrimidin-4-ol; 2-amino-6-hydroxymethyl-5-methyl-4-hydroxypyrimidin	a⁻ p⁻ w⁻ g⁻ h⁻
062/21 XXI	R409238	6-hydroxymethyl-2-methylamino-5-methylpyrimidin-4-ol; 2-methylamino-6-hydroxymethyl-5-methyl-4-hydroxypyrimidin	a⁻ (L) p⁻ w⁻ g⁻ h⁻
062/22 XXIII	R409464	Glucose conjugate of 062/06 2-dimethylamino-5,6-dimethyl-4-(beta-D-glycos-6-yl)pyrimidine	a⁻ g⁻ h⁻
062/23 XXIV	R4715	2-amino-6-hydroxy-4-methylpyrimidine-5-carboxylic acid; 2-amino-5-carboxy-6-methyl-4-hydroxypyrimidine	a⁻ p⁻ g⁻ h⁻
062/25 XXV	R59480	2-amino-6-hydroxypyrimidine-4-carboxylic acid; 2-amino-6-carboxy-4-hydroxypyrimidin	a⁻ p⁻ g⁻ h⁻
062/26	R99366	2,4-dihydroxy-5,6-dimethylpyrimidine	p⁻
062/31	R413303	Glutathione conjugate of 062/06 (R)-3-aza-5-glutamino-4-oxo-6-(2-dimethylamino-5,6-dimethylpyrimidin-4-ylthio)hexanoic acid	a⁻
062/32	R35251	2-dimethylamino-4-hydroxy-6-methylpyrimidin-5-al	a⁻
062/33	R414656	2-dimethylamino-6-hydroxy-5-methylpyrimidin-4-al	a⁻
062/34	R414657	2-dimethylamino-6-hydroxy-5-methylpyrimidin-4-carboxylic acid	a⁻
XI	-	2-amino-4-hydroxy-6-methylpyridin	g⁻ h⁻

Codes	Number	Trivial and systematic chemical names	Found in
XII	-	2-amino-4,6-dihydroxypyrimidin	g^- h^-
XIII	-	2,4-dihydroxy-6-methylpyrimidin	g^- h^-
XIV	-	2,4-dihydroxy-5,6-dimethylpyrimidin	g^- h^-
Guanidines			
062/09 X	R12378	guanidine (sulphate)	a^- p^- W g^- h^- R
062/10 VIII	R16210	N,N-dimethylguanidine (sulphate)	A P W g^- h^- r^-
062/11 IX	R16192	N-methylguanidine (hydrochloride)	a^- P W g^- h^- r^-
062/12	-	1,1-dimethylurea	p^-
062/13	-	1-methylurea	p^-
062/27	R16229	1-acetylguanidine	a^- p^-
062/28	R32379	1-acetyl-3,3-dimethylguanidine	a^- p^- w^-
062/29	R411934	2,3-diacetyl-1,1-dimethylguanidine	a^- p^-
062/30	R411893	3,3-dimethyl-1-(2-oxopropionyl)guanidine	a^-
Others			
-	-	urea	A

X metabolite used as reference compound in metabolism study and found in apple (A), lettuce (L), potato (P), wheat (W), goat (G), hen (H) or confined rotational crop study (R).

(X) metabolite used as reference compound in metabolism study and tentatively identified (one system only) in apple (A), lettuce (L), potato (P), wheat (W), goat (G), hen (H) or confined rotational crop study (R).

x- metabolite used as reference compound in metabolism study but not found in apple (a-), lettuce (l-), potato (p-), wheat (w-), goat (g-), hen (h-), rotational crops (r-)

METABOLISM AND ENVIRONMENTAL FATE

Animal metabolism

The Meeting received information on the fate of orally dosed pirimicarb in the lactating goat and in laying hens. Experiments were carried out with pirimicarb ^{14}C labelled at the pyrimidinyl-2 position (see Figure 1). Metabolism in laboratory animals (rats) was summarized and evaluated by the WHO panel of the JMPR in 2004.

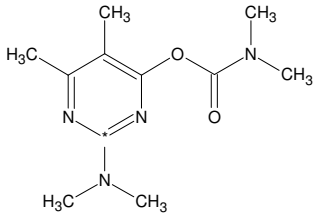


Figure 1. Label position (*) in ^{14}C labelled pirimicarb used in metabolism studies.

Study 1. One non-pregnant lactating Alpine goat was dosed twice daily for five consecutive days with ^{14}C-labelled pirimicarb (see Figure 1, Murray *et al.*, 1998: PP62/0531). The goat was 4 years of age and the average bodyweight during the treatment period was 42.0 kg. The radiopurity was > 95% (w/w). Pirimicarb was administered via gelatin capsules via a balling gun. The actual dosing rate based on the observed daily feed consumption (1.646 kg) was 17.2 ppm, equivalent to 0.68 mg ai/kg bw/d. During the treatment period, daily collections of milk, faeces and urine were made. The treated goat was sacrificed approximately 21 hrs after the last dose. Blood, liver, kidney, composite skeletal muscle, and composite fat (subcutaneous, renal and visceral) were collected from the carcass. Samples were stored at -20 °C for up to 37 months until first extraction.

Homogenised samples were analysed for total ^{14}Clevels by combustion LSC or by extraction and (combustion) LSC. The overall recovery was 77.3% TAR. The largest amount of radioactivity was found in the urine and faeces, which contained 62.9% TAR and 11.4% TAR, respectively. The edible tissues, (liver, kidney, muscle and fat) contained 1.3% TAR, while milk contained 0.29% TAR. The goat was monitored for expired ^{14}C-volatiles, but none were found.

The total residue in the pm milk was higher than the residue in the am milk, indicating that a plateau was reached within 24 hrs after dosing. Residue levels in milk during the dosing period were on average 0.043 mg/kg eq. Residue levels in the pm milk were on average 0.054 mg/kg eq (range 0.019 - 0.075 mg/kg eq), while residue levels in the am milk were on average 0.030 mg/kg eq (range 0.024 - 0.034 mg/kg eq). Levels found in tissues are shown in Table 2.

Liver and muscle tissues were extracted with acetone/water (1:1, v/v) using both conventional and microwave methods. Conventional methods extracted 18% and 23% TRR in liver and muscle, respectively, while microwave extracted 34% and 35% TRR. In view of the higher extractability offered by microwave techniques, metabolite identification, characterisation and quantification of residues in liver and muscle was carried out on microwave extracts. Kidney was extracted in the same way, but by conventional methods (18% TRR extracted). An individual sample of milk with the highest residue concentration (day 5 evening milk, 0.075 mg/kg eq) was filtered and the filtrate analysed directly (77.2% TRR). Fat was subsequently extracted with ACN, hexane and hexane/MeOH by conventional methods. Extractables from the fat accounted for only 2.8% TRR and no further analysis was conducted. Remaining solids and protein precipitates derived from liver, kidney and muscle were subjected to acid hydrolysis (1 M HCl, 2 hours, 90 °C) to achieve further release of bound residues. Hydrolysis released a substantial part of the residues: 45.7% - 59.4% TRR. Identification of residues was carried out by chromatography with reference standards (listed in Table 1) using TLC and HPLC.

According to the study author, the high temperature and high pressure generated in the microwave extraction did not provide enough energy to break covalent bonds. Metabolites containing the carbamate moiety were not present in either conventional or microwave extracts. Three metabolites were identified in the liver, kidney and muscle as the hydroxypyrimidines R31805, R34865 and R31680. Individually these metabolites did not exceed 0.03 mg/kg eq (< 10% TRR). The same three metabolites were identified in milk, together with a fourth metabolite which was tentatively identified as the hydroxypyrimidine R406405. Individually these four metabolites represented no more than 0.01 mg/kg eq in milk. Table 2 summarises the distribution of radiolabelled residues in tissue and milk samples.

Table 2. Metabolite profile in goat tissues and milk (in mg/kg eq and % TRR) from extracts and hydrolysates.

Tissue	TRR [a]	TRR [b]	R31805 [c]	R34865 [c]	R31680 [c]	R406405 [c]	Unknowns [d]	Solids	Total
Liver	0.450	0.499	0.017 (3.6%)	0.024 (4.8%)	0.029 (5.9%)	ND	0.325 (65.1%)	0.061 (12.2%)	0.456 (91.6%)
Kidney	0.345	0.386	0.003 (0.8%)	0.012 (3.0%)	0.025 (6.4%)	ND	0.290 (75.1%)	0.011 (2.8%)	0.341 (88.2%)
Muscle	0.081	0.091	0.001 (1.1%)	0.004 (4.0%)	0.004 (4.7%)	ND	0.062 (67.6%)	< 0.001 (0%)	0.071 (80.2%)
Fat	0.018	0.018	-	-	-	-	0.001 (6.1%)	0.017 (93.9%)	0.018 (100%)
Milk [e]	0.075	0.075	0.002 (2.6%)	0.010 (12.7%)	0.010 (13.9%)	0.010 (13.6%)	0.026 (34.4%)	0.017 (22.8%)	0.075 (100%)

ND = not detected, - = not analysed

a TRR determined by direct combustion LSC of homogenised samples

b TRR determined from sum of extracts and remaining solids by (combustion LSC). This value was used to calculate %TRR

c including metabolites released after hydrolysis

d containing between 1-8 metabolites, unresolved areas, and origin material, each < 10% TRR.

e milk from day 5 pm milk, containing the highest residue concentration

Study 2. Ten white leghorn laying hens were dosed once daily for ten consecutive days with ^{14}C-labelled pirimicarb (see Figure 1, Akhavan *et al*., 1997: PP62/0530). The hens were 50 weeks of age at the start of the treatment and the average bodyweight during the treatment period was 1.5 kg. The radiopurity was > 95% (w/w). Pirimicarb was administered via gelatin capsules via a hollow plastic tube which was placed in the throat behind the tongue. The actual dosing rate based on the observed daily feed consumption (average 0.142 kg/day) was calculated to be 7.67 mg ai/kg feed, equivalent to 0.72 mg ai/kg bw/d. Eggs and excreta were collected daily during the dosing period and the eggs were separated into whites, yolks and shells. Six of the ten treated hens were monitored for expired $^{14}CO_2$ and volatile metabolites. The hens were sacrificed approximately 21-24 hrs after the final dose. Blood, liver, kidney, muscle (breast, thigh) and fat (subcutaneous, visceral) were collected from the carcasses. Samples were stored at -20 °C for up to 12 months until first extraction.

Homogenised samples were analysed for total ^{14}C-levels by combustion LSC or by extraction and (combustion) LSC. The overall recovery was 89.3% TAR. The largest amount of radioactivity was found in the excreta, which contained 88.1% TAR. The edible tissues (liver, kidney, muscle and fat) contained 0.57% TAR, while eggs contained 0.32% TAR. The hens were monitored for expired ^{14}C-volatiles, but none were found.

The residue levels in egg yolk and white reached a plateau at day 6 and day 3, respectively. The residue levels at the plateau level were on average 0.13 mg/kg eq (range 0.11-0.15 mg/kg eq) in egg yolks and on average 0.080 mg/kg eq (range 0.065-0.088 mg/kg eq) in egg whites. Total radioactive residues for tissues and day 9 egg samples are given in Table 3.

Liver and muscle tissues were extracted with acetone/water (1:1, v/v) using both conventional and microwave methods. Conventional methods extracted 23.1%, 43.3% and 38.0% TRR in liver, breast and thigh muscle, respectively, while microwave extracted 38.9%, 60.0% and 48.9% TRR. In view of the higher extractability offered by microwave techniques, metabolite identification, characterisation and quantification of residues in liver and muscle were carried out on microwave extracts. Proteins in the tissue extracts were precipitated using cold acetone and the supernatant was analysed. Eggs were extracted with acetone/water (1:5, v/v) using conventional extraction methods. Subsequently egg yolk extracts were partitioned with DCM. Fat was extracted with ACN using conventional methods. Extractables from the fat accounted for only 0.003 mg/kg eq and no further analysis was conducted. Kidney was not investigated. Remaining solids derived from liver and egg yolks were subjected to acid hydrolysis (1 M HCl and 6 M HCl, 2 hours at 90 °C) to achieve further release of bound residues. The liver hydrolysate was treated by cold acetone to remove precipitates. Identification of residues was carried out by chromatography with reference standards (listed in Table 1) using TLC and HPLC-DAD. Selected fractions were also analysed by MS.

The microwave extract of liver had a very similar profile to a conventional extract of liver which had been hydrolysed with 1 M HCl. According to the study author, the high temperature and high pressure generated in the microwave extraction did not provide enough energy to break covalent bonds, suggesting that the microwave extraction cleaved metabolites non-covalently bound to natural products like peptides. Metabolites containing the carbamate moiety were not found in either the pre-hydrolysis conventional extracts or microwave extracts. One major metabolite was present in liver, thigh and breast muscle. This was identified as the hydroxypyrimidine R31680 and accounted for 12.6% TRR in liver, 47.4% TRR in breast muscle and 31.6% TRR in thigh muscle. Liver also contained low levels of a second hydroxypyrimidine, R34865. Both of these metabolites, together with a third hydroxypyrimidine, R31805 were found in egg yolks and whites. The principal metabolite in eggs was metabolite R34865. Table 3 summarises the distribution of radiolabelled residues in tissue and egg samples. No individual unidentified metabolite accounted for more than 0.01 mg/kg eq in any of the samples.

Table 3. Metabolite profile in hen tissues and eggs (in mg/kg eq and % TRR) from extracts and hydrolysates.

Tissue	TRR [a]	TRR [b]	R31805 [c]	R34865 [c]	R31680 [c]	Unknowns [d]	Solids [e]	Total
Liver [e]	0.302	0.283	ND	0.001 (0.5%)	0.036 (12.6%)	0.18 (63.7%)	0.037 (13.0%)	0.254 (89.8%)
Kidney	0.112	-	-	-	-	-	-	-
Breast muscle [e]	0.145	0.151	ND	ND	0.072 (47.4%)	0.036 (24.0%)	0.036 (23.8%)	0.144 (95.2%)
Thigh muscle [e]	0.123	0.134	ND	ND	0.042 (31.6%)	0.045 (33.8%)	0.036 (26.6%)	0.123 (92.0%)
Fat [f]	0.020	0.020	-	-	-	0.003 (17.0%)	0.017 (83%)	0.020 (100%)
Egg white [f,g]	0.081	0.077	0.004 (5.4%)	0.037 (48.0%)	0.009 (11.8%)	0.021 (26.8%)	0.006 (7.5%)	0.077 (99.5%)
Egg yolk [f,g]	0.144	0.155	0.003 (2.0%)	0.039[h] (25.3%)	0.011 (7.2%)	0.077 (49.4%)	0.011 (6.9%)	0.141 (90.8%)

ND - not detected; - not analysed

a TRR determined by direct combustion LSC of homogenised samples

b TRR determined from sum of extracts and remaining solids by (combustion LSC). This value was used to calculate %TRR

c including metabolites released after hydrolysis

d containing at least 2-4 metabolites (up to 5.6% TRR), unresolved areas plus origin material (6.9%-43.6% TRR), protein precipitates remaining after first extraction (9.4%-20.1% TRR), and fractions not further specified (10% aqueous fraction from egg yolk, 4.4% organic fraction from egg yolk)

e solids remaining after hydrolysis of post-extracted liver solids or after extraction of muscle/fat

d Microwave extraction,

f Conventional extraction

g eggs from day 9 interval

h 11.0% TRR contains metabolites R34865 and R31680.

Plant metabolism

The Meeting received information on the fate of pirimicarb after foliar treatment of apple trees, lettuce, potatoes and wheat. Experiments were carried out with pirimicarb ^{14}C labelled at the pyrimidinyl-2 position (see Figure 1).

Pirimicarb has been registered for use since the 1970s. First registrations were supported by a diverse selection of crop metabolism studies which were carried out during the 1970s and early 1980s. All of the original metabolism studies have now been superseded by a full set of new studies, carried out to meet current regulatory guidelines and full GLP requirements; these are summarised below.

Study 1. Two apple trees (variety Golden Delicious) were grown in pots in a caged area open to normal weather conditions in the UK (Wilson and Muir, 1998: PP62/0265). The trees were sprayed with a WG formulation containing ^{14}C-labelled pirimicarb. The radiopurity was > 97.8% (w/w). The trees were sprayed three times: at petal fall, after the "June drop" and at 21 d PHI. Interval periods were 70 and 46 d, respectively. The first tree was treated with 3× 1.2 kg ai/ha and the second tree with 3x 1.1 kg ai/ha. Apples (4.4-5.3 kg) were harvested at DAT= 21. Homogenised apples were stored frozen for up to 2 years until analysis.

Total ^{14}C-radioactivity was determined by ACN extraction and (combustion) LSC. The total radioactive residues (TRRs) in the apples were 2.4 mg/kg eq and 1.7 mg/kg eq for the first and second tree, respectively. Hence apples from the first tree only were used for analysis of residues.

Apples were extracted with solvents of increasing polarity: hexane, DCM, ACN, ACN:water and water. A total of 94% TRR was extractable. The 6% TRR in the remaining solids and the 1.7% TRR in the water extract were not further analysed. Extracted residues were fractionated by TLC and

HPLC and metabolites were identified by co-chromatography with reference compounds (listed in Table 1). Selected fractions were analysed by HPLC-MS.

The majority of the residues in the hexane and DCM extracts (total 41% TRR) was identified as parent pirimicarb (30% TRR). Other carbamates were identified at low levels: R34885 (1.2% TRR), R34836 (1.8% TRR). An amount of 7.8% TRR remained unidentified.

The polar residues in the ACN and ACN:water extracts (total 51% TRR) did not contain any significant levels of carbamates. The combined extracts were subjected to acid hydrolysis (concentrated HAc, 24 hour, 100 °C) to cleave any simple glycosides of pirimicarb metabolites, followed by partitioning into EtOAc. 6.8% TRR was lost during hydrolysis. The EtOAc fraction (21% TRR) contained at least 2 unknown components with neutral properties (2.1% and 2.8% TRR) and 2 unknown components with acidic properties (total 10% TRR). The residual water soluble fraction (23% TRR) was highly polar in nature and remained at the TLC origin.

Further characterisation of the polar demonstrated that individual components represented well below 10% TRR in all cases.

Further characterization of the EtOAc and residual water soluble fraction by HPLC indicated that 1% TRR was associated with hydroxypyrimidines R34856 and R404094 or R404137 and 1.4% TRR was identified as hydroxypyrimidine R31805 (giving a total of 1.6% TRR). N,N-dimethylguanidine (R16210) was found at trace levels and urea was found at 1.4% TRR.

Study 2. Lettuce plants (variety Ravel) were grown in pots in sandy loam soil in a glasshouse in the UK (Mathis and Wilson, 1998: PP62/0266). Lettuce foliage was sprayed with a WG formulation containing ^{14}C- labelled pirimicarb. The radiopurity was > 97.7% (w/w). Plants were treated with three foliar applications using a handheld spray gun producing atomised solutions. The first pot was treated with 3x 0.255 kg ai/ha and the second pot with 3× 0.265 kg ai/h at intervals of 7 d with 8 week old plants. The heads of mature lettuce plants (180–270 g) were collected at DAT = 3 (first pot) and DAT = 7 (second pot). Homogenised samples were stored frozen for up to 6 months until analysis.

Total ^{14}C-radioactivity was determined by MeOH extraction and (combustion) LSC. Total radioactive residues in lettuce leaves were 14 and 12 mg/kg eq at DAT= 3 and 7, respectively.

Samples were extracted with MeOH, followed by partitioning into DCM. Acid hydrolysis was carried out on the remaining aqueous phase and on the remaining solids.

In the DAT =3 and DAT=7 samples, 91% and 88% TRR could be extracted with MeOH, respectively. Metabolite profiles of the residues are shown in Table 4. Pirimicarb and the carbamate metabolite R34836 together accounted for the majority of the radioactivity. Other carbamates and hydroxypyrimidines R31805 and R34865 were also identified at much lower levels. The figures quoted for the DAT=7 sample include contributions from both conjugated and unextractable hydroxypyrimidines.

Table 4. Metabolite profile for lettuce heads, treated by a foliar spray containing ^{14}C-pirimicarb.

		DAT = 3		DAT = 7	
Component/fraction		%TRR	mg/kg eq	%TRR	mg/kg eq
Total residue		100	14	100	12
Carbamates	parent	51.7	7.07	38.4	4.61
	R34836	17.0	2.32	20.9	2.51
	R35140	1.4	0.20	2.0	0.24
	R34885	1.4	0.20	1.2	0.14
	R238177	ND	ND	0.5	0.06
Hydroxypyrimidines	R31805	3.5	0.48	8.5 [a]	1.02
	R34865	0.6	0.09	6.0 [b]	0.72
	R31680 [c]	ND	ND	0.7	0.08
	R404094 [c]	ND	ND	0.5	0.06
	R407392 [c]	ND	ND	0.3	0.04
	R406405 [c]	ND	ND	0.2	0.02

		DAT = 3		DAT = 7	
Component/fraction		%TRR	mg/kg eq	%TRR	mg/kg eq
	R409238 [c]	ND	ND	1.2	0.14
organosoluble [d]		3.0	0.41	4.9	0.58
aqueous soluble [e]		9.4	1.28	5.5	0.66
unassigned [f]		-	-	1.1	0.13
solids [g]		8.8	1.20	5.0	0.60
losses		3.2	0.44	3.1	0.37

ND - not detected

a Includes 2.8% conjugated residue, 1.1% bound residue

b Includes 0.3% conjugated residue, 2.5% bound residue

c tentative identification by HPLC-UV only

d unidentified components remaining in the DCM phase, consisting of at least 4 discrete components, each < 1.1% TRR (DAT = 3) or < 2.3% TRR (DAT = 7)

e unidentified components remaining in the aqueous phase after DCM partitioning and after acid hydrolysis, consisting of at least 5-7 discrete components, each < 4.5% TRR (DAT = 3) or < 2.5% TRR (DAT = 7).

f areas of streaked radioactivity and baseline material

g solids remaining after the extraction procedure (without acid hydrolysis for DAT = 3 and with acid hydrolysis for DAT = 7)

Study 3. Potato plants (variety Manna) were grown in pots in sandy loam soil in a caged area open to normal weather conditions in the United Kingdom (Grout and Benner, 1998: PP62/0262). Potato foliage was sprayed with a WG formulation containing ^{14}C- labelled pirimicarb. The radiopurity was > 98.3% (w/w). Two separate experiments were conducted: one at a low dose of 2× 0.78 kg ai/ha with an interval of 13 d, the other at a high dose of 4× 2.8 kg ai/ha with intervals of 7, 6 and 8 days. Potato tubers (1.8–2.0 kg) were harvested at DAT = 17 and 18 for the low and high dose, respectively. Samples were stored at -10 °C for up to 6 months until analysis.

Total ^{14}C-radioactivity was determined by (combustion) LSC. The total radioactive residues found in tubers were 0.040 and 0.23 mg/kg eq for the low and high dose experiment, respectively.

Samples were extracted with ACN and water. The extracts were combined and partitioned into EtOAc. Identification of the residues in the extracts was carried out by co-chromatography against standard reference markers using TLC (listed in Table 1).

In the low dose experiment, 95.1% TRR could be extracted and very little residue partitioned into EtOAc (6.8% TRR). Neither parent nor any metabolites containing the carbamate functionality were found. The majority of the residue (90.2% TRR) remained in the aqueous phase and was comprised of highly polar water-soluble components of which no single metabolite exceeded 0.01 mg/kg eq.

In the high dose experiment, 95.0% TRR could be extracted and a small amount partitioned into EtOAc (13.0% TRR). Trace amounts of parent (1.7% TRR), carbamates R34836 (1.0% TRR) and R34885 (0.7% TRR) and a hydroxypyrimidine R31805 (1.1% TRR) were identified in this fraction. A total of four unknown components partitioned into EtOAc but none exceeded 4.6% TRR. The majority of the residue (81.6% TRR) remained in the aqueous phase and was very polar in nature.

Study 4. Wheat (variety Tonic) was grown in the field and then transplanted into a circular tub which was placed in a caged area open to normal weather conditions in the UK (Jessop, 1998: PP62/0264). Transplanting was carried out 55 days prior to the first treatment. Wheat foliage was sprayed with a WG formulation containing ^{14}C- labelled pirimicarb (see Figure 1). The radiopurity was > 98.8% (w/w). The crop was sprayed twice with a handheld sprayer at rates of 0.28 and 0.29 kg ai/ha, respectively. The first treatment was at growth stage BBCH 70–80 (just after completion of the flowering stage) and the second treatment was 35 days later. Wheat was collected at DAT = 14 and grain heads were separated from the straw. Grain was removed from chaff. Both straw and grains were stored at -10 °C for up to 18 months until analysis.

Total ^{14}C-radioactivity was determined by (combustion) LSC. Total radioactive residues found in wheat straw and grain were 16 mg/kg eq and 0.72 mg/kg eq, respectively. Straw and grain were extracted with ACN, ACN:water (1:1) and water. When TRR values were calculated from extracts and solids, the TRR was found to be 14 mg/kg eq and 0.67 mg/kg eq, respectively. The latter values were used for calculation of %TRR values in the extracts.

For grain 86.6% TRR was extracted, while 13.4% TRR remained as solids. The grain extracts were partitioned between DCM and water resulting in transfer of 36.0% TRR into the organic phase. The organic phase was composed primarily of carbamates with pirimicarb itself constituting the single largest carbamate (25.2% TRR). Other identified compounds were carbamates R34836 (2.8% TRR) and R34885 (1.3% TRR) and hydroxypyrimidine R31805 (1.6% TRR). The remaining aqueous phase (41.3% TRR) comprised highly polar, water soluble constituents.

Further examination of the whole wheat grains (RAC) demonstrated that approximately 8% TRR was incorporated into the natural grain components starch and glucose. In the solids remaining after extraction of wheat grain, 6.4% TRR was attributed to starch.

For straw 80.1% TRR was extracted. The extracts were partitioned between DCM and water and this resulted in transfer of 25.5% TRR into the organic phase. The majority of this was composed of carbamates with pirimicarb itself constituting the major carbamate (13.4% TRR).

TLC profiles of the extracted residues in both straw and grain were performed within the first 6 months after harvest and again at 18 months after harvest using the same extracts. The profiles were identical, indicating that no degradation occurred upon storage of the extracts.

Environmental fate in soil

The meeting received information on aerobic and anaerobic degradation in soil, photolysis on the soil surface, field dissipation, adsorption/desorption in soil, leaching into groundwater and confined and field rotational crop studies. Because pirimicarb is intended for use as a foliar treatment close to harvest, only the rotational crop studies were considered relevant for the present evaluations. The other information was not summarized.

Rotational crop studies

Study 1. A confined rotational crop study was undertaken to determine the accumulation and metabolic fate of ^{14}C-labelled pirimicarb (see Figure 1) under field conditions (Vispetto *et al.*, 1998: PP62/0529). The crops used were lettuce (variety Prize Head Red Leaf), radish (variety White Icicle) and millet (variety White Proso). A single application of pirimicarb in an aqueous ACN solution, at a rate of 1.48 kg ai/ha was made to bare soil in Visalia, California. The radiopurity was > 96.4% (w/w). The soil type was USDA sandy loam, pH 8.20, 0.92% om, CEC 7.38 meq/100g and 12.4% clay particles. Crops were planted into the soil at DAT = 29, 61, 119 and were harvested at maturity. Millet was also harvested at the forage and hay stages. Mature millet was separated into straw (leaves, stems), chaff and grain. Radishes were separated into roots and leaves. Analysis of soil was not conducted. Samples were stored frozen until analysis for up to 336 days.

Crops were extracted and total ^{14}C residues in the crops were quantified by direct combustion LSC and by (combustion) LSC of extracts and remaining solids. Any remaining solids representing > 10% TRR or 0.05 mg/kg eq were further hydrolysed with 1 M HCl at 100 °C. All fractions containing > 0.01 mg/kg eq and > 10% TRR were analysed by TLC and HPLC. In view of similar metabolite profiles across crops, only the extracts from 29DAT grain and 61DAT radish leafs were used for identifying and characterizing metabolites.

Results are shown in Tables 5 and 6. Control samples contained up to 0.007 mg/kg eq ^{14}C residues, indicating a minimum LOQ of 0.03 mg/kg eq for this study. The total radioactive residues in the crops declined significantly as the rotation interval increased. Millet straw had the highest residues at all rotations, while the lowest residue was found in the radish root. Leafy, root and small grain crops all show the same metabolic profiles. Low levels of pirimicarb and carbamate metabolites

(R34836, R34885 and R35140) are found in some samples and levels of carbamates decrease as the rotation interval increases. Other identified compounds are hydroxypyrimidines (R31805, R34865 and R31680) and guanidine (R12378). Levels of hydroxypyrimidine metabolites decreased as the rotation interval increases.

Crop extracts were profiled within 6 months after harvest, except for the 119 DAT grain and straw samples (11 months). HPLC metabolite profiles from initial extractions and profiles from re-chromatographed extracts at the end of the study, showed good agreement.

Table 5. Metabolite profile of residues in rotational crops lettuce and radish.

Crop		Lettuce			Radish roots			Radish leaves		
Planting interval	DAT	29	61	119	29	61	119	29	61	119
Harvest interval	DAP	46	58	56	32	37	40	32	37	40
Control	mg/kg eq	ND	ND	ND	ND	ND	ND	ND	0.001	ND
TRR a	mg/kg eq	0.338	0.298	0.139	0.154	0.055	0.029	2.15	1.03	0.264
TRR b	mg/kg eq	0.299	0.366	0.125	0.179	0.064	0.034	1.81	1.11	0.331
parent	%TRR	ND	ND	ND	9.33	20.0	11.5	1.00	1.36	1.30
R34885	%TRR	3.84	1.91	ND	1.86	4.14	3.33	0.205	0.434	0.291
R34836	%TRR	3.37	2.66	2.17	7.17	14.5	9.71	3.11	3.8	2.66
R35140	%TRR	5.88	2.71	1.66	1.27	1.69	0.78	1.22	2.58	1.42
R31805	%TRR	1.23	2.83	2.35	4.02	2.27	3.16	4.16	4.48	2.70
R34865	%TRR	6.10	7.61	8.76	4.46	3.09	2.46	7.25	6.89	3.84
R31680	%TRR	3.91	5.02	5.05	8.00	9.84	3.73	9.74	13.4	16.1
R12378	%TRR	-	-	-	-	-	-	-	5.1	-
Met A	%TRR	2.13	0.602	0.071	2.69	1.12	1.13	4.49	5.36	5.13
Met B	%TRR	0.427	2.40	1.30	1.67	ND	0.030	2.40	3.83	2.34
Met C	%TRR	7.95	6.81	3.84	2.58	0.716	ND	3.83	6.53	3.56
unkn	%TRR	53.36	35.88	42.6	50.05	25.53	40.80	40.7	52.9	47.3
solids	%TRR	11.5	15.5	24.1	6.29	11.9	12.2	4.45	2.68	11.3
Total	%TRR	99.7	83.9	91.9	99.4	94.8	88.8	82.6	109.3	97.9

DAT = planting expressed as days after treatment,

DAP = harvest expressed as days after planting,

ND = not detected (< 0.001 mg/kg eq)

- not analyzed for this compound, no data obtained.

a calculated from direct combustion data

b. calculated from extraction/combustion data; these values were used to calculate %TRR

unkn = unknown residues

solids = solids remaining after first (and only) extraction or after hydrolysis

Table 6. Metabolite profile of residues in rotational crop millet.

Crop		Millet forage			Millet hay			Millet straw			Millet grain		
Planting interval	(DAT)	29	61	119	29	61	119	29	61	119	29	61	119
Harvest interval	(DAP)	32	28	15	55	56	40	89	98	82	89	98	82
Control	mg/kg eq	0.003	0.002	0.001	ND	0.003	ND	ND	ND	0.007	ND	ND	0.003
TRR [a]	mg/kg eq	1.75	0.679	0.180	1.61	0.628	0.157	4.98	1.32	0.944	0.261	0.118	0.077
TRR [b]	mg/kg eq	1.79	0.626	0.166	1.59	0.594	0.182	5.04	1.36	0.951	0.258	0.125	0.071
parent	%TRR	1.05	0.546	3.29	0.136	ND	ND	ND	ND	ND	0.886	ND	ND
R34885	%TRR	1.44	0.435	0.742	0.536	0.385	ND	0.72	0.759	ND	ND	0.358	ND
R34836	%TRR	1.47	1.3	4.69	1.57	0.213	ND	1.01	0.894	0.798	1.12	ND	7.72
R35140	%TRR	2.66	1.67	ND	2.36	1.06	0.554	1.19	1.29	0.926	0.837	ND	3.9
R31805	%TRR	6.32	3.16	3.54	5.04	4.3	1.84	2.36	0.948	2.49	2.67	1.53	0.41
R34865	%TRR	6.24	2.22	3.29	5.21	4.02	2.91	4.68	4.47	2.78	2.93	1.65	2.53
R31680	%TRR	8.52	8.66	9.85	10.5	13	12.4	11.3	8.38	15.1	6.7	10	7.93

Crop		Millet forage			Millet hay			Millet straw			Millet grain		
Planting interval	(DAT)	29	61	119	29	61	119	29	61	119	29	61	119
Harvest interval	(DAP)	32	28	15	55	56	40	89	98	82	89	98	82
R12378	%TRR	-	-	-	-	-	-	-	-	-	6.35	-	-
Met A	%TRR	1.90	0.041	ND	1.74	1.63	1.09	1.35	1.5	1.46	0.848	0.524	ND
Met B	%TRR	1.31	1.16	ND	0.841	2.86	ND	2.22	1.77	1.13	0.361	0.505	2.53
Met C	%TRR	4.77	2.57	0.349	2.99	2.97	3.55	2.73	1.85	2.21	2.06	0.692	ND
unkn	%TRR	50.7	55.8	51.95	52.5	50.67	44.66	45.47	55.2	55.0	58.39	61.051	55.88
solids	%TRR	11.2	10.8	14.0	12.5	11.7	14.2	15.3	9.7	11.3	19.5	21.3	22.4
Total	%TRR	97.6	88.4	91.7	95.9	92.8	81.2	88.3	86.8	93.2	102.7	97.6	103.3

DAT = planting expressed as days after treatment,

DAP = harvest expressed as days after planting

ND = not detected (< 0.001 mg/kg eq)

N/A Not applicable, no data obtained.

a calculated from direct combustion data

b. calculated from extraction/combustion data; these values were used to calculate %TRR

Study 2. Two supervised field trials (Jones and Miller, 1998: PP62/0951) were carried out in the USA (Whitakers, North Carolina and Visalia, California) during 1997 to determine the magnitude of residues of pirimicarb in rotational crops (millet, mustard and turnip). At each site two plots were used with a primary crop of lettuce (variety Black Seeded Simpson in NC and Romain Green Towers in CA). Pirimicarb was applied as a WG formulation at 4× 0.56 kg ai/ ha with 5 day intervals on the first plot and as 1× 0.37 kg ai/ha + 2× 0.56 kg ai/ha with a 5 and 10 day interval on the second plot. The last application in the NC trial was at the vegetative growth stage. In the CA lettuce trial the last application was at the full grown vegetative growth stage. The lettuce was removed and separate plots of millet, mustard and turnip were planted back at 30, 60 and 120 days after the last application. Crop varieties were Southern Giant Curled for NC and Florida Broadleaf for CA for mustard; Purple Top for NC and Purple Top White Globe for CA for turnips; and Brown Top for millet. The rotated crops were sampled at normal harvest for the rotational crop. Millet forage, hay, grain and straw, mustard leaves and turnip roots and tops were collected. Samples were stored at -10 °C for up to 428 days (14 months), extracts were stored for a maximum of 15 days. Samples were analysed for pirimicarb, R34836, R34885 and R238177

No residues of pirimicarb or its metabolites were measured in any of the samples from North Carolina from any of the plant back intervals (< 0.01 mg/kg, each analyte). Low pirimicarb and R34836 residues were measured in some of samples from the Californian trials. The highest total pirimicarb residue residues were found in millet forage (0.05-0.07 mg/kg eq) and mustard leaves (0.03-0.04 mg/kg eq) from the 30 day planting interval. No residues of R238177 were measured in any of the samples (< 0.01 mg/kg). No residues (< 0.01 mg/kg, each analyte) were measured in any of the untreated samples. A summary of the results for the Californian trials is given in Table 7.

Table 7. Pirimicarb residue levels in rotational crops from the Californian trials.

Dose rate (kg ai/ha)	Planting interval (DAT)	Harvest interval (DAP)	Commodity	Pirimicarb (mg/kg)	R34836 [a] (mg/kg)	R238177 (mg/kg)	Total Pirimicarb residue [b] (mg/kg eq)
4x 0.56	30	24	Millet - forage	**0.06**	< 0.01	< 0.01	**0.06**
1x 0.37 + 2x 0.56	30	24	Millet - forage	**0.04**	< 0.01	< 0.01	**0.04**
4x 0.56	30	42	Millet - hay	**0.01**	**0.02**	< 0.01	**0.03**
1x 0.37 + 2x 0.56	30	42	Millet - hay	< 0.01	**0.02**	< 0.01	**0.02**
4x 0.56	30	83	Millet - grain	< 0.01	< 0.01	< 0.01	< 0.01
1x 0.37 + 2x 0.56	30	83	Millet - grain	< 0.01	< 0.01	< 0.01	< 0.01
4x 0.56	30	83	Millet - straw	< 0.01	< 0.01	< 0.01	< 0.01
1x 0.37 + 2x 0.56	30	83	Millet - straw	< 0.01	< 0.01	< 0.01	< 0.01
4x 0.56	30	36	Mustard - leaves	**0.01**	**0.03**	< 0.01	**0.04**

Dose rate (kg ai/ha)	Planting interval (DAT)	Harvest interval (DAP)	Commodity	Pirimicarb (mg/kg)	R34836 [a] (mg/kg)	R238177 (mg/kg)	Total Pirimicarb residue [b] (mg/kg eq)
1x 0.37 + 2x 0.56	30	36	Mustard - leaves	< 0.01	**0.02**	< 0.01	**0.02**
4x 0.56	30	45	Turnip - roots	< 0.01	< 0.01	< 0.01	< 0.01
1x 0.37 + 2x 0.56	30	45	Turnip - roots	< 0.01	< 0.01	< 0.01	< 0.01
4x 0.56	30	45	Turnip - tops	< 0.01	< 0.01	< 0.01	< 0.01
1x 0.37 + 2x 0.56	30	45	Turnip - tops	< 0.01	< 0.01	< 0.01	< 0.01
4x 0.56	60	29	Millet - forage	**0.01**	**0.02**	< 0.01	**0.03**
1x 0.37 + 2x 0.56	60	29	Millet - forage	< 0.01	< 0.01	< 0.01	< 0.01
4x 0.56	60	42	Millet - hay	**0.01**	**0.03**	< 0.01	**0.04**
1x 0.37 + 2x 0.56	60	42	Millet - hay	< 0.01	**0.01**	< 0.01	**0.01**
4x 0.56	60	73	Millet - grain	< 0.01	< 0.01	< 0.01	< 0.01
1x 0.37 + 2x 0.56	60	73	Millet - grain	< 0.01	< 0.01	< 0.01	< 0.01
4x 0.56	60	73	Millet - straw	< 0.01	< 0.01	< 0.01	< 0.01
1x 0.37 + 2x 0.56	60	73	Millet - straw	< 0.01	< 0.01	< 0.01	< 0.01
4x 0.56	60	37	Mustard - leaves	< 0.01	< 0.01	< 0.01	< 0.01
1x 0.37 + 2x 0.56	60	37	Mustard - leaves	< 0.01	< 0.01	< 0.01	< 0.01
4x 0.56	60	42	Turnip - roots	< 0.01	< 0.01	< 0.01	< 0.01
1x 0.37 + 2x 0.56	60	42	Turnip - roots	< 0.01	< 0.01	< 0.01	< 0.01
4x 0.56	60	42	Turnip - tops	< 0.01	< 0.01	< 0.01	< 0.01
1x 0.37 + 2x 0.56	60	42	Turnip - tops	< 0.01	< 0.01	< 0.01	< 0.01
4x 0.56	120	32	Millet - forage	**0.01**	**0.01**	< 0.01	**0.02**
1x 0.37 + 2x 0.56	120	32	Millet - forage	< 0.01	< 0.01	< 0.01	< 0.01
4x 0.56	120	40	Millet - hay	< 0.01	**0.02**	< 0.01	**0.02**
1x 0.37 + 2x 0.56	120	40	Millet - hay	< 0.01	**0.02**	< 0.01	**0.02**
4x 0.56	120	89	Millet - grain	< 0.01	< 0.01	< 0.01	< 0.01
1x 0.37 + 2x 0.56	120	89	Millet - grain	< 0.01	< 0.01	< 0.01	< 0.01
4x 0.56	120	89	Millet - straw	< 0.01	< 0.01	< 0.01	< 0.01
1x 0.37 + 2x 0.56	120	89	Millet - straw	< 0.01	< 0.01	< 0.01	< 0.01
4x 0.56	120	35	Mustard - leaves	**0.01**	**0.02**	< 0.01	**0.03**
1x 0.37 + 2x 0.56	120	35	Mustard - leaves	< 0.01	**0.01**	< 0.01	**0.01**
4x 0.56	120	55	Turnip - roots	< 0.01	< 0.01	< 0.01	< 0.01
1x 0.37 + 2x 0.56	120	55	Turnip - roots	< 0.01	< 0.01	< 0.01	< 0.01
4x 0.56	120	55	Turnip - tops	< 0.01	< 0.01	< 0.01	< 0.01
1x 0.37 + 2x 0.56	120	55	Turnip - tops	< 0.01	< 0.01	< 0.01	< 0.01

a R34836 = R34836 plus R34885, determined as R34836, LOQ = 0.01 mg/kg (expressed as R34836)

b Total pirimicarb residue = pirimicarb + 1.06x R34836 Because residues of the demethyl metabolites did not generally contribute significantly to the total residue, they are only included in the total where they were reported at levels above the LOQ

Environmental fate in water/sediment systems

The Meeting received information on the hydrolysis and photolysis of pirimicarb in water and degradation in water/sediment systems. Only the hydrolysis and photolysis studies were considered relevant for the present evaluation. The other studies were not summarized.

Hydrolysis in water

The hydrolysis of ^{14}C-pirimicarb in sterile buffer solutions was investigated under laboratory conditions (Huynh and Mathis, 1996: PP62/0915). The actual test substance concentration at initiation was 1.09 mg/L. Vials were incubated in the dark at 25 ± 1 °C for 0, 2, 7, 14, 21 and 30 days. Duplicate samples were analysed by LSC and TLC against standard reference compounds parent, R34885, R31805 and R34836.

Recovery of total radioactivity was 90.3–101.7%. The pH remained constant during incubation. Less than 5% degradation was observed after 30 d at 25 °C at pH 5, 7 and 9.

Photolysis in water

The photolysis of ^{14}C-pirimicarb in sterile buffer solutions was investigated under laboratory conditions (Hamlet, 1997: PP62/0916). The actual test substance concentration at initiation was 1.04 mg/L. Sterile solutions at pH 5 and 7 were maintained at 25 ± 1 °C and exposed to a Xenon arc lamp. Duplicate samples were taken after 1, 2, 3.5, 5.5, 7.5 and 24 hrs of continuous irradiation. Samples were stored in the dark at -10 °C for up to 3 weeks until analysis. Samples were analysed by LSC, TLC, HPLC and HPLC-MS against standard reference compounds parent, R35140, R34885, R31805, R34836, R12378, R16210 and R16192.

Recovery of total radioactivity was 99.3–101.9%. The pH remained constant during irradiation. No degradation was apparent in the dark controls. Pirimicarb is rapidly degraded by photolysis in aqueous solution with DT_{50} values of 3.20 hours and 2.28 hours at pH 5 and 7, respectively. After a period equivalent to 31 hrs summer sunlight, only 1.2% and 1.4% TAR parent remained at pH 5 and 7. The major degradation products formed are compounds R34885, R31805 and R16210, which accounted for up to 17.9%, 27.8% and 14.1% TAR, respectively at pH 5 and 16.4%, 25.5% and 26.9% TAR at pH 7.

Overview of metabolism in livestock, primary crops and rotational crops

Figure 2 gives an overview of metabolism in livestock, primary crops and confined rotational crops. Neither pirimicarb nor any carbamate containing metabolites were found in livestock (ruminant tissues, poultry tissues, eggs, milk). The metabolites identified in livestock were hydroxypyrimidines, which were readily excreted in the urine and faeces.

In plants (apple, lettuce, potato, wheat and rotational crops) pirimicarb undergoes extensive metabolism to give a diverse range of metabolites. The early stages of metabolism involve modification of the dimethylamino moiety on position 2 of the pyrimidine ring, hydroxylation of pirimicarb on a methyl position and loss of the carbamate moiety to form hydroxypirimidines. Further degradation of the hydroxypyrimidines takes place, resulting in ring opening of the pyrimidine moiety and further degradation to form small polar molecules such as guanidines.

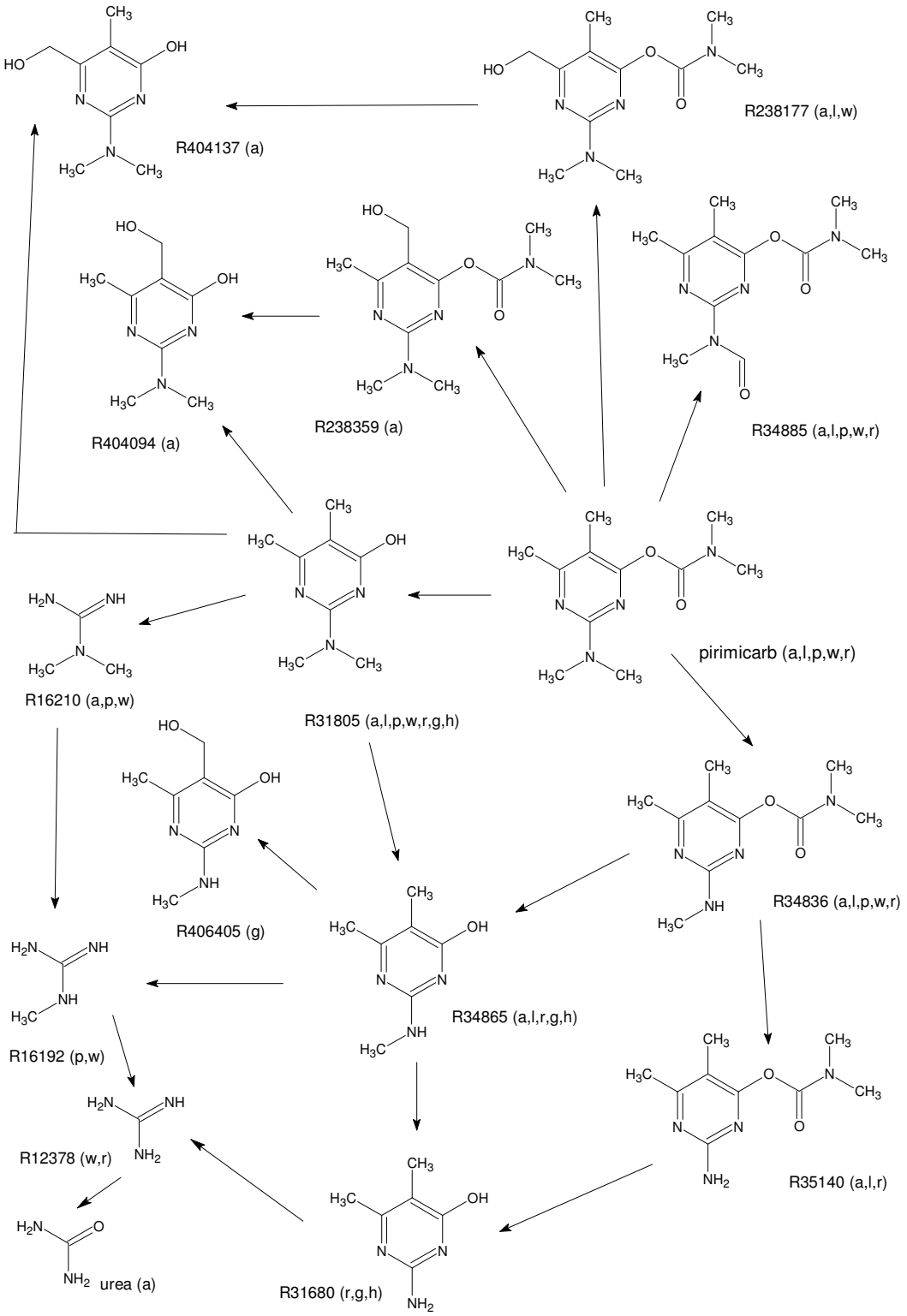


Figure 2. Metabolism scheme for plants and livestock (a= apple, l = lettuce, p = potato, w = wheat, r= rotational crops, g = goat, h= hen).

METHODS OF RESIDUE ANALYSIS

The Meeting received data on analytical methods for enforcement and monitoring of pirimicarb and its carbamate metabolites (R34836, R34855, and R238177) in plant commodities and pirimicarb and its carbamate metabolite R34836 in animal commodities. Further The Meeting received information on analytical methods actually used in the study reports.

Analytical methods for enforcement and monitoring

Method RAM 265 is intended for use as an enforcement-monitoring method for the determination of pirimicarb and its carbamate metabolites (R34836, R34855, and R238177) in plant commodities. Because the method is also used in study reports, the method is described under "analytical methods used in study reports". Method RAM 265 is not a published method. Method RAM 265 is considered a special method and cannot be included in a multi-residue method because of the acid treatment required for the conversion of metabolite R34855 into R34836.

Method DFG S19 is intended for use as enforcement-monitoring method for the determination of pirimicarb and its carbamate metabolite (R34836) in animal commodities. Method DFG S19 is a published German multi-residue method (DFG S19, 1995 and 1999 and Specht *et al*., 1995

Tillkes, 1995: PP62/0243 validated the DFG S19 method with modified extraction (see DFG S19, 1995 and Specht *et al*., 1995) for the determination of pirimicarb and demethyl-pirimicarb (R34836) in milk, muscle, kidney, liver and eggs. The animal species of the samples was not indicated. The study author used acetone extraction followed by liquid-liquid partition with EtOAc-cyclohexane, clean-up by GPC, supplemental clean-up by silica gel and quantification by GC-MS for pirimicarb and demethyl-pirimicarb. Calibration was carried out with external standards in EtOAc using a single point calibration either at 0.02 or at 0.2 mg/L for each analyte (corresponding to 0.02 or 0.2 mg/kg in the sample). The reported LOQ was 0.01 mg/kg for each analyte. Validation results are presented in Tables 8 and 9.

Lakaschus, 2005: PP62/1468 validated the extended revision of DFG S19 method (see DFG S19, 1999) for the determination of pirimicarb in milk, eggs, liver, meat, kidney and fat. The animal species of the samples was not indicated. The study author used extraction module E8 for milk, eggs, liver, kidney and meat (cold n-hexane-acetone (2:1, v/v) extraction), extraction module E6 for fat (dissolution in cyclohexane-HAc (1:1, v/v)), GPC clean-up and quantification by HPLC-MS-MS for quantification confirmation of pirimicarb. Calibration was carried out between 0.2 µg/L–50 µg/L. The reported LOQ was 0.01 mg/kg for pirimicarb.

Reichert, 2005: PP62/1469 contains an independent laboratory validation of the extended revision of DFG S19 method (see DFG S19, 1999) for the determination of pirimicarb in egg and milk as described by Lakashus, 2005: PP62/1468. Calibration was carried out using an eight point calibration between 0.2 µg/L–20 µg/L. Actual analytical concentrations were within 20% of the calibration standards (corresponding mg/kg in the sample not stated).

Table 8. Validation results for parent using method DFG S19.

Commodity	Reported LOQ mg/kg	spike level mg/kg	n	% recovery mean	% recovery range	RSD_r	Control samples mg/kg (n)	Calibration	Reference, method
milk	0.01	0.02 0.2	2 2	85 92	79-91 90-95	- -	< 0.01 (1)	in solvent single point at 0.02 or 0.2 mg/kg	PP62/0243 GC-MS
muscle	0.01	0.02 0.2	2 2	86 84	86-87 82-86	- -	< 0.01 (1)	in solvent single point at 0.02 or 0.2 mg/kg	PP62/0243 GC-MS
kidney	0.01	0.02 0.2	2 2	104 104	95-112 102-105	- -	< 0.01 (1)	in solvent single point at 0.02 or 0.2 mg/kg	PP62/0243 GC-MS

Commodity	Reported LOQ mg/kg	spike level mg/kg	n	% recovery mean	range	RSD_r	Control samples mg/kg (n)	Calibration	Reference, method
liver	0.01	0.02 0.2	2 2	102 88	97-106 86-91	- -	< 0.01 (1)	in solvent single point at 0.02 or 0.2 mg/kg	PP62/0243 GC-MS
eggs	0.01	0.02 0.2	2 2	90 90	84-97 88-91	- -	< 0.01 (1)	in solvent single point at 0.02 or 0.2 mg/kg	PP62/0243 GC-MS
milk	0.01	0.01 0.1	5 5	107 101	91-118 90-107	10% 6.5%	< 0.3LOQ (2)	in solvent six points 0.2-50 µg/L linear, $r^2 > 0.999$	PP62/1468 HPLC-MS-MS m/z= 182
eggs	0.01	0.01 0.1	5 5	94 94	91-97 93-96	3.0% 1.2%	< 0.3LOQ (2)	in solvent six points 0.2-50 µg/L linear, $r^2 > 0.999$	PP62/1468 HPLC-MS-MS m/z= 182
liver	0.01	0.01 0.1	5 5	81 77	76-88 74-79	5.7% 2.5%	< 0.3LOQ (2)	in solvent six points 0.2-50 µg/L linear, $r^2 > 0.999$	PP62/1468 HPLC-MS-MS m/z= 182
meat	0.01	0.01 0.1	5 5	97 98	96-100 94-100	1.8% 2.6%	< 0.3LOQ (2)	in solvent six points 0.2-50 µg/L linear, $r^2 > 0.999$	PP62/1468 HPLC-MS-MS m/z= 182
kidney	0.01	0.01 0.1	5 5	86 82	74-99 79-86	11% 3.3%	< 0.3LOQ (2)	in solvent six points 0.2-50 µg/L linear, $r^2 > 0.999$	PP62/1468 HPLC-MS-MS m/z= 182
fat	0.01	0.01 0.1	5 5	101 101	96-106 95-107	4.7% 4.5%	< 0.3LOQ (2)	in solvent six points 0.2-50 µg/L linear, $r^2 > 0.999$	PP62/1468 HPLC-MS-MS m/z= 182
eggs ILV	0.01	0.01 0.1	5 5	70 73	61-81 57-82	13% 14%	< 0.3LOQ (2)	in solvent 8 points 0.2-20 µg/L linear, r > 0.999	PP62/1468 HPLC-MS-MS m/z= 182
milk ILV	0.01	0.01 0.1	5 5	92 98	71-104 88-105	14% 6%	< 0.3LOQ (2)	in solvent 8 points 0.2-20 µg/L linear, r > 0.999	PP62/1468 HPLC-MS-MS m/z= 182

Table 9. Validation results for R34836 using method DFG S19.

Commodity	Reported LOQ mg/kg	spike level mg/kg	n	% recovery mean	range	RSD_r	Control samples mg/kg (n)	Calibration	Reference, method
milk	0.01	0.02 0.2	2 2	114 100	109-118 93-107	- -	< 0.01 (1)	in solvent single point at 0.02 or 0.2 mg/kg	PP62/0243 GC-MS
muscle	0.01	0.02 0.2	2 2	86 90	85-86 88-93	- -	< 0.01 (1)	in solvent single point at 0.02 or 0.2 mg/kg	PP62/0243 GC-MS
kidney	0.01	0.02 0.2	2 2	108 104	106-110 100-107	- -	< 0.01 (1)	in solvent single point at 0.02 or 0.2 mg/kg	PP62/0243 GC-MS
liver	0.01	0.02 0.2	2 2	104 109	98-110 104-114	- -	< 0.01 (1)	in solvent single point at 0.02 or 0.2 mg/kg	PP62/0243 GC-MS
eggs	0.01	0.02 0.2	2 2	92 97	77-108 96-98	- -	< 0.01 (1)	in solvent single point at 0.02 or 0.2 mg/kg	PP62/0243 GC-MS

Analytical methods used in study reports

In the methods described below, the metabolite concentrations are measured against metabolite standards. As such, the results for each metabolite are in metabolite equivalents. This means that all values presented in the study reports have to be multiplied by a factor 1.063 for R34836 and a factor 0.937 for R238177 to convert the concentration levels into parent equivalents.

Method PPRAM 15 (1972-1997)

Several versions of method PPRAM 15 exist: PPRAM 15, PRAM 15/1, PPRAM 15/2 and RAM 015/02. The original method was developed for the determination of pirimicarb and its carbamate metabolites (R34836, R34855) in plant commodities. Method PPRAM 15/1 was extended to water samples. Method PPRAM 15/2, which was renamed RAM 015/02, was restricted to apples, alfalfa, lettuce, pecans and cole crops.

The original method PPRAM 15 (1972-1974) was not used in any of the studies. A full method description is available (Bullock, 1972: PP62/0761). Samples were macerated with chloroform in the presence of anhydrous Na_2SO_4. Crops with high water content require higher amounts of anhydrous Na_2SO4. Filtered extracts were evaporated to dryness. After addition of n-hexane and 0.1 M HCl (1:2, v/v), the samples were left to stand overnight. During this time any demethyl formamido pirimicarb (R34855) was converted into demethyl pirimicarb (R34836). The acid aqueous layer was washed with EtOAc, neutralized with 1 M NaOH, adjusted to pH 6.7 by addition of phosphate buffer and extracted with EtOAc. The EtOAc extract was dried over anhydrous Na_2SO_4, evaporated to dryness and redissolved in acetone. For brassica and tobacco, an additional clean-up step was necessary. The acetone solution was subjected to TLC (silica gel) and TLC spots for pirimicarb and R34836 were transferred to a glass column and eluted with acetone. Pirimicarb and its carbamate metabolite (R34836) were analysed by GC-NPD. The reported LOQ was 0.01 mg/kg for each analyte.

PPRAM 15/1 (1974-1983) was developed for crop and water samples. The method was used in residue trials and processing studies on Brussels sprouts, head cabbage and lettuce. A full method description is available (Bullock *et al*., no date: PP62/0760). Plant samples were macerated with MeOH instead of chloroform and Na_2SO_4. After addition of n-hexane and 0.1 M HCl (1:1, v/v), the samples were left to stand overnight. The extract was treated further as in the original PPRAM 15 method, although volumes were changed. Water samples were acidified with concentrated HCl and shaken for 2 min with hexane and left to stand overnight. The acid aqueous layer was treated further as in the original PPRAM 15 method Analysis was by GC-NPD with modifications. The reported LOQ was 0.01 mg/kg for each analyte.

Method PPRAM 15/2 (1983-1992) was restricted to apples, alfalfa, lettuce and cole crops. Method PPRAM 15/2 is the same as method PPRAM 15/1, but uses only the default GC-column. Method PPRAM 15/2 was renamed later as RAM 015/02 (1993-1997). The method was used in residue trials, processing studies and storage stability studies on peach, cucumber and sweet corn. A full method description is available (Bullock *et al*., no date: PP62/0225 and Dick, 1993: PP62/0225). The reported LOQ was 0.01 mg/kg for each analyte.

A modification method PPRAM 15/2 and RAM 015/02 (1990-1997) was used in residue trials on apples, peaches, broccoli, cauliflower, Brussels sprouts, and sugarbeets (leaves, roots). GC-NPD was replaced by GC-MS. No method description is available. The reported LOQ was 0.01 mg/kg for each analyte.

Extraction efficiency was verified in peppers with incurred residues obtained from a drench treatment of soil (Edwards and Dick, 1975: PP62/0224). Pepper samples were extracted and generally further treated as in the original PPRAM 15 method. Results are shown in Table 10. Chloroform, methanol, and 3 hrs reflux in MeOH show efficient extraction of incurred residues.

No validation reports are available for methods PPRAM and PPRAM 15/1. Validation of method PPRAM 15/2 was described for apples, tomatoes, broad beans and potatoes (Cullen, 1993: PP62/0239). Results from this validation study are shown in Tables 11, 12 and 13.

Reduced validation results from individual study reports for method PPRAM 15/1 and the GC-MS modification are summarized in Tables 11, 12 and 13.

Table 10. Extraction efficiency of various solvents for incurred residues in peppers.

Extraction solvent	pirimicarb (mg/kg)	Efficiency	R34836 (incl R34885) (mg/kg)	Efficiency
chloroform	0.52	100%	0.01	100%
MeOH	0.52	100%	0.01	100%
MeOH 3 hrs hot reflux	0.52	100%	0.01	100%
acetone	0.45	87%	0.01	100%
acetone:aqueous NH_4Cl (1:1, v/v)	0.34	65%	0.01	100%

Table 11. Validation results for parent using method PPRAM 15.

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference, method
Method version PPRAM 15/1 with GC-NPD detection									
Brussels sprouts	0.01	5	1	100	-	-	0.02 (1)	-	PP62/0523 alternative GC-column
Brussels sprouts, boiled	0.01	1	1	99	-	-	0.01 (1)	-	PP62/0523 alternative GC-column
head cabbage	0.01	10	1	84	-	-	< 0.01 (1)	-	PP62/0523 alternative GC-column
head cabbage, boiled	0.01	1	1	92	-	-	< 0.01 (1)	-	PP62/0523 alternative GC-column
water (washing, boiling)	0.01	1	3	88	61-94	21%	< 0.01 (3)	-	PP62/0523 alternative GC-column
lettuce	0.01	1 5	1 1	81 78	- -	-	< 0.01 (2)	-	PP62/0523 alternative GC-column
Method version PPRAM 15/2 or RAM 015/02 with GC-NPD detection									
apple	0.02	0.02 0.1 0.5 5	2 2 2 2	76 72 69 78	74-77 62-82 67-71 74-82	- - - -	< 0.02 (1)	-	PP62/0239 column clean-up omitted
tomato	0.02	0.02 0.1 0.5 5	2 2 2 2	84 81 93 76	83-84 77-84 91-95 68-84	- - - -	< 0.02 (1)	-	PP62/0239 column clean-up omitted
broad beans	0.05	0.1 0.5 5	4 3 3	97 84 83	87-111 84-85 80-86	10% 0.7% 3.7%	< 0.05 (1)	-	PP62/0239 column clean-up omitted
potato	0.02	0.02 0.05 0.1 0.5	2 2 2 2	68 69 69 73	65-74 61-75 67-71 73-73	6.2% 9.5% - -	< 0.02 (1)	-	PP62/0239 column clean-up omitted
Method version PPRAM 15/2 or RAM 015/02 with GC-MS detection									
apple	0.01	0.02 0.05 0.1 0.25 0.5 1	1 1 2 2 1 2	78 63 87 84 85 85	- - 68-105 82-85 - 83-86	- - - - - -	-	-	PP62/0406
peach	0.01	0.02 0.05 0.1 0.2	1 1 2 1	96 102 95 91	- - 90-99 -	- - - -	-	-	PP62/0419

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference, method
nectarine	0.01	0.1	2	96	92-100	-	-	-	PP62/0419
		0.2	1	98	-	-			
		0.25	1	90	-	-			
broccoli	0.01	0.02	2	96	93-94	-	-	-	PP62/0310
		0.1	1	83	-	-			
		0.2	1	87	-	-			
cauliflower	0.01	0.02	3	98	92-105	6.7%	-	-	PP62/0310
		0.2	1	104	-	-			
broccoli	0.01	0.05	1	82	-	-	-	-	PP62/0311
		0.1	1	86	-	-			
cauliflower	0.01	0.1	1	117	-	-	-	-	PP62/0311
		0.2	1	95	-	-			
Brussels sprouts	0.01	0.1	2	73	73-73	-	-	-	PP62/0330
cauliflower	0.01	0.1	2	81	76-81	-	-	-	PP62/0333
sugarbeet, roots	0.01	0.05	1	100	-	-	-	-	PP62/0268
		0.1	5	85	72-97	12%			
sugarbeet, leaves	0.01	0.1	4	94	85-101	7%	-	-	PP62/0268
		1.0	1	87	-	-			
		2.0	1	82	-	-			
sugarbeet, roots	0.01	0.05	1	76	-	-	-	-	PP62/0269
		0.1	3	90	85-94	5%			
sugarbeet, leaves	0.01	0.1	1	77	-	-	-	-	PP62/0269
		0.2	1	102	-	-			
		0.25	2	101	97-105	-			

Table 12. Validation results for desmethyl pirimicarb (R34836) using method PPRAM 15.

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference, method
Method version PPRAM 15/1									
Brussels sprouts	0.01	5	1	102	-	-	< 0.01 (1)	-	PP62/0523 alternative GC-column
Brussels sprouts, boiled	0.01	1	1	88	-	-	< 0.01 (1)	-	PP62/0523 alternative GC-column
head cabbage	0.01	10	1	88	-	-	< 0.01 (1)	-	PP62/0523 alternative GC-column
head cabbage, boiled	0.01	1	1	73	-	-	< 0.01 (1)	-	PP62/0523 alternative GC-column
water (washing, boiling)	0.01	1	3	93	80-117	23%	< 0.01-0.17 (3)	-	PP62/0523 alternative GC-column
lettuce	0.01	1	1	110	-	-	0.06-0.09 (2)	-	PP62/0523 alternative GC-column
		5	1	82	-	-			
Method version PPRAM 15/2 or RAM 015/02									
apple	0.02	0.02	2	83	74-91	-	< 0.02 (1)	-	PP62/0239 column clean-up omitted
		0.1	2	79	67-91	-			
		0.5	2	76	74-78	-			
		5	2	81	72-90	-			
tomato	0.02	0.02	2	85	83-87	-	< 0.02 (1)	-	PP62/0239 column clean-up omitted
		0.1	2	85	84-85	-			
		0.5	2	93	89-96	-			
		5	2	84	79-88	-			
broad beans	0.05	0.1	4	70	63-84	14%	< 0.05 (1)	-	PP62/0239 column clean-up omitted
		0.5	3	83	79-87	4.8%			
		5	3	82	78-84	3.9%			
potato	0.02	0.02	2	58	54-63	7.1%	< 0.02 (1)	-	PP62/0239 column clean-up omitted
		0.05	2	65	53-76	19%			
		0.1	2	69	68-70	-			
		0.5	2	72	69-75	-			

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference, method
Method version PPRAM 15/2 or RAM 015/02 with GC-MS detection									
apple	0.01	0.02	1	83	-	-	-	-	PP62/0406
		0.05	1	78	-	-			
		0.1	2	86	63-	-			
		0.25	2	108		-			
		0.5	1	80	77-83	-			
		1	2	79	-	-			
				92	88-96				
peach	0.01	0.02	1	83	-	-	-	-	PP62/0419
		0.05	1	98	-	-			
		0.1	2	87	80-94	-			
		0.2	1	93	-	-			
nectarine	0.01	0.1	2	93	89-96	-	-	-	PP62/0419
		0.2	1	95	-	-			
		0.25	1	91	-	-			
broccoli	0.01	0.02	2	93	87-99	-	-	-	PP62/0310
		0.1	1	80	-	-			
		0.2	1	87	-	-			
cauliflower	0.01	0.02	3	99	92-	11%	-	-	PP62/0310
		0.2	1	111		-			
				111	-				
broccoli	0.01	0.05	1	66	-	-	-	-	PP62/0311
		0.1	1	72	-	-			
cauliflower	0.01	0.1	1	108	-	-	-	-	PP62/0311
		0.2	1	94	-	-			
Brussels sprouts	0.01	0.1	2	75	73-76	-	-	-	PP62/0330
cauliflower	0.01	0.1	2	89	83-94	-	-	-	PP62/0333
sugarbeet, roots	0.01	0.05	1	84	-	-	-	-	PP62/0268
		0.1	5	86	71-	16%			
				102					
sugarbeet, leaves	0.01	0.1	4	96	87-	10%	-	-	PP62/0268
		1.0	1	110		-			
		2.0	1	80	-	-			
				82	-				
sugarbeet, roots	0.01	0.05	1	78	-	-	-	-	PP62/0269
		0.1	3	83	77-88	5%			
sugarbeet, leaves	0.01	0.1	1	79	-	-	-	-	PP62/0269
		0.2	1	118	-	-			
		0.25	2	98	92-	-			
				104					

Table 13. Validation results for desmethyl formamido pirimicarb (R34885, determined as R34836) using method PPRAM 15.

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference, method
Method version PPRAM 15/2 or RAM 015/02 with GC-NPD detection									
apple	0.02	0.1	2	82	74-89	-	< 0.02 (1)	-	PP62/0239 column clean-up omitted
tomato	0.02	0.1	2	88	87-89	-	< 0.02 (1)	-	PP62/0239 column clean-up omitted
broad beans	0.05	0.1	2	85	70-103	-	< 0.05 (1)	-	PP62/0239 column clean-up omitted
potato	0.02	0.1	2	69	68-70	-	< 0.02 (1)	-	PP62/0239 column clean-up omitted

Method PPRAM 38 (1978)

Method PPRAM 38 (1978) was developed for the determination of parent and its carbamate metabolites (R34836 and R34855) in milk, eggs, and animal tissues. The method was used in the feeding studies on cows and hens (feed, milk, eggs and tissues).

A full method description was available (Edwards and Dick, 1978: PP62/0229). Homogenised tissues were extracted with MeOH. Eggs were separated into whites and yolks. Filtered extracts from tissues, milk or eggs were evaporated to near dryness and resuspended and partitioned with hexane:0.1 M HCl (1:1). The samples were left to stand overnight. During this time any demethyl formamido pirimicarb (R34855) was converted into demethyl pirimicarb (R34836). Extracts from tissues and (optionally) eggs were additionally cleaned on silica gel columns, whereby pirimicarb and R34836 were eluted in different fractions. Pirimicarb and its carbamate metabolite (R34836) were determined by GC-NPD. The reported LOQ was 0.005 mg/kg in milk and 0.01 mg/kg in tissues for each analyte.

Extraction efficiency was verified in fat samples with incurred residues obtained from cow A1 from feeding study PP62/0537 (Edwards *et al*., 1978: PP62/0537). Fat samples were extracted with MeOH, chloroform or ACN:chloroform. Extracts were treated further as in method PPRAM 38. All three extracts showed that no residues were found in this fat sample (< 0.01 mg/kg for each analyte). Therefore no conclusions can be drawn on extraction efficiency.

Extraction efficiency was verified in milk samples spiked with unlabelled (1.00 mg/kg) and ^{14}C-labelled pirimicarb (0.027 mg/kg)(Edwards *et al*., 1978: PP62/0537). Milk samples were treated further, as in method PPRAM 38. Final extracts were analysed both by LSC and GC-NPD. GC-NPD revealed a recovery of 69% for the parent compound, while LSC revealed a recovery of 68% for total ^{14}C radioactivity.

GC-MS (electron ionisation) for parent and forR34836 was considered as a confirmation method (Edwards *et al*., 1978: PP62/0537). Extracts from milk with incurred residues obtained from cows in feeding study PP62/0537 were analysed both with GC-NPD and GC-MS (Table 17). There was good agreement between parent results (MS 75%-100% of NPD results), and less agreement between R34836 results (MS 55%−100% of NPD results).

No validation reports are available for method PPRAM 38. Reduced validation results from feeding studies are presented in Tables 14, 15 and 16.

Table 14. Validation results for parent using method PPRAM 38.

matrix	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference, method
cow feed (grass nuts)	-	150 300 400	2 2 8	96 95 91	94-98 94-96 83-100	- - 5.8%	-	-	PP62/0537 modification
cow milk	0.005	0.005 0.01 0.025 0.05 0.1 0.25	3 4 4 4 5 6	81 76 84 50 72 75	76-88 71-79 70-100 27-100 56-80 58-84	8.0% 4.7% 15% 39% 13% 13%	< 0.005- 0.01 (38)	-	PP62/0537
cow liver	0.01	0.01 0.05 0.1	1 1 1	79 73 72	- - -	- - -	< 0.01 (3)	-	PP62/0537
cow kidney	0.01	0.01 0.05 0.1	1 1 1	85 92 90	- - -	- - -	< 0.01 (3)	-	PP62/0537
cow muscle	0.01	0.01 0.02 0.05 0.1 0.2	2 1 3 3 1	108 67 90 75 75	79-136 - 79-105 60-93 -	- - 15% 22% -	< 0.01 - 0.01 (11)	-	PP62/0537

matrix	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference, method
		0.5	1	66	-	-			
cow fat	0.01	0.01	3	100	92-110	9.3%	< 0.01 (8)	-	PP62/0537
		0.05	4	78	57-99	27%			
		0.1	4	74	66-88	10%			
hen feed (basal diet)	0.01	2	2	80	79-72	-	< 0.01-0.05 (6)	-	PP62/0536 modification
		6	3	81	79-85	4.0%			
		20	2	84	82-87	-			
hen egg yolks	0.01	0.01	2	70	55-86	-	< 0.01 (14)	-	PP62/0536
		0.05	7	84	52-121	33%			
		0.10	4	63	50-75	22%			
hen egg whites	0.01	0.01	2	69	66-72	6.1%	< 0.01-0.02 (12)	-	PP62/0536
		0.02	4	96	87-105	10%			
		0.05	3	74	70-76	4.4%			
		0.1	3	87	76-102	15%			
hen muscle + skin with adhering fat	0.01	0.01	1	67	-	-	< 0.01 (4)	-	PP62/0536
		0.05	1	66	-	-			
		0.1	1	53	-	-			
hen liver	0.01	0.01	2	72	57-88	-	< 0.01 (4)	-	PP62/0536
		0.05	2	77	67-88	-			
		0.1	2	78	65-91	-			

Table 15. Validation results for metabolite R34836 using method PPRAM 38.

matrix	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference, method
cow milk	0.005	0.005	2	102	96-109	-	< 0.005-0.005 (38)	-	PP62/0537
		0.01	2	88	87-88	-			
		0.025	2	96	75-118	-			
		0.05	2	89	81-97	-			
		0.1	3	88	81-94	7.5%			
		0.25	4	90	78-105	11%			
cow liver	0.01	0.01	1	91	-	-	< 0.01 (3)	-	PP62/0537
		0.05	1	73	-	-			
cow kidney	0.01	0.01	1	64	-	-	< 0.01 (3)	-	PP62/0537
		0.05	1	86	-	-			
cow muscle	0.01	0.01	2	92	80-104	-	< 0.01 (11)	-	PP62/0537
		0.02	1	84	-	-			
		0.05	3	91	86-96	5.6%			
		0.1	1	85	-	-			
cow fat	0.01	0.01	3	99	92-104	6.2%	< 0.01 (8)	-	PP62/0537
		0.05	4	92	72-124	24%			
chicken feed (basal diet)	0.01	2	2	110	101-118	-	0.04 0.07 (6)	-	PP62/0536 modification
		6	3	109	98-120	10%			
		20	2	96	96-96	-			
hen egg yolks	0.01	0.01	1	93	-	-	< 0.01 (14)	-	PP62/0536
		0.05	5	96	80-115	16%			
		0.10	2	82	78-87	-			
hen egg whites	0.01	0.01	1	91	-	-	< 0.01-0.03 (12)	-	PP62/0536
		0.02	2	100	96-104	-			
		0.05	2	95	85-105	-			
		0.1	3	97	87-108	11%			
hen muscle + skin with adhering fat	0.01	0.01	1	95	-	-	< 0.02 (4)	-	PP62/0536
		0.05	1	88	-	-			
						-			
hen liver	0.01	0.01	2	74	69-80	-	< 0.01-0.01 (4)	-	PP62/0536
		0.05	2	57	49-65	-			

Table 16. Validation results for metabolite R34855 (measured as R34836) using method PPRAM 38.

matrix	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean	range	RSD_r	control samples mg/kg (n)	calibration	reference, method
cow milk	0.005	0.005	1	82	-	-	< 0.005- 0.005 (38	-	PP62/0537
		0.01	2	90	85-94	-			
		0.025	2	92	83-100	-			
		0.05	2	92	85-100	-			
		0.1	2	84	78-90	-			
		0.25	2	87	77-97	-			
cow liver	0.01	0.1	1	78	-	-	< 0.01 (3)	-	PP62/0537
cow kidney	0.01	0.1	1	93	-	-	< 0.01 (3)	-	PP62/0537
cow muscle	0.01	0.1	1	71	-	-	< 0.01 (11)	-	PP62/0537
		0.2	1	89	-				
		0.5	1	60	-				
cow fat	0.01	0.1	4	83	67-93	15%	< 0.01 (8)	-	PP62/0537
hen egg yolks	0.01	0.01	1	87	-	-	< 0.01 (14)	-	PP62/0536
		0.05	2	86	78-95	-			
		0.10	2	96	86-107	-			
hen egg whites	0.01	0.01	1	122	-	-	< 0.01- 0.03 (12)	-	PP62/0536
		0.02	2	85	76-94	-			
		0.05	1	101	-	-			
hen muscle + skin with adhering fat	0.01	0.1	1	73	-	-	< 0.02 (4)	-	PP62/0536
hen liver	0.01	0.1	2	96	95-97	-	< 0.01- 0.01 (4)	-	PP62/0536

Table 17. Confirmation of residues in extracts of cow milk.

	Parent			R34836		
Sample	GC-NPD	GC-MS	Ratio MS:NPD	GC-NPD	GC-MS	Ratio NPD:MS
A1 - day 5	0.004	0.004	1.0	0.030	0.020	0.67
A2 - day 5	0.024	0.022	0.92	0.066	0.051	0.77
A3 - day 5	0.005	0.005	1.0	0.037	0.026	0.70
D70 - day5	< 0.005	< 0.005	1.0	< 0.005	< 0.005	1.0
A1 - day 24	0.010	0.010	1.0	0.056	0.046	0.82
A2 - day 24	0.024	0.020	0.83	0.044	0.031	0.70
A3 - day 24	0.004	0.003	0.75	0.047	0.026	0.55
B6 - day 29	< 0.005	< 0.005	1.0	0.009	0.005	0.56
A3 - day 33	< 0.005	< 0.005	1.0	< 0.005	< 0.005	1.0

Method RAM 265 (1995-2004)

Several versions of method RAM 265 exist: RAM 265/01 (1995-1996), RAM 265/02 (1996-1997), RAM 265/03 (1997-1999), RAM 265/04 (1999-2004). The method was originally developed for the determination of pirimicarb and its carbamate metabolites (R34836, R34855) in pome fruit, root and tuber vegetables, fruiting vegetables (edible peel), brassica, leafy vegetables, cereal grains and straw. In the /02 version the method was extended to processed potato fractions. The /01 version of the method describes only GC-NPD quantification, while version /02, /03 and /04 describe both GC-NPD and HPLC-MS-MS quantification. Extraction and clean-up of the samples is the same in all four methods, and therefore validation results are interchangable as long as the same quantification technique is used. Method RAM 265 is intended for use as an enforcement-monitoring method (enforcement section).

Full method descriptions are available (Robinson, 1995: PP62/0837, Robinson and Patel, 1996, 1997, 1999: PP62/0834, PP62/0857, PP62/0219). Samples were extracted by maceration, filtered, evaporated, resuspended and further extracted and partitioned.

Pirimicarb and its carbamate metabolites (R34836 and R238177) were quantitated by GC-NPD. Calibration was carried out with external standards in acetone at 0–1 mg/L . The reported LOQ for each analyte was 0.01 mg/kg except for brassica and straw (LOQ 0.05 mg/kg) because of interference with R34836 and R238177.

A modification of the GC-NPD version of method RAM 265 was used in a processing study on tomatoes (PP62/0525). The DCM extract was immediately analysed for tomatoes, tomato peel, tomato juice and tomato canned.

A modification of the GC-NPD version of method RAM 265 was used in a storage stability study on cauliflower (PP62/0232) and a processing study on tomatoes (PP62/0525). For cauliflower, tomato puree and tomato ketchup, the extracts required further purification because of interference. The reported LOQ for each analyte was 0.01 mg/kg for tomato puree and tomato ketchup and 0.05 mg/kg for cauliflower.

For the HPLC-MS-MS version of the method, the reported LOQ for each analyte was 0.01 mg/kg.

Extraction efficiency of methanol was verified in the apple metabolism study (PP62/0265) and the lettuce metabolism study (PP62/0266). For apples the MeOH extract contained 88% TRR of which 39% TRR was found to chromatograph on TLC with carbamates: parent, R34836 and R34885. For lettuce the MeOH extract contained 88–91% TRR of which 63–72% TRR was identified as carbamates: parent, R34836, R34855, R238177 and R35140.

GC-NPD method RAM 265 was used in residue trials, storage stability studies or processing studies on apples, peach/nectarine, blackberries, currants, gooseberries, raspberries, strawberries, cabbage, cauliflower, cucumber, melon, peppers, tomatoes, lettuce, green beans with pods, snap beans, green peas with or without pods, potatoes, artichokes, asparagus and wheat (grains, straw).

Validation of GC-NPD method RAM 265 was described for apples, cabbage, tomatoes, lettuce, sugar beet, potatoes, processed potatoes, wheat (grains, straw) (Harradine, 1995: PP62/0241 and Robinson and Patel, 1996: PP62/0834). Results on processed potatoes are not summarized. All other results are shown in Tables 18, 20, and 22.

An independent laboratory validation for the GC-NPD method RAM 265 was described for apples, potatoes, and cereals (grain, straw, forage) (Coombe, 1996: PP62/0226). Results are shown in Tables 18, 20 and 22.

Validation of the additional clean-up step as used in the modified GC-NPD method was described for white cabbage and snap beans (Bolton, 1998: PP62/1534). Results are shown in Tables 18, 20, and 22.

HPLC-MS-MS method RAM 265 was used in storage stability studies, processing studies or rotational crop studies on oranges, apples, cherries, peaches, plums, strawberries, onion bulbs, cabbage, cauliflower, cucumber, courgette, melons (flesh, peel), peppers, tomatoes, kale, lettuce, mustard leaves, green beans with pods, green peas without pods, dry beans, dry broad beans, dry peas (seeds, straw), carrots, potatoes, sugarbeet (leaves, roots), turnip (tops, roots), artichoke, asparagus, barley (forage, grains, straw), maize (forage, grains, fodder), millet (forage, hay, straw, grain), wheat (grain, straw), oilseed rape, and sunflower seeds.

Validation of HPLC-MS-MS method RAM 265 was described for potatoes and processed potatoes (Robinson and Patel, 1996: PP62/0834). Results on processed potatoes are not summarized. All other results are shown in Tables 19, 21, and 23.

An independent laboratory validation for HPLC-MS-MS method RAM 265 was described for lettuce, runner beans with pods, maize grains, oilseed rape seeds (Wright, 1998: PP62/1100) under the laboratory specific method name CLE 38/229-01R. Calibration in the range 0.005-0.5 mg/L was equivalent to 0.005-0.5 mg/kg in the sample. Results are shown in Tables 19, 21 and 23.

An independent laboratory validation for the HPLC-MS-MS method RAM 265 was described for orange, orange pulp, plums, tobacco (Croucher, 2000: PP62/1101) and maize (cobs, grains,

fodder, forage) (Croucher, 2002: PP62/1186) under the laboratory specific method name CLE 38/229-03R. Results are shown in Tablea 19, 21 and 23.

An independent laboratory validation for HPLC-MS-MS method RAM 265 was described for tomatoes and oilseed rape seeds (Doran and McGuire, 2001: PP62/1012). Results are shown in Tables 19, 21 and 23.

Table 18. Validation results for parent using GC-NPD method RAM 265/01, /02, or /03.

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean, range	RSD_r	control samples mg/kg (n)	calibration	reference, method
apple	0.01	0.01 0.02 0.05 0.1 0.5	4 4 4 4 4	80 76-90 78 68-84 75 67-82 81 75-87 83 80-85	8.0% 9.0% 9.0% 6.5% 3.2%	< 0.01 (1)	in solvent 1x 6 points 0.01-1.0 mg/L linear, r > 0.99999	PP62/0241
cabbage	0.05	0.05 0.1 0.5	3 4 4	77 73-81 78 62-97 84 74-94	5.3% 20% 10%	< 0.05 (1)	in solvent 1x 6 points 0.01-1.0 mg/L linear, r > 0.99999	PP62/0241
tomato	0.01	0.01 0.02 0.05 0.1 0.5	4 4 4 4 4	80 70-94 80 74-89 85 59-97 88 91-95 81 80-84	13% 8.6% 21% 6.5% 2.3%	< 0.01 (1)	in solvent 1x 6 points 0.01-1.0 mg/L linear, r > 0.99999	PP62/0241
lettuce	0.01	0.01 0.02 0.05 0.1 0.5	4 3 4 4 4	76 70-81 82 80-86 84 81-86 84 81-91 87 74-100	8.0% 3.9% 2.8% 5.3% 13%	< 0.01 (1)	in solvent 1x 6 points 0.01-1.0 mg/L linear, r > 0.99999	PP62/0241
sugarbeet	0.01	0.01 0.02 0.05 0.1 0.5	4 4 4 4 4	91 76-99 73 66-83 79 77-83 76 74-79 69 59-79	12% 9.3% 3.6% 3.2% 13%	< 0.01 (1)	in solvent 1x 6 points 0.01-1.0 mg/L linear, r > 0.99999	PP62/0241
wheat, grains	0.01	0.01 0.02 0.05 0.1 0.5	4 4 4 4 4	86 80-95 84 75-90 84 79-86 80 78-82 83 80-87	8.4% 8.0% 3.7% 2.2% 4.0%	< 0.01 (1)	in solvent 1x 6 points 0.01-1.0 mg/L linear, r > 0.99999	PP62/0241
wheat, straw	0.05	0.05 0.10 0.20 0.50 1.0	4 4 4 4 4	96 87-106 84 69-94 92 88-98 92 88-97 93 88-96	9.5% 13% 4.8% 5.2% 3.7%	< 0.05 (1)	in solvent 1x 6 points 0.01-1.0 mg/L linear, r > 0.99999	PP62/0241
potato	0.01	0.01 0.05 0.1	3 1 1	79 69-85 83 - 72 -	11% - -	< 0.01 (1)	-	PP62/0834
apple	0.01	0.01 0.05	2 2	88 80-97 91 89-93	- -	< 0.3LOQ	in solvent 3x 6 points 0-0.2 mg/L linear r > 0.999	PP62/0226 ILV
potato	0.01	0.01 0.05	2 2	88 87-88 95 94-96	- -	< 0.3LOQ (2)	in solvent 3x 6 points 0-0.2 mg/L linear r > 0.999	PP62/0226 ILV
cereal grain	0.01	0.01 0.05	2 2	84 81-87 98 97-99	- -	< 0.3LOQ (2)	in solvent 3x 6 points 0-0.2 mg/L linear r > 0.999	PP62/0226 ILV

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean, range	RSD_r	control samples mg/kg (n)	calibration	reference, method
cereal straw	0.05	0.05 0.25	2 2	111 110-113 95 94-96	- -	< 0.3LOQ (2)	in solvent 3x 6 points 0-1.0 mg/L linear r > 0.99	PP62/0226 ILV
cereal forage	0.01	0.05 0.25	2 2	102 101-103 90 88-91	- -	< 0.3LOQ (2)	in solvent 3x 6 points 0-1.0 mg/L linear r > 0.99	PP62/0226 ILV
white cabbage	0.05	0.05 0.10 0.25 0.50 1.0	4 3 3 3 3	80 69-84 89 88-91 88 88-89 82 79-87 78 71-84	9.2% 1.7% 0.65% 5.3% 8.4%	< 0.3 LOQ (4)	in solvent 6 points 0-1 mg/L linear, r > 0.9999	PP62/1534 additional clean-up
snap bean	0.01	0.01 0.05 0.20 0.50 1.0	4 3 3 3 3	79 73-83 81 78-86 80 76-83 84 83-85 83 79-86	5.5% 5.4% 4.7% 1.4% 4.2%	< 0.3 LOQ (4)	in solvent 6 points 0-1 mg/L linear, r > 0.9999	PP62/1534 additional clean-up

Table 19. Validation results for parent using HPLC-MS-MS method RAM 265/02, /03, or /04.

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean, range	RSD_r	control samples mg/kg (n)	calibration	reference, method
potato	0.01	0.01 0.05 0.1	3 1 1	86 83-89 84 - 87 -	3.6% - -	< 0.01 (1)	in solvent 1x 5 points 0.01-0.5 mg/L linear r > 0.999	PP62/0834
lettuce	0.01	0.01 0.10 0.50	2 2 2	82 74-90 94 88-101 97 89-105	- - -	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, r^2 > 0.999	PP62/1100 ILV
runner beans (with pods)	0.01	0.01 0.10 0.50	2 2 2	90 80-100 80 78-82 93 85-101	- - -	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, r^2 > 0.999	PP62/1100 ILV
maize, grains	0.01	0.01 0.10 0.50	2 2 2	93 85-101 100 99-101 98 98-98	- - -	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, r^2 > 0.999	PP62/1100 ILV
oilseed rape, seed	0.01	0.01 0.10 0.50	2 2 2	87 85-89 84 83-85 88 85-90	- - -	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, r^2 > 0.999	PP62/1100 ILV
orange	0.01	0.01 0.10	5 5	90 85-99 90 86-93	5.9% 2.9%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, r^2 > 0.999	PP62/1101 ILV
orange, pulp	0.01	0.01 0.10	5 5	94 88-98 95 92-98	4.1% 2.5%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, r^2 > 0.999	PP62/1101 ILV

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean, range	RSD_r	control samples mg/kg (n)	calibration	reference, method
plums	0.01	0.01 0.10	5 5	95 84-107 94 90-97	9.2% 2.7%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1101 ILV
maize, whole cobs	0.01	0.01 0.10	5 5	85 83-87 88 85-90	1.8% 2.7%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1186 ILV
maize, grains	0.01	0.01 0.10	5 5	88 77-94 80 57-91	7.5% 18%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.99$	PP62/1186 ILV
maize, fodder	0.01	0.01 0.10	5 5	94 89-107 92 87-95	7.8% 3.8%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.9999$	PP62/1186 ILV
maize, forage	0.01	0.01 0.30	5 5	90 84-96 89 83-91	4.9% 3.8%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1186 ILV
tomato	0.01	0.01 0.10	5 5	97 93-100 95 93-99	3.2% 2.5%	< 0.3LOQ (2)	in solvent 1x 10 points 0.002-1 mg/L $1/x^2$ model $r^2 > 0.99$	PP62/1012 ILV
oilseed rape, seed	0.01	0.01 0.10	5 5	101 97-106 111 106-116	3.2% 3.9%	< 0.3LOQ (2)	in matrix 1x 10 points 0.002-1 mg/L $1/x^2$ model $r^2 > 0.99$	PP62/1012 ILV

Table 20. Validation results for metabolite R34836 using GC-NPD method RAM 265/01, /02, or /03.

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean, range	RSD_r	control samples mg/kg (n)	calibration	reference, method
apple	0.01	0.01 0.02 0.05 0.1 0.5	4 4 4 4 4	79 74-87 76 73-78 76 71-82 71 77-85 86 85-88	7.2% 3.5% 7.0% 4.2% 1.7%	< 0.01 (1)	in solvent 6 points 0.01-1.0 mg/L linear, r > 0.99999	PP62/0241
cabbage	0.05	0.05 0.1 0.5	3 4 4	99 98-100 91 71-114 94 87-104	1.0% 21% 7.7%	< 0.05 (1)	in solvent 6 points 0.01-1.0 mg/L linear, r > 0.9999	PP62/0241
tomato	0.01	0.01 0.02 0.05 0.1 0.5	4 4 4 4 4	88 84-94 89 86-92 82 65-91 95 90-99 98 92-106	5.2% 3.6% 14% 4.3% 6.1%	< 0.01 (1)	in solvent 6 points 0.01-1.0 mg/L linear, r > 0.9999	PP62/0241
lettuce	0.01	0.01 0.02 0.05 0.1 0.5	4 3 4 4 4	98 81-119 90 82-102 87 84-92 88 82-94 90 77-103	18% 12% 4.1% 5.6% 12%	< 0.01 (1)	in solvent 6 points 0.01-1.0 mg/L linear, r > 0.9999	PP62/0241

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean, range	RSD_r	control samples mg/kg (n)	calibration	reference, method
sugarbeet	0.01	0.01 0.02 0.05 0.1 0.5	4 4 4 4 4	92 88-97 82 78-88 85 74-90 86 82-89 86 83-92	4.6% 5.8% 8.7% 3.5% 5.0%	< 0.01 (1)	in solvent 6 points 0.01-1.0 mg/L linear, r > 0.99999	PP62/0241
wheat, grains	0.01	0.01 0.02 0.05 0.1 0.5	4 4 4 4 4	75 70-80 77 73-89 85 82-90 82 77-86 92 88-96	6.7% 10% 4.6% 4.6% 4.0%	< 0.01 (1)	in solvent 6 points 0.01-1.0 mg/L linear, r > 0.9999	PP62/0241
wheat, straw	0.05	0.05 0.10 0.20 0.50 1.0	4 4 4 3 4	96 82-113 89 70-101 100 91-119 102 97-107 103 96-108	17% 15% 13% 4.9% 4.9%	< 0.05 (1)	in solvent 6 points 0.01-1.0 mg/L linear, r > 0.9999	PP62/0241
potato	0.01	0.01 0.05 0.1	3 1 1	82 73-88 85 - 75 -	9.9% - -	< 0.01 (1)	-	PP62/0834
apple	0.01	0.01 0.05	2 2	91 90-92 98 96-100	- -	< 0.3LOQ	in solvent 3x 6 points 0-0.2 mg/L linear r > 0.99	PP62/0226 ILV
potato	0.01	0.01 0.05	2 2	108 106-110 101 99-103	- -	< 0.3LOQ (2)	in solvent 3x 6 points 0-0.2 mg/L linear r > 0.99	PP62/0226 ILV
cereal grain	0.01	0.01 0.05	2 2	98 92-103 112 109-115	- -	< 0.3LOQ (2)	in solvent 3x 6 points 0-0.2 mg/L linear r > 0.99	PP62/0226 ILV
cereal straw	0.05	0.05 0.25	2 2	88 80-86 92 91-93	- -	< 0.3LOQ (2)	in solvent 3x 6 points 0-1.0 mg/L linear r > 0.99	PP62/0226 ILV
cereal forage	0.01	0.05 0.25	2 2	93 91-95 91 89-93	- -	< 0.3LOQ (2)	in solvent 3x 6 points 0-1.0 mg/L linear r > 0.99	PP62/0226 ILV
white cabbage	0.05	0.05 0.10 0.25 0.50 1.0	4 3 3 3 3	79 69-91 91 78-99 88 85-91 75 65-83 78 71-84	11% 12% 3.5% 12% 8.5%	< 0.3 LOQ (4)	in solvent 6 points 0-1 mg/L linear, r > 0.9999	PP62/1534 additional clean-up
snap bean	0.01	0.01 0.05 0.20 0.50 1.0	4 3 3 3 3	94 83-104 86 78-102 83 81-86 86 77-91 85 77-94	11% 16% 3.2% 9.1% 10%	< 0.3 LOQ (4)	in solvent 6 points 0-1 mg/L linear, r > 0.9999	PP62/1534 additional clean-up

Table 21. Validation results for metabolite R34836 using HPLC-MS-MS method RAM 265/02, /03, or /04.

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean, range	RSD_r	control samples mg/kg (n)	calibration	reference, method
potato	0.01	0.01 0.05 0.10	3 1 1	83 75-90 77 - 81 -	9.1% - -	< 0.01 (1)	in solvent 1x 5 points 0.01-0.5 mg/L linear r > 0.999	PP62/0834
runner beans (with pods)	0.01	0.01 0.10 0.50	2 2 2	88 79-98 84 82-85 96 84-109	- - -	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1100 ILV
lettuce	0.01	0.01 0.10 0.50	2 2 2	91 87-95 94 86-101 100 93-107	- - -	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1100 ILV
maize, grains	0.01	0.01 0.10 0.50	2 2 2	87 79-95 95 90-100 104 101-107	- - -	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1100 ILV
oilseed rape, seed	0.01	0.01 0.10 0.50	2 2 2	94 86-92 92 90-93 90 89-91	- - -	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1100 ILV
orange	0.01	0.01 0.10	5 5	93 86-98 95 92-96	5.0% 1.8%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1101 ILV
orange, pulp	0.01	0.01 0.10	5 5	94 90-97 101 96-105	2.8% 3.2%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1101 ILV
plums	0.01	0.01 0.10	5 5	86 79-92 90 88-92	6.6% 2.1%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1101 ILV
maize, whole cobs	0.01	0.01 0.10	5 5	90 84-97 91 90-93	6.2% 1.3%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.9999$	PP62/1186 ILV
maize, grains	0.01	0.01 0.10	5 5	94 87-100 84 65-94	5.0% 16%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.99$	PP62/1186 ILV
maize, fodder	0.01	0.01 0.10	5 5	95 88-102 91 87-93	5.2% 2.5%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1186 ILV
maize, forage	0.01	0.01 0.30	5 5	95 89-100 93 90-95	4.5% 2.1%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.99$	PP62/1186 ILV

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean, range	RSD_r	control samples mg/kg (n)	calibration	reference, method
tomato	0.01	0.01 0.10	5 5	89 87-90 90 88-91	1.3% 1.4%	< 0.3LOQ (2)	in solvent 1x 10 points 0.002-1 mg/L $1/x^2$ model $r^2 > 0.99$	PP62/1012 ILV
oilseed rape, seed	0.01	0.01 0.10	5 5	91 90-92 95 91-99	1.2% 3.1%	< 0.3LOQ (2)	in matrix 1x 10 points 0.002-1 mg/L $1/x^2$ model $r^2 > 0.99$	PP62/1012 ILV

Table 22. Validation results for metabolite R238177 using GC-NPD method RAM 265/01, /02, or /03.

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean, range	RSD_r	control samples mg/kg (n)	calibration	reference, method
apple	0.01	0.01 0.02 0.05 0.1 0.5	4 4 4 4 4	76 71-85 72 69-75 76 70-80 79 74-82 82 80-85	8.8% 3.6% 5.7% 5.0% 2.9%	< 0.01 (1)	in solvent 6 points 0.01-1.0 mg/L linear, r > 0.99999	PP62/0241
cabbage	0.05	0.05 0.1 0.5	3 4 4	86 84-88 86 65-114 92 84-101	2.4% 24% 7.7%	< 0.05 (1)	in solvent 6 points 0.01-1.0 mg/L linear, r > 0.9999	PP62/0241
tomato	0.01	0.01 0.02 0.05 0.1 0.5	4 4 4 4 4	79 70-99 86 82-88 94 83-102 98 92-100 95 90-101	17% 3.1% 8.7% 4.1% 4.8%	< 0.01 (1)	in solvent 6 points 0.01-1.0 mg/L linear, r > 0.9999	PP62/0241
lettuce	0.01	0.01 0.02 0.05 0.1 0.5	4 3 4 4 4	81 71-92 86 85-88 84 81-87 87 81-93 89 76-101	11% 1.8% 3.2% 6.1% 12%	< 0.01 (1)	in solvent 6 points 0.01-1.0 mg/L linear, r > 0.9999	PP62/0241
sugarbeet	0.01	0.01 0.02 0.05 0.1 0.5	4 4 4 4 4	90 78-100 86 85-88 84 74-88 87 83-90 86 82-93	12% 1.5% 8.1% 3.3% 5.4%	< 0.01 (1)	in solvent 6 points 0.01-1.0 mg/L linear, r > 0.99999	PP62/0241
wheat, grains	0.01	0.01 0.02 0.05 0.1 0.5	4 4 4 4 4	98 89-104 90 85-97 86 80-90 78 75-80 86 83-91	6.5% 5.7% 4.9% 3.0% 4.0%	< 0.01 (1)	in solvent 6 points 0.01-1.0 mg/L linear, r > 0.9999	PP62/0241
wheat, straw	0.05	0.05 0.10 0.20 0.50 1.0	4 4 4 3 4	101 97-104 90 67-100 98 94-108 96 93-101 96 91-100	3.1% 17% 6.6% 4.5% 3.9%	< 0.05 (1)	in solvent 6 points 0.01-1.0 mg/L linear, r > 0.9999	PP62/0241
potato	0.01	0.01 0.05 0.1	3 1 1	82 71-88 87 - 73 -	12% - -	< 0.01 (1)	-	PP62/0834
apple	0.01	0.01 0.05	2 2	84 75-93 98 96-99	- -	< 0.3LOQ	in solvent 3x 6 points 0-0.2 mg/L linear r > 0.99	PP62/0226 ILV

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean, range	RSD_r	control samples mg/kg (n)	calibration	reference, method
potato	0.01	0.01 0.05	2 2	76 74-78 102 98-107	- -	< 0.3LOQ (2)	in solvent 3x 6 points 0-0.2 mg/L linear r > 0.99	PP62/0226 ILV
cereal grain	0.01	0.01 0.05	2 2	88 84-92 94 94-94	- -	< 0.3LOQ - 0.0054 (2)	in solvent 3x 6 points 0-0.2 mg/L linear r > 0.99	PP62/0226 ILV
cereal straw	0.05	0.05 0.25	2 2	88 86-91 90 88-92	- -	< 0.3LOQ (2)	in solvent 3x 6 points 0-1.0 mg/L linear r > 0.99	PP62/0226 ILV
cereal forage	0.01	0.05 0.25	2 2	86 84-89 87 86-88	- -	< 0.3LOQ - 0.0054	in solvent 3x 6 points 0-1.0 mg/L linear r > 0.99	PP62/0226 ILV
white cabbage	0.05	0.05 0.10 0.25 0.50 1.0	4 3 3 3 3	75 67-79 95 91-101 86 84-90 78 76-81 76 72-79	7.4% 5.8% 3.7% 3.2% 4.6%	< 0.3 LOQ (4)	in solvent 6 points 0-1 mg/L linear, r > 0.999	PP62/1534 additional clean-up
snap bean	0.01	0.01 0.05 0.20 0.50 1.0	4 3 3 3 3	89 73-100 79 78-80 78 71-82 77 73-79 79 74-87	13% 1.3% 8.1% 4.2% 8.9%	< 0.3 LOQ (4)	in solvent 6 points 0-1 mg/L linear, r > 0.999	PP62/1534 additional clean-up

Table 23. Validation results for metabolite R238177 using HPLC-MS-MS method RAM 265/02, /03 or /04.

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean, range	RSD_r	control samples mg/kg (n)	calibration	reference, method
potato	0.01	0.01 0.05 0.1	3 1 1	78 74-80 79 - 84 -	4.1% - -	< 0.01 (1)	in solvent 1x 5 points 0.01-0.5 mg/L linear r > 0.999	PP62/0834
runner beans (with pods)	0.01	0.01 0.10 0.50	2 2 2	96 86-106 85 82-88 94 83-106	- - -	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1100 ILV
lettuce	0.01	0.01 0.10 0.50	2 2 2	88 85-90 92 88-97 100 93-108	- - -	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1100 ILV
maize, grains	0.01	0.01 0.10 0.50	2 2 2	82 72-92 93 90-96 104 104-105	- - -	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1100 ILV
oilseed rape, seed	0.01	0.01 0.10 0.50	2 2 2	90 82-97 88 83-94 88 85-92	- - -	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1100 ILV

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean, range	RSD_r	control samples mg/kg (n)	calibration	reference, method
plums	0.01	0.01 0.10	5 5	90 79-101 90 88-95	8.7% 3.1%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1101 ILV
orange	0.01	0.01 0.10	5 5	91 88-97 92 88-94	3.9% 2.7%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1101 ILV
orange, pulp	0.01	0.01 0.10	5 5	90 86-93 92 88-94	3.1% 2.7%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1101 ILV
maize, whole cobs	0.01	0.01 0.10	5 5	90 84-92 88 82-93	3.8% 5.4%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1186 ILV
maize, grains	0.01	0.01 0.10	5 5	91 86-96 84 68-92	4.6% 13%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.99$	PP62/1186 ILV
maize, fodder	0.01	0.01 0.10	5 5	89 86-92 88 87-90	3.4% 1.6%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.999$	PP62/1186 ILV
maize, forage	0.01	0.01 0.30	5 5	90 85-96 88 84-90	4.3% 3.1%	< 0.3LOQ (2)	in solvent 1x 7 points 0.005-0.50 mg/kg, linear, $r^2 > 0.99$	PP62/1186 ILV
tomato	0.01	0.01 0.10	5 5	81 79-83 83 82-85	2.2% 1.8%	< 0.3LOQ (2)	in solvent 1x 10 points 0.002-1 mg/L $1/x^2$ model $r^2 > 0.99$	PP62/1012 ILV
oilseed rape, seed	0.01	0.01 0.10	5 5	88 86-92 87 82-91	3.3% 4.7%	< 0.3LOQ (2)	in matrix 1x 10 points 0.002-1 mg/L $1/x^2$ model $r^2 > 0.99$	PP62/1012 ILV

Method RAM 277 (2000-2004)

Method RAM 277/02 (2000-2004) was developed for the determination of parent and its carbamate metabolite R34836 in water.

A full method description is available (Harradine, 2000: PP62/0552). Pirimicarb and R34836 were determined by HPLC with fluorescence detection . The reported LOQ was 0.1 µg/L for each analyte.

Validation of method RAM 277/02 was described for tap water (Harradine, 2000: PP62/0552). Results are shown in table 24 and table 25.

Table 24. Validation results for parent using method RAM 277/02.

matrix	reported LOQ µg/L	spike level µg/L	n	% recovery mean range	RSD_r	control samples µg/L (n)	calibration	reference, method
tap water	0.1	0.1 0.2 0.5 1.0	4 2 2 2	94 86-100 109 107-111 109 109-109 94 89-98	6.7% - - -	< 0.1 (1)	in solvent 5 points 0.1-1.0 µg/L linear, r > 0.999	PP62/0552
apple wash water	0.1	0.1 0.5	5 5	82 77-90 79 62-85	7.6% 12%	< 0.1 (2)	-	PP62/0982
plum wash water	0.1	0.1	2	83 78-87	-	< 0.1 (1)	-	PP62/1389
tomato wash water blanch water	0.1	0.1	6	97 89-106	7.8%	< 0.1-0.23 (2)	-	PP62/1392

Table 25. Validation results for metabolite R34836 using method RAM 277/02.

matrix	reported LOQ µg/L	spike level µg/L	n	% recovery mean range	RSD_r	control samples µg/L (n)	calibration	reference, method
tap water	0.1	0.1 0.2 0.5 1.0	4 2 2 2	89 83-95 101 98-104 98 98-99 98 97-100	6.6% - - -	< 0.1 (1)	in solvent 5 points 0.1-1.0 mg/L linear, r > 0.999	PP62/0552
apple wash water	0.1	0.1 0.5	5 5	83 77-89 81 63-86	6.5% 12%	< 0.1 (2)	-	PP62/0982
plum wash water	0.1	0.1	2	75 70-80	-	< 0.1 (1)	-	PP62/1389
tomato wash water blanch water	0.1	0.1	4	88 80-96	10	< 0.1 (2)	-	PP62/1392

Method RAM 319 (2000-2002)

Method RAM 319/01 (2000-2002) was developed for the determination of parent and its carbamate metabolites (R34836, R34855 and R238177) in citrus fruits, pome fruits, stone fruits, small fruits and berries, bulb vegetables, brassica vegetables, fruiting vegetables (edible/inedible peel), leafy vegetables, legume vegetables, root and tuber vegetables, stem vegetables, cereal grains, fodders and straws and oil seeds. The method was actually used in residue trials or storage stability studies on oilseed rape (seeds) and kale.

A full method description is available (Kwiatkowski, 2000: PP62/0220). Pirimicarb and its carbamate metabolites (R34836 and R238177) were determined by HPLC-MS-MS. The reported LOQ for each analyte was 0.01 mg/kg, except for fodders and straw for which it has been set at 0.05 mg/kg.

Validation of method RAM 319/01 was described for lemon, apple, plums, grapes, leeks, cabbage, melon, peppers, spinach, beans, potato, celery, winter wheat (grains, straw), linseed, red fescue grass (Hill, 1999: PP62/0237). Validation results are shown in Tables 26 – 28.

Table 26. Validation results for parent using method RAM 319/01.

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean range	RSD_r	Control samples mg/kg (n)	calibration	Reference, method
lemon, whole fruit	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	98 95-102 92 89-94 110 107-113 86 72-99 102 98-106	3.0% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean	range	RSD_r	Control samples mg/kg (n)	calibration	Reference, method
apple	0.01	0.01	4	97	91-102	4.8%	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237
		0.1	2	90	90-91	-			
		0.5	2	97	95-99	-			
		2.0	2	100	99-100	-			
		5.0	2	88	83-92	-			
plums	0.01	0.01	4	101	95-104	4.0%	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237
		0.1	2	89	87-91	-			
		0.5	2	110	105-114	-			
		2.0	2	112	107-116	-			
		5.0	2	102	98-107	-			
grapes	0.01	0.01	4	100	98-103	2.4%	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237
		0.1	2	98	97-99	-			
		0.5	2	94	90-97	-			
		2.0	2	104	97-111	-			
		5.0	2	91	89-93	-			
leeks	0.01	0.01	4	90	86-96	4.9%	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237
		0.1	2	84	84-85	-			
		0.5	2	103	103-103	-			
		2.0	2	92	92-92	-			
		5.0	2	102	101-102	-			
cabbage	0.01	0.01	4	108	104-113	3.8%	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237
		0.1	2	97	96-98	-			
		0.5	2	106	106-107	-			
		2.0	2	105	103-107	-			
		5.0	2	106	105-107	-			
melon, whole fruit	0.01	0.01	4	93	92-95	2.2%	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237
		0.1	2	88	86-89	-			
		0.5	2	89	86-92	-			
		2.0	2	94	94-94	-			
		5.0	2	90	90-90	-			
peppers	0.01	0.01	4	104	102-108	2.5%	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237
		0.1	2	102	101-102	-			
		0.5	2	113	110-116	-			
		2.0	2	106	102-109	-			
		5.0	2	114	113-115	-			
spinach	0.01	0.01	4	106	104-108	1.6%	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237
		0.1	2	96	96-97	-			
		0.5	2	115	115-115	-			
		2.0	2	108	108-109	-			
		5.0	2	110	110-110	-			
green beans with pods	0.01	0.01	4	101	97-110	6.1%	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237
		0.1	2	100	99-101	-			
		0.5	2	109	109-109	-			
		2.0	2	74	72-75	-			
		5.0	2	94	90-99	-			
potato	0.01	0.01	4	102	97-106	4.1%	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237
		0.1	2	99	95-103	-			
		0.5	2	102	101-102	-			
		2.0	2	106	105-106	-			
		5.0	2	106	105-107	-			
celery	0.01	0.01	4	93	90-96	3.4%	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237
		0.1	2	87	82-92	-			
		0.5	2	86	85-87	-			
		2.0	2	103	103-103	-			
		5.0	2	90	87-93	-			
wheat, grains	0.01	0.01	4	89	84-92	3.8%	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237
		0.1	2	102	102-103	-			
		0.5	2	98	98-98	-			
		2.0	2	104	102-105	-			
		5.0	2	128	126-129	-			

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean range	RSD_r	Control samples mg/kg (n)	calibration	Reference, method
wheat, straw	0.05	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	95 86-101 98 90-105 98 91-104 100 94-107 106 103-108	6.8% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237
linseed	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	105 97-125 98 96-100 113 109-117 98 96-100 104 104-105	13% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237
grass, red fescue	0.05	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	101 94-105 86 82-89 105 104-106 92 89-94 101 97-105	4.9% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.99$	PP62/0237

Table 27. Validation results for metabolite R34836 using method RAM 319.

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean range	RSD_r	control samples mg/kg (n)	calibration	reference, method
lemon, whole fruit	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	98 94-103 99 97-101 109 108-110 99 98-100 108 107-110	5.3% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
apple	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	99 97-101 95 92-98 94 92-96 102 100-104 84 79-88	1.6% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
plums	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	93 89-98 90 90-91 108 102-113 111 107-115 96 87-104	5.0% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
grapes	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	107 99-116 98 96-100 92 92-93 108 104-111 96 96-96	7.7% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
leeks	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	88 83-94 84 83-85 106 105-106 94 93-96 100 98-101	5.1% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
cabbage	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	102 101-103 96 94-98 106 105-107 104 103-105 108 108-108	0.9% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
melon, whole fruit	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	96 85-105 85 85-85 84 81-86 92 92-93 94 93-94	8.7% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
peppers	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	104 102-107 97 96-98 114 112-116 120 119-122 106 106-107	2.3% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237

spinach	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	106 102-112 94 92-96 111 111-111 107 106-108 108 107-110	4.5% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
green beans with pods	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	107 100-115 90 89-91 104 102-106 104 100-109 102 98-106	6.5% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
potato	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	96 94-98 94 94-94 102 101-104 100 99-101 105 103-107	1.8% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
celery	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	84 78-90 89 87-91 85 82-88 106 104-108 99 98-110	6.7% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
wheat, grains	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	99 93-106 90 88-92 94 92-96 106 106-106 90 90-90	7.3% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
wheat, straw	0.05	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	86 74-110 112 107-116 98 83-113 90 82-98 118 96-141	19% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
linseed	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	100 97-102 91 89-93 112 110-114 96 96-97 105 104-106	2.1% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
grass, red fescue	0.05	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	99 96-104 86 78-93 100 94-105 93 92-94 100 99-100	3.6% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237

Table 28. Validation results for metabolite R238177 using method RAM 319.

Commodity	reported LOQ mg/kg	spike level mg/kg	n	% recovery mean range	RSD_r	control samples mg/kg (n)	calibration	reference, method
lemon, whole fruit	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	98 96-101 100 98-102 112 111-114 98 97-99 93 78-108	2.1% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
apple	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	98 94-101 93 92-94 86 83-90 100 99-101 82 77-88	3.6% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
plums	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	102 94-111 92 91-94 106 102-111 110 107-113 98 94-103	6.8% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237

grapes	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	106 104-108 92 84-100 91 90-92 106 102-110 96 94-97	1.6% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
leeks	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	88 87-91 82 82-83 100 99-101 94 94-95 100 100-101	2.0% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
melon, whole fruit	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	94 91-96 90 89-90 83 82-84 91 91-91 94 94-95	2.4% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
peppers	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	104 97-108 101 101-102 110 110-110 116 113-118 109 107-111	4.6% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
cabbage	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	102 99-106 98 93-102 106 105-108 104 101-107 106 106-107	3.2% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
spinach	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	102 96-108 91 90-92 106 106-107 105 104-106 106 105-106	4.9% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
green beans with pods	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	101 98-103 95 94-96 88 88-89 100 96-104 102 101-104	2.2% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
potato	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	98 90-106 96 95-97 102 101-103 104 104-105 103 102-104	6.8% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
celery	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	85 81-88 86 85-88 82 82-83 104 102-106 95 92-98	3.5% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
wheat, grains	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	95 93-98 100 98-101 98 96-99 107 103-111 96 91-100	2.5% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
wheat, straw	0.05	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	94 86-100 104 102-105 89 84-94 96 81-98 112 108-117	7.1% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
linseed	0.01	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	103 95-111 93 92-94 111 108-114 100 100-100 106 106-106	7.3% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237
grass, red fescue	0.05	0.01 0.1 0.5 2.0 5.0	4 2 2 2 2	99 94-105 91 80-102 97 95-99 98 98-98 95 94-96	4.6% - - - -	< 0.3LOQ (1)	5 points, 0.002-1 mg/L, linear, $r^2 > 0.999$	PP62/0237

Method RAM 360 (2001)

Method RAM 360/02 (2001)was developed for the determination of parent and its carbamate metabolites (R34836, R34855 and R238177) in water.

A full method description is available (Robinson, 2001: PP62/0948). Pirimicarb and its carbamate metabolites were determined by GC-MS (EI) . The reported LOQ for each analyte was 0.1 µg/L.

Validation of method RAM 360/02 was described for river water, sea water, ground water and drinking water (Robinson, 2001: PP62/0948). The results for drinking water were summarized in Tables 29 – 31. Matrix effects in drinking water caused 0-4% enhancement of the standard signals. The matrix effects were not considered enough by the study author to warrant the use of matrix matched standards.

Table 29. Validation results for parent using method RAM 360/02.

matrix	reported LOQ µg/L	spike level µg/L	n	% recovery mean range	RSD_r	control samples µg/L (n)	calibration	reference, method
drinking water	0.1	0.1 1.0	5 5	88 83-93 93 88-99	4.3% 5.9%	< 0.1 µg/L (1)	6 points, in solvent 0.005-1.0 mg/L; linear, $r^2 > 0.999$	PP62/0948
kale, wash water boiling water steaming water	1	1 10 50 100	3 2 4 3	88 86-91 88 84-92 94 75-102 92 73-108	3.0% - 13% 19%	< 1 (8)	-	PP62/1290

Table 30.Validation results for metabolite R34836 using method RAM 360/02.

matrix	reported LOQ µg/L	spike level µg/L	n	% recovery mean range	RSD_r	control samples µg/L (n)	calibration	reference, method
drinking water	0.1	0.1 1.0	5 5	84 79-89 90 87-97	4.4% 5.5%	< 0.1 µg/L (1)	6 points, in solvent 0.005-1.0 mg/L; linear, $r^2 > 0.999$	PP62/0948
kale, wash water boiling water steaming water	10	10 50 100	2 4 3	80 79-82 90 71-101 90 72-107	- 15% 19%	< 10 (8)	-	PP62/1290

Table 31. Validation results for metabolite R238177 using method RAM 360/02.

matrix	reported LOQ µg/L	spike level µg/L	n	% recovery mean range	RSD_r	control samples µg/L (n)	calibration	reference, method
drinking water	0.1	0.1 1.0	5 5	86 84-92 91 88-95	3.7% 3.4%	< 0.1 µg/L (1)	6 points, in solvent 0.005-1.0 mg/L; linear, $r^2 > 0.999$	PP62/0948
kale, wash water boiling water steaming water	1	1 10 50 100	3 2 4 3	81 73-88 89 89-89 94 80-101 94 81-107	9.3% - 10% 14%	< 1 (8)	-	PP62/1290

Stability of pesticide residues in stored analytical samples

The Meeting received data on the stability of residues in various crops.

Pomefruits

Apples were fortified with a mixture of pirimicarb, R34836 and R238177 at 0.1 mg/kg each (Miles, 1998: PP62/0248). The samples were stored at -18 °C for periods of up to 13 months. Duplicate samples were analyzed for pirimicarb, R34836 and R238177 by GC-NPD method RAM 265/02. The reported LOQ was 0.01 mg/kg for each analyte.

Results are shown in Table 32. Samples were corrected for mean concurrent method recoveries analysed within the run. Uncorrected results are not available. Samples were not corrected for matrix interferences (< 0.01 mg/kg for each analyte). Results show that residues are stable for up to 13 months when stored at -18 °C.

Table 32. Storage stability data for apples stored at -18 °C.

Analyte:	parent				R34836				R238177			
Level:	0.1 mg/kg				0.1 mg/kg				0.1 mg/kg			
Storage time (months)	% remaining [a] (n=2) mean	range	RSD_r	concur recov	% remaining [a] (n=2) mean	range	RSD_r	concur recov	% remaining [a] (n=2) mean	range	RSD_r	concur recov
0	102	99-105	-	82-88	102	99-106	-	78-86	106	101-110	-	72-85
3	100	96-104	-	85-88	94	86-101	-	92-93	90	84-97	-	86-86
5	78	75-82	-	66-96	92	87-98	-	67-99	92	88-97	-	61-87
9	106	100-111	-	98-100	106	102-110	-	94-97	102	99-105	-	91-92
13	102	101-103	-	92-98	98	97-98	-	96-103	95	93-97	-	90-102

concur recov = concurrent recovery

a Corrected for mean concurrrent recovery analysed within the run; uncorrected results are not available.

Brassica vegetables (head cabbages and flowerhead cabbages)

Study 1. Cauliflowers were fortified with a mixture of 0.1 mg/kg pirimicarb, 0.1 mg/kg R34836 and 0.1 mg/kg R238177 (Bolton, 1998: PP62/0236). The samples were stored at -18 °C for periods of up to 12 months. Duplicate samples were analyzed for pirimicarb, R34836 and R238177 using GC-NPD method RAM 265/02 and /03 with an additional clean-up step. The reported LOQ was 0.05 mg/kg for each analyte.

Results are shown in Table 33. Samples were not corrected for concurrent method recoveries nor for matrix interferences (< 0.3 LOQ). Results show that residues are stable for up to 12 months when stored at -18 °C.

Table 33. Storage stability data for cauliflower stored at -18 °C.

Analyte:	parent				R34836				R238177			
Level:	0.1 mg/kg				0.1 mg/kg				0.1 mg/kg			
Storage time (months)	% remaining (n=2) mean	range	RSD_r	concur recov	% remaining (n=2) mean	range	RSD_r	concur recov	% remaining (n=2) mean	range	RSD_r	concur recov
0 [a]	87	85-90	-	84-92	77	76-77	-	82-86	87	82-91	-	87-89
3 [a]	75	74-76	-	80-83	74	71-77	-	80-81	72	70-75	-	71-78
6 [b]	82	76-89	-	85-109	95	92-97	-	102-103	80	74-85	-	76-106
9 [b]	87	86-89	-	93-96	91	90-92	-	92-96	112	111-113	-	87-93
12 [b]	71	57-85	-	80-83	76	68-84	-	76-89	90	80-99	-	76-91

concur recov = concurrent recovery

a Analysed by method RAM 265/02 with extra cleanup

b Analysed by method RAM 265/03 with extra cleanup

Study 2. Cabbages were fortified with a mixture of 0.5 mg/kg pirimicarb, 0.1 mg/kg R34836 and 0.1 mg/kg R238177 (Hill and Miles, 1997: PP62/0247). The samples were stored at -18 °C for periods of up to 12 months. Duplicate samples were analyzed for pirimicarb, R34836 and R238177 using GC-NPD method RAM 265/01 and /02. The reported LOQ was 0.05 mg/kg for each analyte.

Results are shown in Table 34. Samples were corrected for mean concurrent method recoveries analysed within the run. Uncorrected results are not available. Samples were not corrected for matrix interferences (< 0.05 mg/kg for each analyte). Results show that residues are stable for up to 12 months when stored at -15 °C.

Table 34. Storage stability data for cabbage stored at -15 °C.

Analyte:	parent				R34836				R238177			
Level:	0.5 mg/kg				0.1 mg/kg				0.1 mg/kg			
Storage time (months)	% remaining [a] (n=2) mean	range	RSD_r	concur recov	% remaining [a] (n=2) mean	range	RSD_r	concur recov	% remaining [a] (n=2) mean	range	RSD_r	concur recov
0 [b]	102	99-104	-	76-77	108	108-109	-	108-112	102	98-107	-	69-75
1 [b]	91	89-92	-	87-88	105	103-107	-	80-102	96	95-96	-	78-100
2 [b]	84	82-86	-	82-92	94	94-94	-	79-90	94	90-99	-	94-111
7 [c]	88	88-89	-	97-100	114	113-114	-	99-102	89	88-89	-	95-98
10 [c]	94	92-96	-	86-87	108	107-109	-	107-110	84	82-86	-	76-84
18 [c]	96	95-98	-	82-85	106	104-109	-	89-94	88	85-90	-	82-83

concur recov = concurrent recovery

a Corrected for mean concurrrent recovery analysed within the run; uncorrected results are not available.

b Analysed by method RAM 265/01

c Analysed by method RAM 265/02

Fruiting vegetables, cucurbits

Cucumbers were fortified with a mixture of 0.5 mg/kg pirimicarb and 0.5 mg/kg R34855 or with a mixture of 0.5 mg/kg pirimicarb and 0.5 mg/kg R34836 (Benet, 1995: PP62/0242). The samples were stored at -18 °C for periods of up to 18 months. Duplicate samples were analyzed for pirimicarb and R34836 using GC-NPD method RAM 015/02. Metabolite R34885 is converted into R34836 during extraction. The reported LOQ was 0.05 mg/kg for each analyte.

Results are shown in Table 35. Samples were not corrected for concurrent method recoveries nor for matrix interferences (< 0.05 mg/kg for each analyte). Results show that residues are stable for up to 18 months when stored at -18 °C.

Table 35. Storage stability data for cucumbers stored at -18 °C.

Analyte:	parent				R34885				R34836			
Level:	0.5 mg/kg				0.5 mg/kg				0.5 mg/kg			
Storage time (months)	% remaining (n=4) mean	range	RSD_r	concur recov	% remaining (n=2) mean	range	RSD_r	concur recov	% remaining (n=2) mean	range	RSD_r	concur recov
0	106	100-118	7.9%	80-98	111	106-116	-	92-96	90	90-90	-	96-110
9	86	72-98	14%	87-90	81	78-84	-	67-68	98	98-98	-	64-76
12	78	74-80	3.2%	76-87	90	86-94	-	67-76	83	82-84	-	62-75
18	86	84-92	4.4%	91-112	101	100-102	-	70-74	76	74-78	-	92-104

concur recov = concurrent recovery

Fruiting vegetables other than cucurbits

Tomatoes were fortified with a mixture of 0.5 mg/kg pirimicarb, 0.1 mg/kg R34836 and 0.1 mg/kg R238177 (Miles, 1997: PP62/0232). The samples were stored at -18 °C for periods of up to 12 months. Duplicate samples were analyzed for pirimicarb, R34836 and R238177 using GC-NPD method RAM 265/01 and /02. The reported LOQ was 0.01 mg/kg for each analyte.

Results are shown in Table 36. Samples were corrected for mean concurrent method recoveries analysed within the run. Uncorrected results are not available. Samples were not corrected for matrix interferences (< 0.01 mg/kg for each analyte). Results show that residues are stable for up to 12 months when stored at -18 °C.

Table 36. Storage stability data for tomatoes stored at -18 °C.

Analyte:	parent				R34836				R238177			
Level:	0.5 mg/kg				0.1 mg/kg				0.1 mg/kg			
Storage time (months)	% remaining [a] (n=2) mean	range	RSD_r	concur recov	% remaining [a] (n=2) mean	range	RSD_r	concur recov	% remaining [a] (n=2) mean	range	RSD_r	concur recov
0 [b]	90	86-94	-	100-105	100	100-100	-	112-115	90	90-90	-	98-99
3 [b]	100	92-108	-	71-91	95	90-110	-	68-90	90	80-100	-	69-92
6 [c]	104	100-108	-	89-94	110	110-110	-	89-101	105	100-110	-	82-91
9 [c]	107	104-110	-	83-85	115	110-120	-	85-86	105	100-110	-	75-87
12 [c]	101	100-102	-	80-82	110	110-110	-	84-91	100	100-100	-	78-84

concur recov = concurrent recovery

a Corrected for mean concurrrent recovery analysed within the run; uncorrected results are not available.

b Analysed by method RAM 265/01

c Analysed by method RAM 265/02

Leafy vegetables including Brassica leafy vegetables

Iceberg lettuce was fortified with a mixture of 0.5 mg/kg pirimicarb, 0.1 mg/kg R34836 and 0.1 mg/kg R238177 (Harradine, 1996: PP62/0244). The samples were stored at -18 °C for periods of up to 12 months. Duplicate samples were analyzed for pirimicarb, R34836 and R238177 using GC-NPD method RAM 265/01 and /02. The reported LOQ was 0.01 mg/kg for each analyte.

Results are shown in Table 37. Samples were corrected for mean concurrent method recoveries analysed within the run. Uncorrected results are not available. Samples were not corrected for matrix interferences (< 0.01 mg/kg for each analyte). Results show that residues are stable for up to 12 months when stored at -18 °C.

Table 37. Storage stability data for iceberg lettuce stored at -18 °C.

Analyte:	parent				R34836				R238177			
Level:	0.5 mg/kg				0.1 mg/kg				0.1 mg/kg			
Storage time (months)	% remaining [a] (n=2) mean	range	RSD_r	concur recov	% remaining [a] (n=2) mean	range	RSD_r	concur recov	% remaining [a] (n=2) mean	range	RSD_r	concur recov
0 [b]	99	98-100	-	83-85	110	100-120	-	86-87	105	100-110	-	81-81
3 [b]	92	90-94	-	93-100	95	90-100	-	96-107	100	100-100	-	93-95
6 [b]	101	100-102	-	81-90	100	100-100	-	90-95	105	100-110		79-85
9 [c]	94	94-94	-	87-94	100	100-100	-	95-96	95	90-100	-	86-86
12 [c]	91	84-98	-	75-83	110	100-120	-	72-90	105	100-110	-	72-89

concur recov = concurrent recovery

a Corrected for mean concurrrent recovery analysed within the run; uncorrected results are not available.

b Analysed by method RAM 265/01

c Analysed by method RAM 265/02

Legume vegetables

Snap beans were fortified with a mixture of 0.1 mg/kg pirimicarb, 0.1 mg/kg R34836 and 0.1 mg/kg R238177 (Bolton, 1998: PP62/0235). The samples were stored at -18 °C for periods of up to 12 months. Duplicate samples were analyzed for pirimicarb, R34836 and R238177 using GC-NPD method RAM 265/02 and /03. The reported LOQ was 0.01 mg/kg for each analyte.

Results are shown in Table 38. Samples were not corrected for concurrent method recoveries nor for matrix interferences (up to 0.0059 mg/kg for parent, up to 0.013 mg/kg for R34836, < 0.3LOQ for R238177). Results show that residues are stable for up to 12 months when stored at -18 °C.

Table 38. Storage stability data for snap beans stored at -18 °C.

Analyte:	parent				R34836				R238177			
Level:	0.1 mg/kg				0.1 mg/kg				0.1 mg/kg			
Storage time (months)	% remaining (n=2) mean	range	RSD_r	concur recov	% remaining (n=2) mean	range	RSD_r	concur recov	% remaining (n=2) mean	range	RSD_r	concur recov
0 [a]	78	76-80	-	74-78	86	84-89	-	87-88	93	90-96	-	93-95
3 [a]	69	69-69	-	71-81	62	58-66	-	74-84	62	60-63	-	68-79
6 [b]	93	89-96	-	91-99	103	99-107	-	106-112	104	102-106	-	93-107
9 [b]	87	83-91	-	90-93	100	94-106	-	99-99	104	99-110	-	107-108
12 [b]	70	64-76	-	63-76	79	72-85	-	82-97	81	76-87	-	75-91

concur recov = concurrent recovery

a Analysed by method RAM 265/02

b Analysed by method RAM 265/03

Root and tuber vegetables

Study 1. Potatoes were fortified with a mixture of 0.1 mg/kg pirimicarb, 0.1 mg/kg R34836 and 0.1 mg/kg R238177 (Hill and Miles, 1997: PP62/0246). The samples were stored at -15 °C for periods of up to 12 months. Duplicate samples were analyzed for pirimicarb, R34836 and R238177 using GC-NPD method RAM 265/01 and /02. The reported LOQ was 0.01 mg/kg for each analyte.

Results are shown in Table 39. Samples were corrected for mean concurrent method recoveries analysed within the run. Uncorrected results are not available. Samples were not corrected for matrix interferences (< 0.01 mg/kg for each analyte). Results show that residues are stable for up to 12 months when stored at -15 °C.

Table 39. Storage stability data for potatoes stored at -15 °C.

Analyte:	parent				R34836				R238177			
Level:	0.1 mg/kg				0.1 mg/kg				0.1 mg/kg			
Storage time (months)	% remaining [a] (n=2) mean	range	RSD_r	concur recov	% remaining [a] (n=2) mean	range	RSD_r	concur recov	% remaining [a] (n=2) mean	range	RSD_r	concur recov
0 [b]	104	104-105	-	72-77	105	103-107	-	79-82	104	102-106	-	78-79
1 [b]	100	97-104	-	69-72	98	96-99	-	78-82	101	99-103	-	71-75
3 [b]	87	84-90	-	75-81	90	89-92	-	89-95	92	92-93	-	73-79
7 [c]	92	91-94	-	77-79	92	92-92	-	80-86	98	97-98	-	75-79
9 [c]	94	91-98	-	70-70	88	84-92	-	79-82	97	94-100	-	71-74
12 [c]	92	85-100	-	64-74	96	86-105	-	77-82	100	90-110	-	71-77

concur recov = concurrent recovery

a Corrected for mean concurrrent recovery analysed within the run; uncorrected results are not available.

b Analysed by method RAM 265/01

c Analysed by method RAM 265/02

Stalk and stem vegetables

Study 1. Artichokes were fortified with a mixture of 0.1 mg/kg pirimicarb, 0.1 mg/kg R34836 and 0.1 mg/kg R238177 (Bolton, 1998: PP62/0233). The samples were stored at -18 °C for periods of up to 12 months. Duplicate samples were analyzed for pirimicarb, R34836 and R238177 by GC-NPD using method RAM 265/02 and from 6 months onwards using method RAM 265/03. The reported LOQ was 0.01 mg/kg for each analyte.

Results are shown in Table 40. Samples were not corrected for concurrent method recoveries nor for matrix interferences (up to 0.0055 mg/kg for parent, < 0.3LOQ for R34836, up to 0.0030 mg/kg for R238177). Results show that residues are stable for up to 12 months at -18 °C.

Because of matrix interferences, the valid LOQ has been adjusted to 0.0055/0.3 = 0.02 mg/kg for parent; however this presents no problem as the fortification level is 0.1 mg/kg.

Table 40. Storage stability data for artichokes stored at -18 °C.

Analyte:	parent				R34836				R238177			
Level:	0.1 mg/kg				0.1 mg/kg				0.1 mg/kg			
Storage time (months)	% remaining (n=2) mean	range	RSD_r	concur recov	% remaining (n=2) mean	range	RSD_r	concur recov	% remaining (n=2) mean	range	RSD_r	concur recov
0 [a]	87	84-89	-	71-71	96	93-100	-	90-91	100	96-105	-	88-92
3 [a]	78	74-83	-	95-98	73	66-80	-	78-79	72	64-79	-	76-82
6 [b]	81	81-82	-	80-82	79	76-82	-	84-86	82	82-82	-	87-87
9 [b]	79	78-81	-	78-81	85	83-87	-	81-86	83	80-87	-	76-79
12 [b]	77	70-85	-	76-84	84	76-92	-	93-98	78	71-85	-	85-92

concur recov = concurrent recovery

a Analysed by method RAM 265/02

b Analysed by method RAM 265/03

Study 2. Asparagus was fortified with a mixture of 0.1 mg/kg pirimicarb, 0.1 mg/kg R34836 and 0.1 mg/kg R238177 (Bolton, 1998: PP62/0234). The samples were stored at -18 °C for periods of up to 12 months. Samples were analyzed for pirimicarb, R34836 and R238177 using GC-NPD using method RAM 265/02 and /03. The reported LOQ was 0.01 mg/kg for each analyte.

Results are shown in Table 41. Results show that residues are stable for up to 12 months at -18 °C.

Because of matrix interferences the valid LOQ for parent, R34836, and R238177 has been increased to 0.0069/0.3 = 0.03 mg/kg, 0.013/0.3 = 0.05 mg/kg and 0.0042/0.3=0.02 mg/kg; however this presents no problem as the fortification level is 0.1 mg/kg.

Table 41. Storage stability data for artichokes stored at -18 °C.

Analyte:	parent				R34836				R238177			
Level:	0.1 mg/kg				0.1 mg/kg				0.1 mg/kg			
Storage time (months)	% remaining (n=2) mean	range	RSD_r	concur recov	% remaining (n=2) mean	range	RSD_r	concur recov	% remaining (n=2) mean	range	RSD_r	concur recov
0 [a]	80	77-83	-	76-83	89	89-89	-	88-95	104	101-107	-	94-105
3 [a]	86	81-92	-	80-84	74	71-77	-	71-79	72	68-75	-	65-73
6 [b]	99	95-103	-	91-102	92	90-94	-	87-112	90	90-90	-	81-106
9 [b]	87	83-90	-	80-85	103	100-106	-	90-95	96	93-100	-	91-91
12 [b]	90	89-90	-	86-90	109	101-118	-	87-104	94	93-96	-	89-93

concur recov = concurrent recovery

a Analysed by method RAM 265/02

b Analysed by method RAM 265/03

Cereal grains

Wheat grains and straw, obtained from residue trial study PP62/0487, were fortified with a mixture of 0.1 mg/kg pirimicarb, R34836 and R238177 for grains and 0.5 mg/kg for straw (Patel, 1997: PP62/0231). The samples were stored at -18°C for periods of up to 12 months. Samples were analyzed for pirimicarb, R34836 and R238177 using GC-NPD method RAM 265/01 and /02. The reported LOQ was 0.01 mg/kg for grains and 0.05 mg/kg for straw.

Results are shown in Table 42. Samples were corrected for mean concurrent method recoveries analysed within the run. Uncorrected results are not available. Results show that residues are stable for up to 12 months when stored at -18°C.

Table 42. Storage stability data for wheat (grains, straw) stored at -18 °C.

Matrix	Analyte: / Level:	parent grains: 0.1 mg/kg straw: 0.5 mg/kg		R34836 grains: 0.1 mg/kg straw: 0.5 mg/kg		R238177 grains: 0.1 mg/kg straw: 0.5 mg/kg	
	Storage time (months)	%remaining [a] (n=2) mean, range, RSD_r	concur recov	%remaining [a] (n=2) mean, range, RSD_r	concur recov	%remaining [a] (n=2) mean, range, RSD_r	concur recov
wheat grains	0 [a]	90 80-100 -	86-92	90 80-100 -	85-92	90 80-100 -	82-88
	3 [a]	105 100-110 -	82-86	100 100-10 -	86-87	100 100-100 -	80-82
	6 [b]	90 90-90 -	98-101	90 90-90 -	100-106	95 90-100 -	93-98
	9 [b]	90 90-90 -	79-86	85 80-90 -	87-96	90 90-90 -	81-88
	12 [b]	85 80-90 -	82-87	95 90-100 -	77-83	85 80-90 -	76-84
wheat straw	0 [a]	109 108-110 -	94-98	105 104-106 -	96-98	100 100-100 -	94-108
	3 [a]	91 86-96 -	91-94	91 86-96 -	98-102	89 86-92 -	90-96
	6 [b]	123 120-126 -	94-98	131 128-134 -	112-118	116 114-118 -	98-102
	9 [b]	94 94-94 -	97-98	110 110-110 -	110-113	98 96-100 -	109-110
	12 [b]	96 92-100 -	84-85	104 98-110 -	94-96	97 90-104 -	84-84

concur recov = concurrent recovery

a Corrected for mean concurrrent recovery analysed within the run; uncorrected results are not available.

b Analysed by method RAM 265/01

c Analysed by method RAM 265/02

Oilseeds

Seeds from oilseed rape were fortified with a mixture of 0.2 mg/kg pirimicarb, R34836 and R238177 (Hill, 2002: PP62/1216). The samples were stored at -18 °C for periods of up to 18 months. Samples were analyzed for pirimicarb, R34836 and R238177 by GC-NPD using method RAM 319/01. The reported LOQ was 0.01 mg/kg for grains and 0.05 mg/kg for straw.

Results are shown in Table 43. Results show that residues are stable for up to 18 months when stored at -18 °C.

Table 43. Storage stability data for oilseed rape (seeds) stored at -18 °C.

Analyte: / Level:	parent 0.2 mg/kg				R34836 0.2 mg/kg				R238177 0.2 mg/kg			
Storage time (months)	% remaining [a] (n=3) mean	range	RSD_r	concur recov	% remaining [a] (n=3) mean	range	RSD_r	concur recov	% remaining [a] (n=2) mean	range	RSD_r	concur recov
0	100	100-100	0.0%	97-99	98	95-100	2.9%	101-102	100	95-105	5.0%	97-98
3	100	95-105	5.0%	95-98	100	95-105	5.0%	95-99	103	100-105	2.8%	93-100
6	100	100-100	0.0%	94-94	103	100-105	2.8%	94-100	102	100-105	2.8%	96-98
10	93	90-100	6.2%	97-100	88	85-95	6.5%	95-102	87	85-90	3.3%	109-110
12	98	90-105	7.8%	90-98	100	95-110	8.7%	93-98	100	95-105	5.0%	90-96
18	105	100-110	4.8%	87-103	102	100-105	2.8%	97-104	105	100-110	4.8%	96-97

concur recov = concurrent recovery

a Corrected for mean concurrrent recovery analysed within the run; uncorrected results are not available.

Animal commodities

Milk was fortified with a mixture of 0.1-10 mg/kg pirimicarb and 0.1-10 mg/kg R34836 (Edwards *et al*., 1978: PP62/0537). The samples were stored at room temperature for 5 d in the dark. Milk samples were analyzed for pirimicarb and R34836 by GC-NPD using method PPRAM 38. The reported LOQ was 0.005 mg/kg in milk.

Results are shown in Table 44. Results show that residues are stable for up to 5 d when stored at room temperature.

Table 44. Storage stability data for milk stored at room temperature for 5 days.

Spike level	parent				R34836			
mg/kg	% remaining (n=1) mean	range	RSD_r	concur recov	% remaining (n=1) mean	range	RSD_r	concur recov
0.01	110	-	-	-	96	-	-	-
0.1	72	-	-	-	72	-	-	-
1.0	79	-	-	-	80	-	-	-
10	82	-	-	-	92	-	-	-

concur recov = concurrent recovery

Milk samples were also fortified with 0.1 mg/kg pirimicarb, 0.1 mg/kg R34836 or 0.1 mg/kg R34855 (Edwards *et al*., 1978: PP62/0537). The samples were stored at -14 °C for periods of up to 24 months. Samples were analyzed for pirimicarb, R34836 and R34855 (analysed as R34836) by GC-NPD using method PPRAM 38. The reported LOQ was 0.005 mg/kg in milk.

Results are shown in Table 45. Results show that residues are stable for up to 24 months when stored at -14 °C.

Table 45. Storage stability data for milk stored at -14 °C for 24 months.

Spike level	parent				R34836				R34855 (analysed as R34836)			
mg/kg	% remaining[a] (n=1) mean	range	RSD_r	concur recov	% remaining[a] (n=1) mean	range	RSD_r	concur recov	% remaining[a] (n=1) mean	range	RSD_r	concur recov
0.1	79	-	-	81	83	-	-	103	73	-	-	103

concur recov = concurrent recovery

a Corrected for mean concurrrent recovery analysed within the run; uncorrected results are not available.

USE PATTERN

Information on registered uses of pirimicarb was provided to the meeting by the Netherlands and the manufacturers, together with labels for representative uses in Europe. These representative uses relating to the crops under consideration are summarised in the following tables.

Table 46. Representative uses of pirimicarb on fruit crops in Europe (from labels provided).

Crop	Country	Form	Application				PHI (days)
			Method	Max No	Conc kg ai/hL	Rate kg ai/ha	
Apple	France	50 WG	foliar		0.0375	0.375	21
Apple	Netherlands	50 WG	foliar	2	0.025	0.25-0.375	7
Apple	Portugal	50 WG	foliar		0.025-0.0375		15
Apricot	France	50 WG	foliar		0.0375	0.375	14
Apricot	Germany	50 WG	foliar	2[2]		0.125[3]	pre-blossom
Berryfruit	Czech Republic	50 WG	foliar	2[1]		0.25	7
Blackberries	Netherlands	50 WG	foliar	2	0.025	0.25-0.3	7
Cherries	France	50 WG	foliar		0.0375	0.375	21
Cherries	Germany	50 WG	foliar	2[2]		0.125[3]	14
Cherries	Netherlands	50 WG	foliar	2	0.025	0.25-0.375	7
Citrus	Portugal	50 WG	foliar	2	0.0375	0.375	15
Citrus	Spain	50 WG	foliar		0.05		7
Currants	France	50 WG	foliar	1	0.0375		14

Crop	Country	Form	Application				PHI (days)
			Method	Max No	Conc kg ai/hL	Rate kg ai/ha	
Currants	Netherlands	50 WG	foliar	2	0.025	0.25-0.3	7
Fruit	Belgium	50 WG	foliar			0.25	7
Fruit (ex citrus)	Spain	50 WG	foliar		0.05		3 (7d topfruit)
Gooseberry	Netherlands	50 WG	foliar	2	0.025	0.25-0.3	7
Peach	France	50 WG	foliar		0.0375	0.375	14
Peach	Germany	50 WG	foliar	2[2]		0.125[3]	pre-blossom
Peach	Netherlands	50 WG	foliar	2	0.025	0.25-0.375	7
Peach	Portugal	50 WG	foliar		0.025-0.0375		15
Pear	France	50 WG	foliar		0.0375	0.375	15
Pear	Netherlands	50 WG	foliar	2	0.025	0.25-0.375	7
Pear	Portugal	50 WG	foliar		0.025-0.0375	0.25-0.375	15
Plums	France	50 WG	foliar		0.0375	0.375	14
Plums	Germany	50 WG	foliar	2[2]		0.125[3]	pre-blossom
Plums	Netherlands	50 WG	foliar	2	0.025	0.25-0.375	7
Pome fruit	Czech Republic	50 WG	foliar	2[1]	0.025-0.038	0.25	7
Pome fruit	Germany	50 WG	foliar	3[2]		0.125[3]	21
Quince	France	50 WG	foliar		0.0375	0.375	15
Raspberries	Netherlands	50 WG	foliar	2	0.025	0.25-0.3	7
Rubus spp	France	50 WG	foliar	1	0.0375		14
Stone fruits	Czech Republic	50 WG	foliar	2[1]	0.025-0.038	0.25	7 (14d plums)
Strawberry	Belgium	50 WG	foliar			0.2	7
Strawberry	France	50 WG	foliar			0.375	15
Strawberry	Netherlands	50 WG	foliar	2	0.025	0.125-0.15	7

1) minimum spray interval of 7-10 days

2) minimum spray interval of 10 days

[3]) application rate expressed on a per ha/metre crop height basis

Table 47. Representative uses of pirimicarb on vegetable crops in Europe (from labels provided).

Crop	Country	Form	Application				PHI	Notes
			Method	Max No	Conc kg ai/hL	Rate kg ai/ha		
Artichoke	France	50 WG	foliar			0.375	7	
Asparagus	France	50 WG	foliar			0.375	200	
Beans	Portugal	50 WG	foliar		0.025-0.0375		7	
Beans (field)	Czech Republic	50 WG	foliar	2[1]		0.25	3	no pods
Beans (field)	Belgium	50 WG	foliar			0.2	7	
Beans (field)	Germany	50 WG	foliar	2		0.15	35	
Beans (field)	Netherlands	50 WG	foliar	4		0.25	7	
Beans (green)	France	50 WG	foliar			0.375	7	
Beans (pole)	Belgium	50 WG	foliar			0.25	7	
Beans	Netherlands	50 WG	foliar	4		0.25	7	Yard-long
Beans, common	Netherlands	50 WG	foliar	2		0.25	7	
Beans, French	Netherlands	50 WG	foliar	4		0.25	4	dwarf
Beet	Belgium	50 WG	foliar			0.175	7	
Beet	Czech Republic	50 WG	foliar	2[1]		0.25	7	
Beet, Fodder	Germany	50 WG	foliar	4		0.15	28	
Beet, Fodder	Netherlands	50 WG	foliar	2		0.2		

Crop	Country	Form	Application				PHI	Notes
			Method	Max No	Conc kg ai/hL	Rate kg ai/ha		
Beet, red	Germany	50 WG	foliar	2[3]		0.15	14	
Beet, red	Netherlands	50 WG	foliar	2		0.25	7	
Beet, Sugar	Czech Republic	50 WG	foliar	2[1]		0.25	7	
Beet, Sugar	Germany	50 WG	foliar	4		0.15	28	
Beet, Sugar	Netherlands	50 WG	foliar	2		0.2		
Beet, Sugar	Spain	50 WG	foliar		0.05		3	
Beetroot	Belgium	50 WG	foliar			0.2	7	
Brassica vegetables	Czech Republic	50 WG	foliar	2[1]		0.25	3	
Brassica vegetables	Germany	50 WG	foliar	3[2]		0.125	7	
Brassica vegetables	France	50 WG	foliar			0.375	7	cabbages, cauliflowers
Broad bean	Belgium	50 WG	foliar			0.2	7	
Broad bean	Netherlands	50 WG	foliar	4		0.25	4	
Broccoli	Netherlands	50 WG	foliar	2		0.25	7	
Brussels sprouts	Netherlands	50 WG	foliar	2		0.25	4	
Brussels sprouts	Portugal	50 WG	foliar		0.025-0.0375		7	
Cabbage Group	Belgium	50 WG	foliar			0.2	7	
Cabbage, Chinese	Netherlands	50 WG	foliar	2		0.25	7	
Cabbages, Head	Netherlands	50 WG	foliar	2		0.25	7	
Carrot	Belgium	50 WG	foliar			0.2	7	
Carrot	France	50 WG	foliar			0.375	7	
Carrot	Germany	50 WG	foliar	2[3]		0.15	7	
Carrot	Netherlands	50 WG	foliar	2		0.25	7	
Cauliflower	Netherlands	50 WG	foliar	2		0.25	7	
Celeriac	Belgium	50 WG	foliar			0.2	7	
Celeriac	Netherlands	50 WG	foliar	2		0.25	7	
Chervil	Netherlands	50 WG	foliar	4		0.25	7	
Chicory (leaves)	Belgium	50 WG	foliar			0.2	7	
Chicory (leaves)	France	50 WG	foliar	2		0.375	7	
Chicory (leaves)	Netherlands	50 WG	foliar	2		0.25	7	
Chicory (roots)	Belgium	50 WG	foliar			0.2		
Chicory (roots)	Netherlands	50 WG	foliar	2		0.25		
Cucurbits	Spain	50 WG	foliar		0.05		7	
Cucumber	Czech Republic	50 WG	foliar	2[1]	0.025-0.038	0.25	7	
Cucumber	France	50 WG	foliar	2		0.375	3	
Cucumber (protected)	Netherlands	50 WG	foliar	2	0.025	0.25-0.37	1	
Cucumber	Portugal	50 WG	foliar		0.025-0.0375		15	
Cucumber (protected)	Belgium	50 WG	foliar			0.25	3	
Egg plant	France	50 WG	foliar			0.375	3	
Egg plant (protected)	Netherlands	50 WG	foliar	2	0.025	0.25-0.37	1	
Egg plant (protected)	Belgium	50 WG	foliar			0.25	3	
Endive	Netherlands	50 WG	foliar	2		0.25	14-28[4]	
Endive	Netherlands	50 WG	foliar	2		0.25	7	

Crop	Country	Form	Application				PHI	Notes
			Method	Max No	Conc kg ai/hL	Rate kg ai/ha		
Garlic	Czech Republic	50 WG	foliar	2[1]		0.15-0.25	14	
Gherkin	France	50 WG	foliar	2		0.375	3	
Gherkin	Netherlands	50 WG	foliar	2	0.025	0.05-0.37	1	
Gherkin (outdoor)	Belgium	50 WG	foliar			0.2	7	
Gherkin (protected)	Belgium	50 WG	foliar			0.25	1	
Horseradish	Germany	50 WG	foliar	2[3]		0.15	7	
Kale	Germany	50 WG	foliar	2		0.125	7	
Kale, curly	Netherlands	50 WG	foliar	2		0.25	7	
Kohlrabi	Netherlands	50 WG	foliar	2		0.25	7	
Leek	Netherlands	50 WG	foliar	4		0.25	7	
Legume vegetables	Germany	50 WG	foliar	3[2]		0.125-0.25[6]	3	
Legumes, ex peas, beans	Czech Republic	50 WG	foliar	2[1]		0.25	3 14d forage	
Lentil	France	50 WG	foliar			0.375	14	
Lettuce	Czech Republic	50 WG	foliar	2[1]		0.25	7-10[5]	
Lettuce (field)	Portugal	50 WG	foliar		0.025-0.0375		7	
Lettuce (protected)	Portugal	50 WG	foliar	3	0.025-0.0375		15	
Lettuce (winter)	Spain	50 WG	foliar		0.05		14	
Lettuces	France	50 WG	foliar	2		0.375	14	
Lettuces (protected)[6]	Netherlands	50 WG	foliar	2		0.25	14-28[4]	
Lettuces (outdoor)[6]	Netherlands	50 WG	foliar	2		0.25	7	
Lettuces[7]	Germany	50 WG	foliar	3[2]		0.125	7	
Lettuces (outdoor)	Belgium	50 WG	foliar	1		0.2	7	
Melon	France	50 WG	foliar			0.375	3	
Melon (protected)	Netherlands	50 WG	foliar	2	0.025	0.25-0.37	3	
Melon (protected)	Belgium	50 WG	foliar			0.25	3	
Onion	Czech Republic	50 WG	foliar	2[1]		0.15-0.25	14	
Parsnip	Germany	50 WG	foliar	2[3]		0.15	7	
Pea	Belgium	50 WG	foliar			0.2	7	
Pea	France	50 WG	foliar			0.375	7	
Pea, field	Czech Republic	50 WG	foliar	2[1]		0.25	3 14d forage	
Pea, field	Netherlands	50 WG	foliar	4		0.25	7	
Pea, garden	Czech Republic	50 WG	foliar	2[1]		0.25	3	
Pea, green	Netherlands	50 WG	foliar	4		0.25	4	
Peppers	Czech Republic	50 WG	foliar	2[1]	0.025-0.038	0.25	3	
Peppers	France	50 WG	foliar			0.375	3	
Peppers (protected)	Belgium	50 WG	foliar			0.25	3	
Peppers, sweet (protected)	Netherlands	50 WG	foliar	2	0.025	0.25-0.37	1	

Crop	Country	Form	Application				PHI	Notes
			Method	Max No	Conc kg ai/hL	Rate kg ai/ha		
Peppers, chilli (protected)	Netherlands	50 WG	foliar	2	0.025	0.25-0.37	1	
Potato	Belgium	50 WG	foliar			0.2	7	
Potato	Czech Republic	50 WG	foliar	2[1]		0.25	7	
Potato	France	50 WG	foliar			0.25	21	
Potato	Germany	50 WG	foliar	2		0.15	7	
Potato	Netherlands	50 WG	foliar	2		0.25	7	
Potato	Portugal	50 WG	foliar		0.025-0.0375		15	
Pulses	Czech Republic	50 WG	foliar	2[1]		0.25	3 14d forage	
Radish	Belgium	50 WG	foliar			0.2	7	
Radishes	Netherlands	50 WG	foliar	2		0.25	7	
Root vegetables	Czech Republic	50 WG	foliar	2[1]		0.25	7	
Scarole	France	50 WG	foliar	2		0.375	14	
Spinach	Czech Republic	50 WG	foliar	2[1]		0.25	7	
Spinach	France	50 WG	foliar			0.375	7	
Spinach	Netherlands	50 WG	foliar	2		0.25	14-28[4]	
Spinach	Netherlands	50 WG	foliar	2		0.25	7	
Spinach	Spain	50 WG	foliar		0.05	0.3-0.5	14	
Squash, summer (protected)	Netherlands	50 WG	foliar	2	0.025	0.25-0.37	1	
Swede	Netherlands	50 WG	foliar	2		0.25	7	
Sweet corn	France	50 WG	foliar			0.2	7	
Sweet corn	Netherlands	50 WG	foliar	2		0.25	7	
Tomato	Czech Republic	50 WG	foliar	2[1]	0.025-0.038	0.25	3	
Tomato	France	50 WG	foliar			0.375	3	
Tomato (protected)	Netherlands	50 WG	foliar	2	0.025	0.25-0.37	1	
Tomato (protected)	Belgium	50 WG	foliar			0.25	3	
Turnip tops	Netherlands	50 WG	foliar	2		0.25	7	
Vegetables, ex cucurbits	Spain	50 WG	foliar		0.05		3	
Witloof , roots	Netherlands	50 WG	foliar	2		0.25		
Witloof, sprouts (protected)	Netherlands	50 WG	foliar	2		0.25	7	
Zucchini	France	50 WG	foliar	2		0.375	3	

AH = after harvesting (fern treatment)

[1]) minimum spray interval of 7-10 days

[2]) minimum spray interval of 10 days

[3]) minimum spray interval of 10-14 days

[4]) PHI of 14 days from 1 March to 1 November, 28 days between 1 November and 1 March

[5]) PHI of 7 days from 1 March to 1 November, 10 days between 1 November and 1 March

[6]) including head lettuce, crisphead lettuce and lambs lettuce

[7]) including endive, chicory, spinach

Table 48. Representative uses of pirimicarb on cereal and oilseed crops in Europe (from labels provided).

Crop	Country	Form	Application				PHI	Notes
			Method	Max No	Conc kg ai/hL	Rate kg ai/ha		
Barley	Netherlands	50 WG	foliar	2		0.125	14	
Barley	Portugal	50 WG	foliar			0.125	up to flowering	
Cereals	Belgium	50 WG	foliar			0.125	7	
Cereals	Czech Republic	50 WG	foliar	2[1]		0.15	to BBCH 83-85 (PHI 14 days)	
Cereals	France	50 WG	foliar			0.125	35	
Cereals	Germany	50 WG	foliar	2		0.1-0.15	35	higher rate <15 C
Cereals	Spain	50 WG	foliar		0.05		45	
Clover	Czech Republic	50 WG	foliar	2[1]		0.15-0.25	14	
Maize	France	50 WG	foliar			0.2	80 (60d forage)	to end of flowering
Oat	Portugal	50 WG	foliar			0.125	up to flowering	
Oil seeds	Czech Republic	50 WG	foliar	2[1]		0.15-0.25	14	
Pea, (fodder)	Germany	50 WG	foliar	2		0.15	35	
Poppy, oilseed	Czech Republic	50 WG	foliar	2[1]		0.15-0.25		
Rape, oilseed	Czech Republic	50 WG	foliar	2[1]		0.15-0.25		
Rape, oilseed	France	50 WG	foliar			0.25	21	
Sorghum	France	50 WG	foliar			0.2	80 (60 d forage)	to end of flowering
Sunflower	Czech Republic	50 WG	foliar	2[1]		0.15-0.25		
Sunflower	France	50 WG	foliar			0.25	21	to end of flowering
Wheat	Netherlands	50 WG	foliar	2		0.125	14	
Wheat	Portugal	50 WG	foliar			0.125	15	

[1]) minimum spray interval of 7-10 days

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials involving pirimicarb for the following crops and commodities.

Crop	Commodity	Countries	Table
orange	whole fruit, pulp	Italy, Spain	49-50
mandarin	whole fruit, pulp	Spain	51-52
apples	whole fruit	France, Germany, Italy, Spain, UK	53
peaches, nectarines	whole fruit, flesh[1]	France, Italy, Spain	54-55
plums	whole fruit, flesh[1]	France, Germany, Italy, Spain, UK	56-57
cherries	whole fruit, flesh[1]	France, Germany, Italy, Spain, UK	58-59
currants, gooseberries	whole fruit	Germany	60
raspberries, blackberries	whole fruit	Germany	61
strawberries[2]	whole fruit	France, Italy, Spain, UK	62
onions	bulbs	France, Germany,Italy, Spain, UK	63
cauliflower	heads	France, UK	64
broccoli	heads	UK	65

Crop	Commodity	Countries	Table
Brussels sprouts	sprouts	Germany, UK	66
cabbage	heads	France, Germany, UK	67
kale	leaves and petioles	UK	68
cucumber[2]	whole fruit	France, Italy, Spain, UK	69-70
summer squash[2]	whole fruit	France, Italy	71-72
melons[2]	whole fruit, pulp	France, Italy, Spain	73-75
tomatoes[2]	whole fruit	France, Italy, Spain, UK	76-77
peppers[2]	whole fruit	France, Italy, Spain, UK	78-79
sweetcorn	kernals	France	80
lettuce[2]	leaves, heads	France, Italy, Spain, UK	81-82
beans, fresh	pods and seeds	France, Germany, Greece, The Netherlands, Spain	83
broad beans	seeds	UK	84
peas, fresh	pods and seeds	Germany, The Netherlands	85
peas, fresh	seeds	France, UK	86
beans	seeds (dry)	France	87
peas	seeds (dry)	France, Spain	88
carrots	roots	France, Italy, Spain	89
sugar beet	roots	France, Italy, Spain, UK	90
potato	tubers	France, Germany, Spain, UK	91
artichoke, globe	heads	France, Italy, Spain	92
asparagus	spears	Germany, Greece	93
barley	grain	France, UK	94
maize	cobs, kernals	France, Germany, Italy	95
wheat	grain	France, UK	96
oil seed rape	seeds and pods	France, Spain, UK	97
sunflower	seeds and heads	France, Italy, Spain	98
sugar beet	tops	France, Italy, Spain, UK	99
barley	forage and straw	France, UK	100
maize	forage and fodder	France, Germany, Italy	101
wheat	straw and fodder	France, UK	102
beans	fodder and forage	France, Spain, UK	103-105
peas	hay and fodder	France, Italy, Spain, UK	106-107

[1]) 'Flesh' means whole fruit without the stone

[2]) Included outdoor and protected crops

Trials were well documented with laboratory and field reports. Laboratory reports included procedural recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables unless residues in control samples exceeded the LOQ. Where residues are reported in samples from control plots, these are recorded as "(c=n.nn)" in the tables. Residue data are recorded unadjusted for recovery.

Results from replicated field plots are presented as individual values while average results are reported for replicate field samples and replicate laboratory samples. When residues were not detected they are shown as below the LOQ (e.g. < 0.01 mg/kg). Residues and application rates have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Where trials have involved two or more applications, the mean or target application rate has been recorded unless the individual rates differ by more than 10%.

In trials involving more than one application, and where samples were taken immediately before the last application, residues from these samples are recorded as being applied at '-0' days.

Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels, STMRs and HRs. These results are <u>double underlined</u>.

Intervals of freezer storage between sampling and analysis were recorded for most trials and were covered by the conditions of the freezer storage stability studies in most cases. Where extended storage periods were reported, these have been noted.

Analytical methods used in the trials measured residues of pirimicarb and also the combined residues of demethyl pirimicarb (R34836) and demethylformamido pirimicarb (R34885), the latter being converted to and measured as R34836. In most trials the methods were also able to measure the carbamate metabolite R238177. In this evaluation, the term 'combined carbamate metabolite residues' refers to the combined residues of these two demethyl metabolites (R34836+R34885, expressed as R34836).

Orange

In trials on oranges in Italy and Spain, 2 foliar applications of pirimicarb (50% WG formulation) were made at 7-9 day intervals to unreplicated plots, using knapsack sprayers and hand lances to obtain full coverage of 1.6-3.5 metre high trees. Mature fruit (2-3 kg or at least 12 units) were sampled and both the whole fruit and the pulp were analysed seperately, using Method RAM 265/03 (HPLC MS-MS) to measure residues of pirimicarb and its carbamate metabolites. The limit of quantification (LOQ) of this method was 0.01 mg/kg for all analytes and the mean recovery rates were 83-91% (whole fruit) and 94-99% (pulp) at fortification levels of 0.01-1.0 mg/kg.

Table 49. Residues in orange (whole fruit) from foliar applications of pirimicarb (50% WG formulation) in supervised trials in Spain and Italy.

ORANGE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Italy, 1999 (Navelina)	0.3	0.038	1	-0	0.12	0.03	< 0.01	PP62/0449 (IT40-99-E395)
			2	0	0.49	0.07	< 0.01	
				3	0.11	0.03	< 0.01	
				7	0.25	0.05	< 0.01	
				14	0.15	0.03	< 0.01	
				21	0.08	0.02	< 0.01	
Spain, 1999 (Valencia Late)	0.38	0.038	1	-0	0.17	0.05	< 0.01	PP62/0440 (ES40-99-S108)
			2	0	0.91	0.11	< 0.01	
				3	0.45	0.08	< 0.01	
				7	0.27	0.05	< 0.01	
				14	0.14	0.02	< 0.01	
				21	0.18	0.03	< 0.01	
Italy, 1999 (Tarocco)	0.38	0.038	1	-0	0.14	0.02	< 0.01	PP62/0449 (IT40-99-E394)
			2	0	0.72	0.06	< 0.01	
				3	0.46	0.05	< 0.01	
				7	0.37	0.03	< 0.01	
				14	0.22	0.02	< 0.01	
				20	0.3	0.02	< 0.01	
Spain, 1999 (Valencia Late)	0.77	0.038	1	-0	0.05	0.02	< 0.01	PP62/0440 (ES40-99-S008)
			2	0	0.38	0.03	< 0.01	
				3	0.12	0.05	< 0.01	
				7	0.1	0.03	< 0.01	
				14	0.11	0.02	< 0.01	
				21	0.08	0.01	< 0.01	
Spain, 1999 (Valencia Late)	0.49	0.05	1	-0	0.27	0.06	< 0.01	PP62/0440 (ES40-99-S108)
			2	0	0.93	0.1	< 0.01	
				3	0.38	0.09	< 0.01	
				7	0.38	0.08	< 0.01	
				14	0.35	0.04	< 0.01	
				21	0.4	0.05	< 0.01	

ORANGE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Spain, 1999 (Valencia Late)	1.0	0.05	1 2	-0 0 3 7 14 21	0.07 0.57 0.22 0.10 0.09 0.11	0.02 0.06 0.07 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0440 (ES40-99-S008)

[1]) combined carbamate metabolite residues (demethyl + demethylformamido pirimicarb, as demethyl pirimicarb)

Table 10. Residues in orange (pulp) from foliar applications of pirimicarb (50% WG formulation) in supervised trials in Spain and Italy.

ORANGE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Italy, 1999 (Navelina)	0.3	0.038	1 2	-0 0 3 7 14 21	< 0.01 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0449 (IT40-99-E395)
Spain, 1999 (Valencia Late)	0.38	0.038	1 2	-0 0 3 7 14 21	< 0.01 0.02 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0440 (ES40-99-S108)
Italy, 1999 (Tarocco)	0.38	0.038	1 2	-0 0 3 7 14 20	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0449 (IT40-99-E394)
Spain, 1999 (Valencia Late)	0.77	0.038	1 2	-0 0 3 7 14 21	< 0.01 0.04 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0440 (ES40-99-S008)
Spain, 1999 (Valencia Late)	0.49	0.05	1 2	-0 0 3 7 14 21	< 0.01 0.03 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 ≤ 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0440 (ES40-99-S108)
Spain, 1999 (Valencia Late)	1.03	0.05	1 2	-0 0 3 7 14 21	< 0.01 0.05 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0440 (ES40-99-S008)

[1]) combined carbamate metabolite residues (demethyl + demethylformamido pirimicarb, as demethyl pirimicarb)

Mandarins

In trials on mandarins in Spain, 2 foliar applications of pirimicarb (50% WG formulation) were made at 7-10 day intervals to unreplicated plots, as broadcast foliar sprays to obtain full coverage of 1-1.6 metre high trees. Mature fruit (1.2-2 kg) were sampled and both the whole fruit and the pulp were analysed seperately, using Method RAM 265/03 (HPLC MS-MS) to measure residues of pirimicarb and its carbamate metabolites. The LOQ of this method was 0.01 mg/kg for all analytes and the mean recovery rates were 91-97% (whole fruit) and 85-91% (pulp) at fortification levels of 0.01-5.0 mg/kg.

Table 51. Residues in mandarins (whole fruit) from foliar applications of pirimicarb (50% WG formulation) in supervised trials in Spain.

MANDARINS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Spain, 1999 (Clemenpons)	0.3	0.038	1 2	-0 0 3 7 14 20	0.83 2.0 1.7 1.6 1.8 1.0	0.09 0.16 0.21 0.21 0.19 0.13	< 0.01 0.01 0.01 < 0.01 < 0.01 < 0.01	PP62/0436 (ES40-99-S309)
Spain, 1999 (Orogrande)	0.5	0.038	1 2	-0 0 3 7 14 20	0.5 1.4 0.78 0.98 1.2 0.67	0.06 0.14 0.13 0.14 0.17 0.09	< 0.01 0.01 < 0.01 < 0.01 0.01 < 0.01	PP62/0436 (ES40-99-S209)
Spain, 1999 (Clausellina)	0.76	0.038	1 2	-0 0 3 7 14 21	0.27 1.0 0.45 0.35 0.28 0.19	0.05 0.12 0.13 0.08 0.05 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0436 (ES40-99-S009)
Spain, 1999 (Okitsu)	0.77	0.038	1 2	-0 0 3 7 14 21	0.43 1.3 0.66 0.72 0.62 0.87	0.06 0.19 0.12 0.1 0.09 0.13	0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0436 (ES40-99-S109)
Spain, 1999 (Clemenpons)	0.4	0.05	1 2	-0 0 3 7 14 20	1.1 3.3 2.7 2.2 2.0 1.4	0.1 0.2 0.28 0.25 0.27 0.16	< 0.01 0.01 0.02 0.01 0.01 < 0.01	PP62/0436 (ES40-99-S309)
Spain, 1999 (Orogrande)	0.67	0.05	1 2	-0 0 3 7 14 20	1.0 2.1 1.3 1.2 0.92 0.84	0.13 0.18 0.19 0.13 0.13 0.17	0.01 0.02 0.01 < 0.01 < 0.01 0.01	PP62/0436 (ES40-99-S209)
Spain, 1999 (Clausellina)	1.0	0.05	1 2	-0 0 3 7 14 21	0.42 0.66 0.67 0.33 0.68 0.54	0.08 0.09 0.12 0.07 0.1 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0436 (ES40-99-S009)
Spain, 1999 (Okitsu)	1.0	0.05	1 2	-0 0 3 7 14 21	0.42 1.2 0.53 0.77 0.7 0.55	0.08 0.15 0.11 0.12 0.11 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0436 (ES40-99-S109)

[1]) combined carbamate metabolite residues (demethyl + demethylformamido pirimicarb, as demethyl pirimicarb)

Table 52. Residues in mandarins (pulp) from foliar applications of pirimicarb (50% WG formulation) in supervised trials in Spain.

MANDARINS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Spain, 1999 (Clemenpons)	0.3	0.038	1 2	-0 0 3 7 14 20	0.03 0.16 0.09 0.04 0.02 0.01	< 0.01 < 0.01 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0436 (ES40-99-S309)
Spain, 1999 (Orogrande)	0.5	0.038	1 2	-0 0 3 7 14 20	< 0.01 0.05 0.04 ≤ 0.01 < 0.01 < 0.01	< 0.01 0.01 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0436 (ES40-99-S209)
Spain, 1999 (Clausellina)	0.76	0.038	1 2	-0 0 3 7 14 21	< 0.01 0.03 0.04 0.03 0.01 < 0.01	< 0.01 < 0.01 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0436 (ES40-99-S009)
Spain, 1999 (Okitsu)	0.77	0.038	1 2	-0 0 3 7 14 21	0.04 0.04 0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0436 (ES40-99-S109)
Spain, 1999 (Clemenpons)	0.4	0.05	1 2	-0 0 3 7 14 20	0.02 0.23 0.11 0.07 0.02 0.04	< 0.01 < 0.01 0.02 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0436 (ES40-99-S309)
Spain, 1999 (Orogrande)	0.67	0.05	1 2	-0 0 3 7 14 20	< 0.01 0.04 0.05 0.01 < 0.01 >0.01	< 0.01 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0436 (ES40-99-S209)
Spain, 1999 (Clausellina)	1.0	0.05	1 2	-0 0 3 7 14 21	0.01 0.04 0.03 < 0.01 0.01 < 0.01	< 0.01 0.01 0.01 < 0.01 ≤ 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0436 (ES40-99-S009)
Spain, 1999 (Okitsu)	1.0	0.05	1 2	-0 0 3 7 14 21	0.04 0.03 0.02 0.02 0.01 < 0.01	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0436 (ES40-99-S109)

[1]) combined carbamate metabolite residues (demethyl + demethylformamido pirimicarb, as demethyl pirimicarb)

Apples

In trials on apples in France, Germany, Italy, Spain and the UK, 2 foliar applications of pirimicarb (50% WG formulation) were made at 7−10 day intervals (3 applications at 17−21 day intervals in Germany) to unreplicated plots, using airblast, knapsack mist blowers or knapsack sprayers and hand lances to obtain full coverage of 2−4 metre high trees. Mature fruit (at least 1 kg or 12 units) were sampled and analysed using either Method RAM 15/02 with GC-MSD detection (Germany), RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its

carbamate metabolites. The LOQ of these methods was 0.01 mg/kg for all analytes and the mean recovery rates were 81-88% at fortification levels of 0.01−1.0 mg/kg.

Table 53. Residues in apple from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France, Germany, Italy, Spain and the UK.

APPLE Country, year (variety)	Application kg ai/ha	kg ai/hL	no	PHI, (days)	Residues (mg/kg) Pirimicarb	R34836 +R34885[1]	R238177	Reference & Comments
France (N), 2000 (Akane)	0.13	0.025	1 2	-0 0 3 8 14 21	0.01 0.18 0.04 0.02 0.02 0.02	< 0.01 0.01 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0930 (FR31-00-S764)
UK, 2001 (Bramley)	0.31	0.025	1 2	-0 0 3 7 14 21	0.01 0.12 0.07 0.04 0.03 0.05	< 0.01 < 0.01 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1187 (AF/5960/SY/3)
France (N), 2000 (Golden)	0.33	0.025	1 2	-0 0 3 8 14 21	0.12 0.35 0.14 0.15 0.15 0.14	0.01 0.02 0.01 0.02 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0930 (FR72-00-S750)
UK, 2001 (Elstar)	0.33	0.025	1 2	-0 0 3 7 14 21	0.13 0.34 0.28 0.28 0.27 0.16	< 0.01 0.02 0.02 0.02 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1187 (AF/5960/SY/5)
UK, 2001 (Golden Delicious)	0.33	0.025	1 2	-0 0 3 7 14 21	0.22 0.6 0.35 0.88 0.41 0.22	< 0.01 0.02 0.02 0.03 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1187 (AF/5960/SY/4)
UK, 2000 (Gala)	0.37	0.025	1 2	-0 0 3 8 14 21	0.09 0.14 0.21 0.13 0.1 0.14	< 0.01 0.02 0.04 0.02 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0930 (GB07-00-S080)
UK, 2000 (Cox)	0.37	0.025	1 2	-0 0 3 8 14 21	0.31 0.75 0.31 0.25 0.3 0.23	0.02 0.04 0.04 0.02 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0930 (GB07-00-S081)
France (N), 2001 (Golden Delicious)	0.28+ 0.36	0.025+ 0.025	1+ 1	-0 0 3 7 14 21	0.11 0.23 0.14 0.18 0.11 0.13	< 0.01 0.01 0.02 0.02 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1187 (AF/5960/SY/1)
France (N), 2001 (Gala)	0.36+ 0.31	0.025+ 0.025	1+ 1	-0 0 3 7 14 21	0.07 0.13 0.07 0.16 0.05 0.06	< 0.01 0.01 < 0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1187 (AF/5960/SY/2)

APPLE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836 +R34885[1]	R238177	
France (N), 2000 (Akane)	0.19	0.038	1 2	-0 0 3 8 14 21	0.02 0.27 0.08 0.04 0.04 0.04	< 0.01 0.01 0.02 0.02 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0930 (FR31-00-S764)
Italy, 1999 (Granny Smith)	0.28	0.038	1 2	-0 0 3 7 14 21	0.02 0.14 0.02 0.03 0.03 0.03	< 0.01 0.02 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0952 (AF/4745/ZE/4)
UK, 2001 (Bramley)	0.45	0.038	1 2	-0 0 3 7 14 21	0.11 0.25 0.23 0.08 0.1 0.07	< 0.01 < 0.01 0.02 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1187 (AF/5960/SY/3)
France (S), 1999 (Gala)	0.45	0.038	1 2	-0 0 3 7 14 21	0.09 0.33 0.22 0.13 0.13 0.1	0.01 0.02 0.03 0.02 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0952 (AF/4745/ZE/1)
France (N), 2000 (Golden)	0.49	0.038	1 2	-0 0 3 8 14 21	0.15 0.7 0.29 0.2 0.25 0.28	0.02 0.03 0.04 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0930 (FR72-00-S750)
UK, 2001 (Elstar)	0.5	0.038	1 2	-0 0 3 7 14 21	0.08 0.25 0.27 0.5 0.34 0.31	< 0.01 0.02 0.02 0.03 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1187 (AF/5960/SY/5)
UK, 2000 (Gala)	0.56	0.038	1 2	-0 0 3 8 14 21	0.13 0.62 0.2 0.41 0.14 0.22	0.01 0.05 0.03 0.04 0.02 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0930 (GB07-00-S080)
UK, 2000 (Cox)	0.56	0.038	1 2	-0 0 3 8 14 21	0.33 1.05 0.48 0.53 0.43 0.38	0.02 0.05 0.06 0.05 0.04 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0930 (GB07-00-S081)
UK, 2001 (Golden Delicious)	0.46+ 0.51	0.038+ 0.038	1+ 1	-0 0 3 7 14 21	0.28 0.96 0.83 0.24 0.78 0.66	0.01 0.03 0.04 0.01 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1187 (AF/5960/SY/4)
France (N), 2001 (Golden Delicious)	0.46+ 0.56	0.038+ 0.038	1+ 1	-0 0 3 7 14 21	0.18 0.28 0.25 0.18 0.12 0.14	0.01 0.01 0.02 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1187 (AF/5960/SY/1)

APPLE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836 +R34885[1]	R238177	
France (N), 2001 (Gala)	0.56+ 0.48	0.038+ 0.038	1+ 1	-0 0 3 7 14 21	0.2 0.27 0.19 0.18 0.1 0.14	0.01 0.02 0.02 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1187 (AF/5960/SY/2)
France (S), 1999 (Braeburn)	0.59+ 0.63	0.038+ 0.038	1+ 1	-0 0 3 10 14 21	0.06 0.21 0.21 0.21 0.17 0.13	0.01 0.03 0.03 0.03 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0952 (AF/4745/ZE/2)
Italy, 1999 (Fuji)	0.72+ 0.45	0.038+ 0.038	1+ 1	-0 0 3 8 14 21	0.09 0.38 0.13 0.12 0.15 0.1	0.03 0.07 0.05 0.05 0.04 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0952 (AF/4745/ZE/3)
France (N), 2003 (Golden)	0.46	0.04	2	7	0.07	0.01	< 0.01	PP62/1390 (AF/7359/SY/1) Processing study
Italy, 2000 (Golden Delicious)	0.5	0.05	2	7 14	0.15 0.11	0.06 0.03	< 0.01 < 0.01	PP62/0982 (IT30-00-S390)
Spain, 2000 (Royal Gala)	0.6	0.05	2	7 14	0.25 (c=0.02) 0.18	0.05 0.04	< 0.01 < 0.01	PP62/0982 (ES30-00-S121)
Italy, 2000 (Red Chief)	0.6	0.05	2	7 14	0.03 0.05	0.02 0.02	< 0.01 < 0.01	PP62/0982 (IT20-00-S391) Processing study
Spain, 2000 (Golden)	0.59+ 0.56	0.05+ 0.05	1+ 1	7 14	0.12 0.09	0.06 0.03	< 0.01 < 0.01	PP62/0982 (ES60-00-S021)
Germany, 1991 (Cox Orange)	0.38	0.025	3	0 7 16 21 28	0.79, 0.66 0.53, 0.45 0.39, 0.39 0.4, 0.25 0.31, 0.14	0.03, 0.03 0.07, 0.04 0.04, 0.04 0.03, 0.02 0.03, 0.02		PP62/0406 (91JH071F-G1)
Germany, 1991 (Idared)	0.38	0.13	3	0 7 14 20 28	0.14, 0.18 0.03, 0.05 0.04, 0.04 0.02, 0.02 0.02, 0.02	0.02, 0.04 0.01, 0.02 0.01, 0.02 < 0.01 (2) < 0.01 (2)		PP620406 (91JH071F-E1)

[1]) combined carbamate metabolite residues (demethyl + demethylformamido pirimicarb, as demethyl pirimicarb)

Peach and nectarine

In trials on peaches and nectarines in France, Italy and Spain, 2 foliar applications of pirimicarb (50% WG formulation) were made at 7–10 day intervals to unreplicated plots, using knapsack mist blowers or knapsack or motorised hand lance to obtain full coverage of 2.5–3.5 metre high trees. Mature fruit (2-3 kg or at least 24 units) were sampled and analysed using either Method RAM 015/01 with GC-NPD detection, RAM 265/02 (with GC-MSD and NPD detection) or RAM 265/03 (with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ of these methods was 0.01 mg/kg for all analytes and the mean recovery rates were 83–98% at fortification levels of 0.01-1.0 mg/kg. Residues were measured after removal of the stones, and both the pulp and calculated whole fruit results have been reported.

Table 54. Residues in peaches and nectarines (whole fruit) from foliar applications of pirimicarb (50% or 17.5% WG formulations) in supervised trials in France, Italy and Spain.

PEACH & NECTARINE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Italy, 1993		0.035	2	7	0.36	0.03	< 0.01	PP62/0419
(Claudia)				14	0.24	0.03	< 0.01	(IT10-93-E336)
				21	0.17	0.02	< 0.01	
Nectarine								175 gai/kg WG
Italy, 1993		0.035	2	7	0.34	0.02	< 0.01	PP62/0419
(Merryl Gem)				14	0.19	0.01	< 0.01	(IT10-93-E335)
				21	0.15	0.01	< 0.01	
Peach				28	0.09	0.01	< 0.01	175 gai/kg WG
Italy, 1993		0.035	2	7	0.08	0.02	< 0.01	PP62/0419
(Red Haven)				14	0.09	0.02	< 0.01	(IT10-93-E334)
				21	0.04	< 0.01	< 0.01	
Peach				28	0.05	< 0.01	< 0.01	175 gai/kg WG
Italy, 1993		0.035	2	7	0.22	0.03	< 0.01	PP62/0419
(Venus)				14	0.14	0.02	< 0.01	(IT10-93-E337)
				21	0.07	0.01	< 0.01	
Nectarine				28	0.04	< 0.01	< 0.01	175 gai/kg WG
Italy, 1994		0.035	2	7	0.22	0.02	< 0.01	PP62/0417
(Merryl Gem)				14	0.1	0.01	< 0.01	(IT10-94-E330)
				21	0.07	0.01	< 0.01	
Peach				28	0.07	0.02	< 0.01	175 gai/kg WG
Italy, 1994		0.035	2	7	0.14	0.02	< 0.01	PP62/0417
(Red Haven)				14	0.15	0.02	< 0.01	(IT10-94-E333)
				21	0.1	0.02	< 0.01	
Peach				28	0.08	0.02	< 0.01	175 gai/kg WG
Italy, 1994		0.035	2	7	0.17	0.02	< 0.01	PP62/0417
(Stark Red Gold)				14	0.14	0.02	< 0.01	(IT10-94-E331)
				21	0.11	0.02	< 0.01	
Nectarine				28	0.07	0.01	< 0.01	175 gai/kg WG
Italy, 1994		0.035	2	7	0.09	0.02	< 0.01	PP62/0417
(Sweet Lady)				14	0.04	0.01	< 0.01	(IT10-94-E332)
				21	0.03	< 0.01	< 0.01	
Nectarine				28	0.02	< 0.01	< 0.01	175 gai/kg WG
Spain, 1999	0.45	0.038	1	-0	0.79	0.04	< 0.01	PP62/0468
(Miraflores)			2	0	1.6	0.05	< 0.01	(ES30-99-S228)
				3	1.1	0.05	< 0.01	
Peach				7	1.2	0.07	< 0.01	
				14	0.87	0.05	< 0.01	
				21	0.8	0.06	< 0.01	
Spain, 1999	0.66	0.038	1	-0	0.19	0.03	< 0.01	PP62/0468
(Merly O'Henry)			2	0	0.99	0.03	< 0.01	(ES60-99-S128)
				3	0.55	0.05	< 0.01	
Peach				7	0.32	0.03	< 0.01	
				14	0.28	0.03	< 0.01	
				21	0.28	0.04	< 0.01	
France (S), 1999	0.32+	0.038+	1+	-0	0.11	0.01	< 0.01	PP62/0468
(Tendresse)	0.38	0.038	1	0	0.66	0.02	< 0.01	(AF4757/ZE/1)
				3	0.54	0.03	< 0.01	
Peach				7	0.25	0.02	< 0.01	
				14	0.24	0.02	< 0.01	
				21	0.21	0.02	< 0.01	
Italy, 1999	0.45+	0.038+	1+	-0	0.19	0.02	< 0.01	PP62/0468
(Maria Bianca)	0.51	0.038	1	0	0.64	0.03	< 0.01	(AF4757/ZE/2)
				3	0.49	0.04	< 0.01	
Peach				7	0.34	0.03	< 0.01	
				14	0.39	0.04	< 0.01	
				21	0.21	0.03	< 0.01	

Residues are calculated on a whole fruit basis (including stones).

[1]) combined carbamate metabolite residues (demethyl + demethylformamido pirimicarb, as demethyl pirimicarb)

Table 55. Residues in peaches and nectarines (flesh) from foliar applications of pirimicarb (50% or 17.5% WG formulations) in supervised trials in Italy, France, and Spain.

PEACH & NECTARINE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Italy, 1993 (Red Haven) Peach		0.035	2	7 14 21 28	0.1 0.11 0.04 0.05	0.02 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/0419 (IT10-93-E334) 175 g ai/kg WG
Italy, 1993 (Merryl Gem) Peach		0.035	2	7 14 21 28	0.37 0.2 0.16 0.1	0.02 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/0419 (IT10-93-E335) 175 g ai/kg WG
Italy, 1993 (Claudia) Nectarine		0.035	2	7 14 21	0.38 0.26 0.18	0.03 0.03 0.02	< 0.01 < 0.01 < 0.01	PP62/0419 (IT10-93-E336) 175 g ai/kg WG
Italy, 1993 (Venus) Nectarine		0.035	2	7 14 21 28	0.24 0.15 0.08 0.04	0.03 0.02 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/0419 (IT10-93-E337) 175 g ai/kg WG
France (S), 1999 (Tendresse) Peach	0.32+ 0.38	0.038+ 0.038	1+ 1	-0 0 3 7 14 21	0.13 0.79 0.61 0.27 0.25 0.22	0.01 0.02 0.03 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0468 (AF4757/ZE/1)
Spain, 1999 (Merly O'Henry) Peach	0.66	0.038	1 2	-0 0 3 7 14 21	0.21 1.04 0.58 0.34 0.3 0.29	0.03 0.03 0.05 0.03 0.03 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0468 (ES60-99-S128)
Spain, 1999 (Miraflores) Peach	0.45	0.038	1 2	-0 0 3 7 14 21	0.87 1.78 1.25 1.28 0.95 0.86	0.05 0.06 0.06 0.08 0.06 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0468 (ES30-99-S228)
Italy, 1999 (Maria Bianca) Peach	0.45+ 0.51	0.038+ 0.038	1+ 1	-0 0 3 7 14 21	0.21 0.7 0.52 0.37 0.42 0.21	0.02 0.03 0.04 0.03 0.04 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0468 (AF4757/ZE/2)

[1]) combined carbamate metabolite residues (demethyl + demethylformamido pirimicarb, as demethyl pirimicarb)

Plums

In trials on plums in France, Germany, Italy, Spain and the UK, 2 foliar applications of pirimicarb (50% WG formulation) were made at 7–10 day intervals to unreplicated plots, using knapsack mist blowers to obtain full coverage of 2.5–5 metre high trees. Mature fruit (2–3 kg or at least 24 units) were sampled and analysed using either Method RAM 265/03 or RAM 265/04 (both with HPLC-MS-

MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ of these methods was 0.01 mg/kg for all analytes and the mean recovery rates were 84–96% at fortification levels of 0.01–1.0 mg/kg. Residues were measured after removal of the stones, and both the pulp and calculated whole fruit results have been reported.

Table 56. Residues in plums (whole fruit) from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France, Germany, Italy, Spain and the UK.

PLUMS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Germany, 2000 (Calacks Fruchtbare)	0.37	0.02	1 2	-0 0 3 7 14 21	0.1 0.23 0.13 0.08 0.15 0.11	< 0.01 0.02 0.01 < 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01	PP62/0953 (38/275/3)
UK, 2000 (Victoria)	0.37	0.025	1 2	-0 0 3 7 14 21	0.1 0.22 0.16 0.03 0.1 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0953 (38/275/2)
Germany, 2000 (President)	0.37	0.025	1 2	-0 0 3 7 14 21 28	0.15 0.22 0.16 0.22 0.13 0.13 0.27	< 0.01 0.02 0.01 0.02 0.01 < 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01 0.02	PP62/0953 (38/275/4)
UK, 2000 (Victoria)	0.38	0.025	1 2	-0 0 3 7 14 21	0.09 0.15 0.12 0.08 0.07 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0953 (38/275/1)
France (S), 1999 (President)	0.54	0.038	1 2	-0 0 3 7 14 21	0.11 0.26 0.27 0.3 0.2 0.12	0.01 0.02 0.04 0.03 0.02 0.01	< 0.01 < 0.01 0.01 0.02 0.03 0.02	PP62/0447 (AF/4746/ZE/1)
UK, 2000 (Victoria)	0.56	0.038	1 2	-0 0 3 7 14 21	0.16 0.38 0.31 0.21 0.2 0.17	< 0.01 0.01 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 0.01 0.01 < 0.01	PP62/0953 (38/275/2)
Germany, 2000 (Calacks Fruchtbare)	0.56	0.038	1 2	-0 0 3 7 14 21	0.17 0.3 0.22 0.23 0.27 0.28	0.01 0.02 0.02 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 0.02 0.02	PP62/0953 (38/275/3)
Germany, 2000 (President)	0.56	0.038	1 2	-0 0 3 7 14 21 28	0.15 0.38 0.46 0.24 0.13 0.21 0.1	0.01 0.02 0.03 0.02 < 0.01 0.02 < 0.01	< 0.01 < 0.01 < 0.01 0.01 0.01 0.02 0.02	PP62/0953 (38/275/4)

PLUMS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Italy, 1999 (Fria)	0.58	0.038	1 2	-0 0 3 7 14 21	0.05 0.13 0.13 0.1 0.09 0.06	< 0.01 0.01 0.01 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0447 (AF/4746/ZE/2)
UK, 2000 (Victoria)	0.58	0.038	1 2	-0 0 3 7 14 21	0.09 0.25 0.25 0.14 0.12 0.1	< 0.01 0.01 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0953 (38/275/1)
UK, 1999 (Victoria)	0.65	0.038	2	0 14 21	0.35 0.24 0.23	0.03 0.02 0.02	< 0.01 0.01 0.01	PP62/0442 (38/242/1)
Germany, 1999 (Ortenauer)	0.67	0.038	2	0 14 21	0.57 0.32 0.24	0.03 0.03 0.02	< 0.01 0.02 0.02	PP62/0442 (38/242/4)
UK, 1999 (Victoria)	0.68	0.038	2	0 14 21	0.44 0.21 0.2	0.03 0.01 < 0.01	< 0.01 0.01 0.01	PP62/0442 (38/242/2)
Germany, 1999 (Averbacher)	0.63+ 0.7	0.038+ 0.038	1+ 1	0 14 21	0.65 0.14 0.34	0.1 0.04 0.06	0.01 < 0.01 0.01	PP62/0442 (38/242/3)
Spain, 2000 (Friar)	0.6	0.05	2	7	0.15	0.07	< 0.01	PP62/0934 (ES30-00-S120)
Spain, 2000 (Friar)	0.66	0.05	2	7 14	0.17 0.13	0.02 0.02	< 0.01 < 0.01	PP62/0934 (ES60-00-S020)
France (S) 2003 (Denthes)	0.68	0.05	2	6	0.29	0.02	0.05	PP62/1389 (AF/7362/SY/1) Processing study

Residues are calculated on a whole fruit basis (including stones).

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Table 57. Residues in plums (flesh) from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France, Germany, Italy, Spain and the UK.

PLUMS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
UK, 2000 (Victoria)	0.37	0.025	1 2	-0 0 3 7 14 21	0.11 0.24 0.17 0.03 0.11 0.08	< 0.01 < 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0953 (38/275/2)
Germany, 2000 (Calacks Fruchtbare)	0.37	0.025	1 2	-0 0 3 7 14 21	0.11 0.25 0.14 0.08 0.16 0.12	< 0.01 0.02 0.01 < 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01	PP62/0953 (38/275/3)

PLUMS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Germany, 2000 (President)	0.37	0.025	1 2	-0 0 3 7 14 21 28	0.17 0.24 0.17 0.24 0.14 0.14 0.28	< 0.01 0.02 0.01 0.02 0.01 < 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 0.01 0.01 0.02	PP62/0953 (38/275/4)
UK, 2000 (Victoria)	0.38	0.025	1 2	-0 0 3 7 14 21	0.1 0.17 0.13 0.09 0.07 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0953 (38/275/1)
France (S), 1999 (President)	0.54	0.038	1 2	-0 0 3 7 14 21	0.14 0.29 0.3 0.33 0.22 0.13	0.01 0.02 0.04 0.03 0.02 0.01	< 0.01 < 0.01 0.01 0.02 0.03 0.02	PP62/0447 (AF/4746/ZE/1)
UK, 2000 (Victoria)	0.56	0.038	1 2	-0 0 3 7 14 21	0.17 0.42 0.33 0.23 0.21 0.18	< 0.01 0.01 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 0.01 0.01 < 0.01	PP62/0953 (38/275/2)
Germany, 2000 (Calacks Fruchtbare)	0.56	0.038	1 2	-0 0 3 7 14 21	0.19 0.33 0.24 0.25 0.29 0.29	0.01 0.02 0.02 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 0.02 0.02	PP62/0953 (38/275/3)
Germany, 2000 (President)	0.56	0.038	1 2	-0 0 3 7 14 21 28	0.16 0.42 0.5 0.26 0.14 0.22 0.1	0.01 0.02 0.03 0.02 < 0.01 0.02 < 0.01	< 0.01 < 0.01 < 0.01 0.01 0.01 0.02 0.02	PP62/0953 (38/275/4)
Italy, 1999 (Fria)	0.58	0.038	1 2	-0 0 3 7 14 21	0.05 0.13 0.14 0.1 0.09 0.06	< 0.01 0.01 0.01 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0447 (AF/4746/ZE/2)
UK, 2000 (Victoria)	0.58	0.038	1 2	-0 0 3 7 14 21	0.1 0.28 0.27 0.15 0.13 0.1	< 0.01 0.01 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0953 (38/275/1)
UK, 1999 (Victoria)	0.65	0.038	2	0 14 21	0.4 0.26 0.24	0.03 0.02 0.02	< 0.01 0.01 0.01	PP62/0442 (38/242/1)
Germany, 1999 (Ortenauer)	0.67	0.038	2	0 14 21	0.63 0.34 0.25	0.03 0.03 0.02	< 0.01 0.02 0.02	PP62/0442 (38/242/4)
UK, 1999 (Victoria)	0.68	0.038	2	0 14 21	0.5 0.27 0.21	0.03 0.01 < 0.01	< 0.01 0.01 0.01	PP62/0442 (38/242/2)
Germany, 1999 (Averbacher)	0.63+ 0.7	0.038+ 0.038	1+ 1	0 14 21	0.72 0.15 0.37	0.11 0.04 0.06	0.01 < 0.01 0.01	PP62/0442 (38/242/3)

PLUMS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Spain, 2000 (Friar)	0.6	0.05	2	7	0.15	0.07	< 0.01	PP62/0934 (ES30-00-S120)
Spain, 2000 (Friar)	0.66	0.05	2	7 14	0.17 0.13	0.02 0.02	< 0.01 < 0.01	PP62/0934 (ES60-00-S020)

[1]) combined carbamate metabolite residues (demethyl + demethylformamido pirimicarb, as demethyl pirimicarb)

Cherries

In trials on cherries in France, Germany, Italy, Spain and the UK, 2 foliar applications of pirimicarb (50% WG formulation) were made at 7–10 day intervals to unreplicated plots, using knapsack mist blowers or motorised hand-guns to obtain full coverage of 3–4.5 metre high trees. Mature fruit were sampled and analysed using Method RAM 265/04 (with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The limit of quantification of these methods was 0.01 mg/kg for all analytes and the mean recovery rates were 79–97% at fortification levels of 0.01–5.0 mg/kg. Residues were measured after removal of the stones and both the pulp residues and the calculated whole fruit results have been reported.

Table 58. Residues in cherries (whole fruit) from foliar applications of pirimicarb (50% WG formulations) in supervised trials in France, Germany, Italy, Spain and the UK.

CHERRIES Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
France (S), 1999 (Stuart Hardy Giant)	0.38+ 0.42	0.038+ 0.038	1+ 1	-0 0 3 7 14 21	1.4 2.0 1.5 0.59 1.2 1.1	0.04 0.04 0.05 0.02 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0445 (AF/4747/ZE/1)
Italy, 1999 (Ferrovia)	0.51+ 0.59	0.038+ 0.038	1+ 1	-0 0 4 7 14 21	0.85 1.5 2.0 0.78 1.1 0.76	0.05 0.06 0.09 0.03 0.04 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0445 (AF/4747/ZE/2)
France (S), 1999 (Stuart Hardy Giant)	0.53	0.05	1 2	-0 0 3 7 14 21	1.9 3.8 2.7 1.9 1.7 1.6	0.05 0.06 0.06 0.07 0.05 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0445 (AF/4747/ZE/1)
Spain, 2000 (Lapins)	0.7	0.05	2	7 14	1.1 (c=0.19) 0.41 (c=0.17)	0.05 0.02	< 0.01 < 0.01	PP62/0963 (ES30-00-S219)
Italy, 1999 (Ferrovia)	0.72	0.05	1 2	-0 0 4 7 14 21	1.0 1.3 2.1 1.4 1.1 0.81	0.04 0.05 0.08 0.06 0.04 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0445 (AF/4747/ZE/2)
Spain, 2000 (Canada Giant)	0.76	0.05	2	7 14	0.28 0.08	0.06 0.03	< 0.01 0.02	PP62/0963 (ES60-00-S019)
Germany, 1999 (Regina)	0.5+ 0.47	0.038+ 0.038	1+ 1	0 7 14	1.5 0.69 0.47	0.06 0.04 0.03	0.03 0.05 0.05	PP62/0438 (38/241/2)
UK, 1999 (Suckley)	0.51+ 0.48	0.038+ 0.038	1 +1	0 7 14	2.2 0.89 0.48	0.13 0.08 0.05	< 0.01 < 0.01 < 0.01	PP62/0438 (38/241/1)

CHERRIES Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
France (N), 2001 (Duromi 3)	0.47	0.038	1 2	-0 0 3 7 14 21	0.36 0.86 0.45 0.43 0.42 0.33	0.02 0.02 0.02 0.02 0.01 0.01	0.01 0.01 < 0.01 0.01 < 0.01 < 0.01	PP62/1177 (AF/5957/SY/1)
France (N), 2001 (Badacsony)	0.49+ 0.46	0.038+ 0.038	1+ 1	-0 0 3 7 14 21	0.21 2.1 1.2 0.71 0.42 0.28	0.02 0.05 0.04 0.02 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1177 (AF/5957/SY/2)

Residues are calculated on a whole fruit basis (including stones).

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Table 59. Residues in cherries (flesh) from foliar applications of pirimicarb (50% WG formulations) in supervised trials in France, Germany, Italy, Spain and the UK.

CHERRIES Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (S), 1999 (Stuart Hardy Giant)	0.38+ 0.42	0.038+ 0.038	1+ 1	-0 0 3 7 14 21	1.6 2.2 1.7 0.63 1.3 1.2	0.05 0.04 0.05 0.02 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0445 (AF/4747/ZE/1)
Italy, 1999 (Ferrovia)	0.51+ 0.59	0.038+ 0.038	1+ 1	-0 0 4 7 14 21	1.0 1.9 2.2 0.91 1.2 0.83	0.06 0.08 0.11 0.04 0.05 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0445 (AF/4747/ZE/2)
France (S), 1999 (Stuart Hardy Giant)	0.53	0.05	1 2	-0 0 3 7 14 21	2.1 4.0 2.9 2.0 1.8 1.7	0.06 0.07 0.07 0.07 0.05 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0445 (AF/4747/ZE/1)
Spain, 2000 (Lapins)	0.7	0.05	2	7 14	1.2 (c=0.21) 0.45 (c=0.18)	0.05 0.02	< 0.01 < 0.01	PP62/0963 (ES30-00-S219)
Italy, 1999 (Ferrovia)	0.72	0.05	1 2	-0 0 4 7 14 21	1.2 1.6 2.6 1.7 1.3 0.87	0.05 0.06 0.1 0.07 0.05 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0445 (AF/4747/ZE/2)
Spain, 2000 (Canada Giant)	0.76	0.05	2	7 14	0.3 0.09	0.06 0.03	< 0.01 0.02	PP62/0963 (ES60-00-S019)
Germany, 1999 (Regina)	0.5+ 0.47	0.038+ 0.038	1+ 1	0 7 14	1.6 0.74 0.52	0.06 0.04 0.03	0.03 0.05 0.06	PP62/0438 (38/241/2)
UK, 1999 (Suckley)	0.51+ 0.48	0.038+ 0.038	1 +1	0 7 14	2.6 0.89 0.48	0.16 0.09 0.06	< 0.01 < 0.01 < 0.01	PP62/0438 (38/241/1)
France (N), 2001 (Duromi 3)	0.47	0.038	1 2	-0 0 3 7 14 21	0.46 0.94 0.5 0.47 0.45 0.36	0.02 0.02 0.02 0.02 0.01 0.01	0.01 0.01 < 0.01 0.01 < 0.01 < 0.01	PP62/1177 (AF/5957/SY/1)

CHERRIES Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (N), 2001 (Badacsony)	0.49+ 0.46	0.038+ 0.038	1+ 1	-0 0 3 7 14 21	0.28 2.7 1.3 0.8 0.45 0.3	0.02 0.06 0.05 0.02 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1177 (AF/5957/SY/2)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Currants and Gooseberries

In trials on currants (red and black) and on gooseberries in Germany, 2 foliar applications of pirimicarb (50% WG formulation) were made at 12–16 day intervals to unreplicated 35–135 square metre plots, using knapsack sprayers and hand lances to obtain full foliar coverage. Mature fruit (0.8–1.0 kg) were sampled and analysed using Method RAM 265/02, with GC-NPD detection, to measure residues of pirimicarb and its carbamate metabolites. The LOQ of the method was 0.01 mg/kg for all analytes and the mean recovery rates were 79–103% at fortification levels of 0.01–1.6 mg/kg.

Table 60. Residues in currants and gooseberries from foliar applications of pirimicarb (50% WG formulation) in supervised trials in Germany.

CURRANTS & GOOSEBERRIES Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Germany, 1998 (Rovata) Red currant	0.25	0.025	2	0 2 6 10 14	0.19 0.09 0.07 0.06 0.05	0.02 0.02 0.01 < 0.01 < 0.01	0.04 0.04 0.01 < 0.01 < 0.01	PP62/0425 (RS-9828- E1)
Germany, 1998 (Rondom) Red currant	0.25	0.025	2	0 4 8 11 15	1.5 0.24 0.14 0.08 0.08	0.02 0.01 ≤ 0.01 < 0.01 < 0.01	0.06 0.15 0.08 0.02 < 0.01	PP62/0425 (RS-9828- K1)
Germany, 1998 (Ojebin) Black currant	0.25	0.025	2	0 2 7 9 13	0.88 0.34 0.23 0.18 0.16	0.03 0.03 0.02 0.02 0.01	0.03 0.04 0.04 0.04 0.04	PP62/0425 (RS-9828- K2)
Germany, 1998 (Ben Lomond) Black currant	0.25	0.025	2	0 4 7 11 14	1.3 0.25 0.18 0.12 0.09	0.03 < 0.01 ≤ 0.01 < 0.01 < 0.01	0.07 0.08 0.07 0.06 0.06	PP62/0425 (RS-9828- R1)
Germany, 1997 (Titania) Black currant	0.25	0.025	2	0 3 7 10 14	0.89 0.3 0.06 0.07 0.08	0.01 0.02 < 0.01 < 0.01 ≤ 0.01	< 0.01 0.02 0.01 0.02 0.03	PP62/0420 (RS-9825- B1)
Germany, 1997 (Black Dawn) Black currant	0.25	0.025	2	0 3 8 11 15	0.66 0.38 0.16 0.28 0.24	0.01 0.02 0.02 0.02 0.02	0.02 0.05 0.06 0.1 0.13	PP62/0420 (RS-9825- H1)
Germany, 1997 (Rondom) Red currant	0.25	0.025	2	0 3 7 11 14	0.64 0.16 0.09 0.02 0.02	0.03 0.02 0.02 < 0.01 < 0.01	0.07 0.04 0.01 < 0.1 < 0.01	PP62/0420 (RS-9825- K1)

CURRANTS & GOOSEBERRIES Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Germany, 1997 (Achilles)	0.25	0.025	2	0	0.36	0.03	0.01	PP62/0420 (RS-9825-H2)
				4	0.16	0.03	0.01	
				8	0.13	0.03	0.01	
Gooseberry				10	0.09	0.02	0.01	
				15	0.08	0.02	< 0.01	

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Raspberries and Blackberries

In trials on raspberries and blackberries in Germany, 2 foliar applications of pirimicarb (50% WG formulation) were made at 15–17 day intervals to unreplicated 40-54 square metre plots, using knapsack sprayers and hand lances to obtain full foliar coverage. Mature fruit (0.5–0.6 kg) were sampled and analysed using Method RAM 265/02 (with GC-NPD detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ of the method was 0.01 mg/kg for all analytes and the mean recovery rates were 80–110% at fortification levels of 0.01-0.1 mg/kg.

Table 61. Residues in raspberries and blackberries from foliar applications of pirimicarb (50% WG formulation) in supervised trials in Germany.

RASPBERRY & BLACKBERRY Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Germany, 1997 (Meeker)	0.25	0.025	2	0	1.4	< 0.01	< 0.01	PP62/0421 (RS-9724-B1)
				3	0.36	0.03	< 0.01	
				7	0.23	0.01	< 0.01	
Raspberry				10	0.17	< 0.01	< 0.01	
				14	0.07	< 0.01	< 0.01	
Germany, 1997 (Schonemann)	0.25	0.025	2	0	1.5	0.04	0.01	PP62/0421 (RS-9724-B2)
				3	0.7	0.06	< 0.01	
				7	0.76	0.06	< 0.01	
Raspberry				10	0.34	0.02	< 0.01	
				13	0.15	< 0.01	< 0.01	
Germany, 1997 (Schonemann)	0.25	0.025	2	0	0.78	0.06	< 0.01	PP62/0421 (RS-9724-H1)
				2	0.47	0.04	< 0.01	
				7	0.34	0.02	< 0.01	
Raspberry				10	0.29	0.02	< 0.01	
				14	0.22	0.01	< 0.01	
Germany, 1998 (Meeker)	0.25	0.025	2	0	0.53	0.02	< 0.01	PP62/0424 (RS-9827-E1)
				2	(c=0.06)	0.02	< 0.01	
				7	0.37	0.02	< 0.01	
Raspberry				10	0.43	0.01	< 0.01	
				13	0.28	0.01	< 0.01	
					(c=0.03)			
					0.21			
Germany, 1997 (Nessie)	0.25	0.025	2	0	2.1	0.1	0.01	PP62/0421 (RS-9724-H2)
				2	2.1	0.08	< 0.01	
				7	1.2	0.03	< 0.01	
Blackberry				10	1.2	0.03	< 0.01	
				14	0.98	0.02	< 0.01	"Fruit not fit for harvest"

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Strawberries

In trials on field strawberries in Italy and Spain and in protected strawberries in France, Italy, Spain and the UK, 2 foliar applications of pirimicarb (50% WG formulation) were made at 7 day intervals (field crops) and 7-12 day intervals (protected crops) to unreplicated 25-100 square metre plots, using

plot sprayes or knapsack sprayers with either hand lances or mini-booms, to obtain full foliar coverage. Mature fruit (1-2 kg) were sampled and analysed using either Method RAM 265/03 (with GC-NPD detection) or RAM 265/04, with HPLC-MS-MS detection, to measure residues of pirimicarb and its carbamate metabolites. The LOQ of these methods was 0.01 mg/kg for all analytes and the mean recovery rates were 80-94% at fortification levels of 0.01-0.5 mg/kg.

Table 62. Residues in strawberries from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France, Italy, Spain and the UK.

STRAWBERRY Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
OUTDOOR CROPS								
Italy, 1997 (Cortina)	0.45	0.05		0	0.34	0.02	< 0.01	PP62/0429 (IT31-97-E392)
				3	0.19	0.02	< 0.01	
				7	0.19	0.03	< 0.01	
				9	0.15	0.02	< 0.01	
				13	0.08	0.01	< 0.01	
Italy, 1997 (Pajaro)	0.45	0.05		0	0.41	0.04	0.01	PP62/0429 (IT31-97-E393)
				3	0.29	0.04	< 0.01	
				7	0.15	0.03	< 0.01	
				9	0.11	0.03	< 0.01	
				13	0.12	0.02	< 0.01	
Spain, 1997 (Pajaro)	0.5	0.05	2	0	0.38	0.02	< 0.01	PP62/0429 (ES10-97-SE009)
				3	0.2	0.03	< 0.01	
				7	0.27	0.04	< 0.01	
				10	0.2	0.04	< 0.01	
				14	0.18	0.03	< 0.01	
Spain, 1997 (Pajaro)	0.5	0.05	2	0	0.35	0.02	< 0.01	PP62/0429 (ES10-97-SE109)
				3	0.48	0.04	< 0.01	
				7	0.33	0.04	< 0.01	
				10	0.4	0.04	< 0.01	
				14	0.19	0.02	< 0.01	
PROTECTED CROPS								
France (N), 2001 (Darselect)	0.38	0.065	2	-0	0.18	< 0.01	< 0.01	PP62/1208 (AF5959/SY/2)
				0	0.83 (c=0.02)	0.01	< 0.01	
				3	0.74 (c=< 0.01)	0.02	< 0.01	
				7	0.28 (c=0.01)	0.01	< 0.01	
UK, 2000 (Elsanta)	0.38	0.075	2	3	0.49	0.02	< 0.01	PP62/1017 (GB07-00-S095)
				6	0.38	0.02	< 0.01	
France (N), 2000 (Darselec)	0.38	0.13	2	3	0.28	< 0.01	< 0.01	PP62/1017 (FR75-00-S751)
				7	0.23	< 0.01	< 0.01	
UK, 2001 (Elsanta)	0.38+ 0.38	0.038+ 0.041	1+ 1	-0	0.03	< 0.01	< 0.01	PP62/1208 (AF5959/SY/3)
				0	0.15	< 0.01	< 0.01	
				3	0.16	< 0.01	< 0.01	
				7	0.14	< 0.01	< 0.01	
France (N), 2001 (Mara des Bois)	0.38+ 0.38	0.036+ 0.045	1+ 1	-0	0.14	0.03	< 0.01	PP62/1208 (AF5959/SY/1)
				0	1.3	0.04	< 0.01	
				3	0.94	0.05	< 0.01	
				7	0.31	0.03	< 0.01	
UK, 2001 (Elsanta)	0.38+ 0.38	0.074+ 0.069	1+ 1	-0	0.03	< 0.01	< 0.01	PP62/1208 (AF5959/SY/4)
				0	0.21	< 0.01	< 0.01	
				3	0.19	< 0.01	< 0.01	
				7	0.12	< 0.01	< 0.01	
Spain, 1997 (Camarrosa)	0.5	0.05	2	0	0.98	0.02	< 0.01	PP62/0427 (ES10-97-SE006)
				3	0.58	0.02	< 0.01	
				7	0.44	< 0.01	< 0.01	
				10	0.36	< 0.01	< 0.01	
				14	0.22	0.01	< 0.01	
Italy, 1997 (Marmolada)	0.5	0.05	2	0	0.76	0.03	< 0.01	PP62/0427 (IT23-97-E394)
				3	0.54	0.04	< 0.01	
				7	0.51	0.04	< 0.01	
				10	0.29	0.03	< 0.01	
				13	0.64	0.04	< 0.01	

STRAWBERRY Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Italy, 1997 (Tulla)	0.5	0.05	2	0	1.9	0.08	< 0.01	PP62/0427 (IT24-97-E395)
				3	1.4	0.07	< 0.01	
				7	0.86	0.07	< 0.01	
				10	0.74	0.05	< 0.01	
				13	0.43	0.04	< 0.01	
Spain, 1997 (Camarrosa)	0.51	0.05	2	0	1.3	0.01	< 0.01	PP62/0427 (ES10-97-SE106)
				3	1.5	0.01	< 0.01	
				7	0.39	0.02	< 0.01	
				10	0.39	0.02	< 0.01	
				14	0.39	< 0.01	< 0.01	

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Onions

In trials on bulb onions in France, Germany, Italy, Spain and the UK, 2 foliar applications of pirimicarb (50% WG formulation) were made at 7–12 day intervals to unreplicated 30–60 square metre plots using knapsack sprayers and hand lances or mini-booms to obtain full foliar coverage. Mature bulbs (2 kg or 12 units) were sampled and trimmed before analysis using Method RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ of the methods was 0.01 mg/kg for all analytes and the mean recovery rates were 81–100% at fortification levels of 0.01–0.5 mg/kg.

Table 63. Residues in onions (bulb) from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France, Germany, Italy, Spain and the UK.

ONIONS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[3]	R238177	
Germany, 2000 (Macho)	0.25	0.042	1	-0	< 0.01	< 0.01	< 0.01	PP62/0002 (DE11-00-S166)
			2	0	< 0.01	< 0.01	< 0.01	
				3	< 0.01	< 0.01	< 0.01	
				7	< 0.01	< 0.01	< 0.01	bulbs lightly
				13	≤ 0.01	≤ 0.01	< 0.01	washed
				21	< 0.01	< 0.01	< 0.01	
Germany, 2000 (Hystar)	0.25	0.042	1	-0	< 0.01	< 0.01	< 0.01	PP62/0002 (DE16-00-S166)
			2	0	< 0.01	< 0.01	< 0.01	
				3	< 0.01	< 0.01	< 0.01	
				7	< 0.01	< 0.01	< 0.01	bulbs lightly
				14	≤ 0.01	≤ 0.01	< 0.01	washed
				21	< 0.01	< 0.01	< 0.01	
Germany, 1998 (Kaigaro)	0.25	0.063	2	14	0.04[1]	< 0.01	< 0.01	PP62/0281 (RS-9825-E1)
Germany, 1998 (various)	0.25	0.063	2	12	< 0.01[1]	< 0.01	< 0.01	PP62/0281 (GB01-00-S094)
France (N), 2000 (Summit)	0.25	0.083	1	-0	< 0.01	< 0.01	< 0.01	PP62/1015 (FR41-00-S761)
			2	0	0.08	< 0.01	< 0.01	
				3	0.02	< 0.01	< 0.01	
				6	< 0.01	< 0.01	< 0.01	
				14	≤ 0.01	≤ 0.01	< 0.01	
				23	< 0.01	< 0.01	< 0.01	
France (N), 2000 (Hystar)	0.25	0.083	1	-0	< 0.01	< 0.01	< 0.01	PP62/1015 (FR72-00-S751)
			2	0	0.07	< 0.01	< 0.01	
				3	0.02	< 0.01	< 0.01	
				7	0.01	< 0.01	< 0.01	
				14	≤ 0.01	≤ 0.01	< 0.01	
				19	< 0.01	< 0.01	< 0.01	
UK, 1998 (Balstora)	0.25	0.083	2	14	≤ 0.01	≤ 0.01	< 0.01	PP62/0284 (GB51-98-S191)
UK, 1998 (Goldito)	0.25	0.083	2	14	≤ 0.01	≤ 0.01	< 0.01	PP62/0284 (GB51-98-S192)

ONIONS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[3]	R238177	
Italy, 2001 (Density)	0.4	0.05	1 2	-0 0 3 7	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/01204 (AF/5973/SY/3)
Italy, 2001 (Density)	0.41	0.05	1 2	-0 0 3 7	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/01204 (AF/5973/SY/4)
Italy, 1997 (Bionda Voghera)	0.44+ 0.83	0.05+ 0.05	1+ 1	0 3 7 10 10+4[2]	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0273 (AF/3457/ZE/4)
Spain, 2001 (Barleta)	0.51	0.05	1 2	-0 0 3 7	0.01 0.11 0.02 < 0.01	< 0.01 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/01204 (AF/5973/SY/1)
Spain, 2001 (Morida de Amposta)	0.51	0.05	1 2	-0 0 3 7	0.01 0.03 0.04 0.05	< 0.01 < 0.01 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01	PP62/01204 (AF/5973/SY/2)
Spain, 1997 (Valenciana)	0.61	0.05	2	0 3 7 10 10+4[2]	0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0273 (AF/3457/ZE/2)
Spain, 1997 (Babosa)	0.84	0.05	2	0 3 7 10 10+4[2]	0.04 0.06 0.05 0.03 0.02	0.02 0.03 0.02 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0273 (AF/3457/ZE/1)
Italy, 1997 (Blanco Duro)	0.88+ 1.2	0.05+ 0.05	1+ 1	0 3 7 10 10+4[2]	0.03 0.01 0.02 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0273 (AF/3457/ZE/3)

[1]) Analysis of bulbs and leaves

[2]) Plants lifted 10 days after treatment and dried 4 days before field sampling

[3]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Cauliflower

In trials on cauliflowers from France and the UK, 2–5 foliar applications of pirimicarb (50% WG or SG formulations) were made at 7–14 day intervals to unreplicated 15–100 square metre plots, using small plot sprayers with mini-booms to obtain full foliar coverage. Wetting agents were included in most trials. Mature heads (12 units) were sampled and trimmed before analysis using Method RAM 15/02 (GC-MSD detection) to measure residues of pirimicarb and the combined residues of demethyl pirimicarb (R34836) and demethylformamido pirimicarb (R34885) or Methods RAM 265/01 (with GC-NPD detection) or RAM 265/03 (with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQs were 0.01 mg/kg (RAM 265/01 and RAM 265/03) and 0.05 mg/kg (RAM 15/02) in the trials from France and the 1997 UK trials. The mean recovery rates were 95–106% at fortification levels of 0.02–0.2 mg/kg.

Table 64. Residues in cauliflower from foliar applications of pirimicarb (50% WG or SG formulations) in supervised trials in France and the UK.

CAULIFLOWER Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[2]	R238177	
UK, 1988 (not specified)	0.13[1]	0.02	4	3	0.02	0.02		PP62/0333 (GB88-88-E905)
UK, 1988 (Vernon)	0.13[1]	0.02	4	3	0.02	< 0.01		PP62/0333 (GB88-88-E903)
UK, 1988 (not specified)	0.21	0.03	4	3	0.01	≤ 0.01		PP62/0330 (GB88-88-E905)
UK, 1988 (Vernon)	0.21	0.03	4	3	0.04	≤ 0.01		PP62/0333 (GB88-88-E903)
UK, 1991 (Batsman)	0.21	0.05	3	3 7	0.02 0.01	≤ 0.01 < 0.01		PP62/0310 (GB15-91-S072)
UK, 1991 (Batsman)	0.21	0.05	5	3 7	0.01 < 0.01	≤ 0.01 < 0.01		PP62/0310 (GB15-91-S072)
UK, 1991 (Batsman)	0.21	0.05	3	3 7	0.01 < 0.01	≤ 0.01 < 0.01		PP62/0310 (GB51-91-S072)
UK, 1991 (Batsman)	0.21	0.05	5	3 7	0.02 0.02	≤ 0.01 < 0.01		PP62/0310 (GB51-91-S072)
UK, 1992 (Carlos)	0.21	0.05	2	3	0.04	0.01		PP62/0311 (GB11-92-S061)
UK, 1992 (Carlos)	0.21	0.05	4	3	0.03	0.01		PP62/0311 (GB11-92-S061)
UK, 1992 (Plana)	0.21	0.05	2	3	0.05	≤ 0.01		PP62/0311 (GB11-92-S062)
UK, 1992 (Plana)	0.21+ 0.23	0.05+ 0.05	2+ 2	3	0.05	0.01		PP62/0311 (GB11-92-S062)
UK, 1997 (Commander)	0.25	0.06	2	0 3 7 10 14	< 0.05 ≤ 0.05 < 0.05 < 0.05 < 0.05	< 0.05 ≤ 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	PP62/0320 (GB15-97-S091)
UK, 1997 (Commander)	0.25	0.06	3	0 3 7 10 14	0.11 ≤ 0.05 < 0.05 < 0.05 < 0.05	< 0.05 ≤ 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	PP62/0320 (GB15-97-S091)
UK, 1997 (Talbot)	0.25	0.06	2	0 3 7 10 14	0.06 ≤ 0.05 < 0.05 < 0.05 < 0.05	< 0.05 ≤ 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	PP62/0320 (GB15-97-S093)
UK, 1997 (Talbot)	0.25	0.06	3	0 3 7 10 14	0.16 ≤ 0.05 < 0.05 < 0.05 < 0.05	< 0.05 ≤ 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	PP62/0320 (GB15-97-S093)
France (S), 1994 (Nautilus)	0.38	0.13	3	2 7	0.22 0.08	< 0.05 < 0.05	< 0.05 < 0.05	PP62/0344 (S602.95)
France (S), 1994 (Nautilus)	0.5	0.17	3	2 7	0.21 0.1	0.05 < 0.05	< 0.05 < 0.05	PP62/0344 (S602.95)

[1]) co-formulated with cyhalothrin

[2]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Broccoli

In trials on broccoli (calabrese) from the UK, 2–5 foliar applications of pirimicarb (50% WG or SG formulations) were made at 14 day intervals to unreplicated 30–50 square metre plots, using small plot sprayers with mini-booms to obtain full foliar coverage. Wetting agents were included in these trials. Mature heads (12 units) were sampled and trimmed before analysis using Method RAM 15/02

with GC-MSD detection to measure residues of pirimicarb and the combined residues of demethyl pirimicarb (R34836) and desmethylformamido pirimicarb (R34885), expressed as demethyl pirimicarb. The LOQ of the method was 0.01 mg/kg for these analytes and the mean recovery rates were 84–91% at fortification levels of 0.02–0.2 mg/kg.

Table 65. Residues in broccoli (calabrese) from foliar applications of pirimicarb (50% WG or SG formulations) in supervised trials from the UK.

BROCCOLI Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
UK, 1991 (Shogun)	0.21	0.05	2	1 7	0.02 < 0.01	< 0.01 < 0.01		PP62/0310 (GB51-91-S071)
UK, 1992 (Marathon)	0.21	0.05	2	3	≤ 0.01	≤ 0.01		PP62/0311 (GB11-91-S063)
UK, 1992 (Shogun)	0.22	0.05	2	3	≤ 0.01	≤ 0.01		PP62/0311 (GB11-92-S064)
UK, 1991 (Shogun)	0.21	0.05	3	3 7	0.39 0.22	0.08 0.05		PP62/0310 (GB15-91-S071)
UK, 1992 (Shogun)	0.21	0.05	3	3	≤ 0.01	≤ 0.01		PP62/0311 (GB11-92-S064)
UK, 1991 (Shogun)	0.21	0.05	4	3 7	0.01 < 0.01	≤ 0.01 < 0.01		PP62/0310 (GB51-91-S071)
UK, 1992 (Marathon)	0.21	0.05	4	3	≤ 0.01	≤ 0.01		PP62/0311 (GB11-92-S063)
UK, 1991 (Shogun)	0.21	0.05	5	3 7	0.41 0.23	0.09 0.05		PP62/0310 (GB15-91-S071)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Brussels sprouts

In trials on Brussels sprouts from Germany and the UK, 3–4 foliar applications of pirimicarb (50% WG or WP formulations) were made at 10–35 day intervals to unreplicated plots, using small plot sprayers with mini-booms to obtain full foliar coverage. Wetting agents were included in these trials. Methods RAM 15/01 and RAM 15/02 (both with GC-NPD detection) were used to measure residues of pirimicarb and the combined residues of desmethyl pirimicarb (R34836) and demethylformamido pirimicarb (R34885), expressed as demethyl pirimicarb. The LOQs of the methods were 0.01 mg/kg and 0.02 mg/kg for the metabolites in the trials in Germany. The mean recovery rates were 73–87% at fortification levels of 0.1–0.5 mg/kg.

Table 66. Residues in Brussels sprouts from foliar applications of pirimicarb (50% WP or WG formulations) in supervised trials from Germany and the UK.

BRUSSELS SPROUTS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[2]	R238177	
UK, 1988 (Lorris)	0.13[1]	0.02	4	3	0.03	0.01		PP62/0330 (GB88-88-E906)
UK, 1988 (not specified)	0.13[1]	0.02	4	3	0.03	0.01		PP62/0330 (GB88-88-E907)
Germany, 1982 (Lancelot)	0.15	0.03	3	0 3 10 14 21	0.12 0.03 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 (c=0.07)		PP62/0329 (DEU82 I 008 01 RU910)
Germany, 1982 (Lunet)	0.15	0.03	3	0 3 7 14 21	0.1 0.07 < 0.01 0.02 < 0.01	< 0.02 < 0.02 < 0.02 ≤ 0.02 < 0.02		PP62/0329 (DEU82 I 008 01 RT041)

BRUSSELS SPROUTS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[2]	R238177	
Germany, 1982 (not specified)	0.15	0.03	3	0 3 7 14 21	0.35 (c=0.04) 0.08 (c= 0.01) 0.02 < 0.01 < 0.01	0.03 0.02 ≤ 0.02 < 0.02 < 0.02		PP62/0329 (DEU82 I 008/01)
UK, 1988 (Lorris)	0.21	0.03	4	3	0.04	0.01		PP62/0330 (GB88-88-E906)
UK, 1988 (not specified)	0.21	0.03	4	3	0.04	0.02		PP62/0330 (GB88-88-E907)
Germany, 1982 (not specified)	0.3	0.03	3	0 3 7 14 21	0.38 0.15 0.15 0.2 (c=0.13) 0.06	< 0.02 0.05 0.03 0.06 (c=0.03) 0.03		PP62/0329 (DEU82 I 008 02)
Germany, 1982 (Wilhelmburg)	0.15+ 0.3	0.03+ 0.03	2+ 1	0 3 7 14 21	0.15 0.05 0.03 < 0.01 < 0.01	< 0.02 ≤ 0.02 < 0.02 < 0.02 < 0.02		PP62/0329 (DEU82 I 008)

[1]) co-formulated with cyhalothrin

[2]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Cabbage

In trials on cabbage from France, Germany and the UK, 2–3 foliar applications of pirimicarb (50% WG formulations) were made at 7–12 day intervals to unreplicated 30-100 square metre plots, using small plot sprayers or knapsacks with mini-booms to obtain full foliar coverage. Wetting agents were included in most trials. Mature heads (12 units) were sampled and trimmed before analysis using Methods RAM 265/01 (using GC-NPD detection to measure residues of pirimicarb and the combined residues of demethyl pirimicarb (R34836) and demethylformamido pirimicarb (R34885) or Methods RAM 265/02 or RAM 265/03 (both with GC-NPD detection) or RAM 265/04 (HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQs were 0.05 mg/kg and 0.01 mg/kg in the more recent (2001) trials in the UK. The mean recovery rates were 68–103% at fortification levels of 0.05–0.5 mg/kg.

Table 67. Residues in cabbage from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France, Germany and the UK.

CABBAGE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Germany, 2000 (Julius) Savoy cabbage	0.38	0.06	1 2	-0 0 3 7 9 14	< 0.05 < 0.05 < 0.05 ≤ 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	PP62/1014 (DE11-00-S164)
Germany, 2000 (Pedrillo) White cabbage	0.38	0.06	1 2	-0 0 3 7 9 14	< 0.05 < 0.05 < 0.05 ≤ 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 ≤ 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	PP62/1014 (DE11-00-S264)

CABBAGE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Germany, 2000 (Rodon) Red cabbage	0.38	0.06	1 2	-0 0 3 6 10 13	< 0.05 0.05 < 0.05 ≤ 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 ≤ 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	PP62/1014 (DE16-00-S164)
Germany, 2000 (Transam) White cabbage	0.38	0.06	1 2	-0 0 3 7 10 15	< 0.05 0.05 < 0.05 ≤ 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 ≤ 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	PP62/1014 (DE16-00-S264)
UK, 2001 (Tundra) Head cabbage	0.38	0.06	1 2	-0 0 3 7	< 0.01 0.18 0.02 0.01	0.01 0.03 0.03 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1198 (AF/5975/SY/2)
UK, 2001 (Wintessa) Head cabbage	0.38	0.07	1 2	-0 0 3 7	< 0.01 0.09 0.01 0.03	< 0.01 0.02 0.01 0.03	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1198 (AF/5975/SY/1)
UK, 1996 (Bison)	0.38	0.13	2	0 3 7	0.13 < 0.05 ≤ 0.05	< 0.05 < 0.05 ≤ 0.05	< 0.05 < 0.05 < 0.05	PP62/0349 (GB11-96-S201)
UK, 1996 (Krypton)	0.38	0.13	2	3	< 0.05	< 0.05	< 0.05	PP62/0349 (GB11-96-S202)
France (S), 1994 (Wirosa)	0.38	0.13	3	2 7	0.21 0.06	0.1 0.06		PP62/0344 (S603.95)
France (S), 1994 (Wirosa)	0.5	0.17	3	2 7	0.28 0.07	0.12 0.08		PP62/0344 (S603.95)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Kale

In trials on kale from the UK, 2–3 foliar applications of pirimicarb (50% WG formulations) were made at 6–12 day intervals to unreplicated 36–120 square metre plots, using small plot sprayers or precision mini-booms to obtain full foliar coverage of plants 30–100 cm in height. Wetting agents were included in most trials. Leaves and petioles (2kg min) were sampled from at least 12 plants and analysed using Methods RAM 319/01 (with GC-NPD detection), RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ were 0.05 mg/kg or 0.01 mg/kg in the trials used in the processing studies. The mean recovery rates were 86–103% at fortification levels of 0.05–2.0 mg/kg.

Table 68. Residues in kale (leaves and petioles) from foliar applications of pirimicarb (50% WG formulation) in supervised trials from the UK.

KALE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
UK, 1997 (Maris Kestral)	0.25	0.08	2	0 3 7 10 14	3.7 0.07 < 0.05 < 0.05 < 0.05	1.7 0.25 0.05 < 0.05 < 0.05	< 0.05 ≤ 0.05 < 0.05 < 0.05 < 0.05	PP62/0316 (GB14-97-S101) 55-80cm plants game cover crop
UK, 1997 (Keeper)	0.25	0.08	2	0 3 7 10 14	3.7 0.08 < 0.05 < 0.05 < 0.05	0.1.3 0.17 < 0.05 < 0.05 < 0.05	< 0.05 ≤ 0.05 < 0.05 < 0.05 < 0.05	PP62/0316 (GB14-97-S102) 50-80cm plants game cover crop

KALE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
UK, 1997 (Marrow Stem)	0.25	0.08	2	0 3 7 10 14	2.8 ≤ 0.05 < 0.05 < 0.05 < 0.05	1.6 0.19 < 0.05 < 0.05 < 0.05	0.05 ≤ 0.05 < 0.05 < 0.05 < 0.05	PP62/0316 (GB14-97-S103) 55-100cm plants animal fodder
UK, 1997 (Maris Kestral)	0.25	0.08	3	0 3 7 10 14	3.6 0.15 < 0.05 < 0.05 < 0.05	1.6 0.42 < 0.05 < 0.05 < 0.05	< 0.05 ≤ 0.05 < 0.05 < 0.05 < 0.05	PP62/0316 (GB14-97-S101) 55-80cm plants game cover crop
UK, 1997 (Keeper)	0.25	0.08	3	0 3 7 10 14	4.9 0.09 < 0.05 < 0.05 < 0.05	1.6 0.24 < 0.05 < 0.05 < 0.05	0.08 ≤ 0.05 < 0.05 < 0.05 < 0.05	PP62/0316 (GB14-97-S102) 50-80cm plants game cover crop
UK, 1997 (Marrow Stem)	0.25	0.08	3	0 3 7 10 14	3.4 0.05 < 0.05 < 0.05 < 0.05	1.7 0.21 < 0.05 < 0.05 < 0.05	0.05 ≤ 0.05 < 0.05 < 0.05 < 0.05	PP62/0316 (GB14-97-S103) 55-100cm plants animal fodder
UK, 2000 (Winterbor) curly kale	0.38	0.09	2	4	0.31	0.51	< 0.01	PP62/1290 (GB05-00-S181) 50-60 cm plants Processing study
UK, 2000 (Winterbor) curly kale	0.38	0.09	2	4	0.35	0.58	< 0.01	PP62/1290 (GB05-00-S182) 84-93 cm plants Processing study
UK, 2004 (Reflex)	0.50	0.13	2	3	1.2	3.1	< 0.01	PP62/1450 (AF/8042/SY/1) 30-35cm plants Processing study

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Cucumber

In trials on outdoor and protected cucumbers from France, Italy, Spain and the UK, 2–5 foliar applications of pirimicarb (50% WG formulations) were made at 7–11 day intervals to unreplicated 20–100 square metre plots, using knapsack sprayers with hand lances, or minibooms to obtain full foliar coverage of plants. Field crops were 30–40 cm in height and protected crops ranged from 1–2.3 metres in height. 1.5–5kg samples (12 units) were analysed using Methods RAM 15/02, using GC-NPD detection to measure residues of pirimicarb and the combined residues of demethyl pirimicarb (R34836) and demethylformamido pirimicarb (R34885) or Methods RAM 265/02 (GC-NPD detection), RAM 265/03 (with either GC-NPD or HPLC-MS-MS detection) or RAM 265/04 (HPLC-MS-MS dtection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 74–90% at fortification levels of 0.01–1.0 mg/kg.

Table 69. Residues in outdoor cucumbers from foliar applications of pirimicarb (50% WG formulation) in supervised trials in Italy and Spain.

CUCUMBERS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Italy, 1997 (Jazzer)	0.35	0.05	2	0	0.22	0.05	< 0.01	PP62/0380 (IT42-97-E384)
				3	0.22	0.02	< 0.01	
				7	< 0.01	< 0.01	< 0.01	
				10	< 0.01	< 0.01	< 0.01	
				14	< 0.01	< 0.01	< 0.01	
Spain, 1997 (Marquetemore)	0.52	0.05	2	0	0.06	< 0.01	< 0.01	PP62/0394 (ES10-97-SE016)
				3	0.03	0.02	< 0.01	
				7	0.02	0.02	< 0.01	
				10	0.01	0.01	< 0.01	
				14	< 0.01	< 0.01	< 0.01	
Spain, 1997 (Marquetemore)	0.53	0.05	2	0	0.06	< 0.01	< 0.01	PP62/0394 (ES10-97-SE116)
				3	0.02	< 0.01	< 0.01	
				7	0.02	≤ 0.01	< 0.01	
				10	0.01	< 0.01	< 0.01	
				14	< 0.01	< 0.01	< 0.01	
Italy, 1997 (Market Moore 76)	0.4+ 0.44	0.05+ 0.05	1+ 1	0	0.01	< 0.01	< 0.01	PP62/0380 (IT52-97-E383)
				3	≤ 0.01	≤ 0.01	< 0.01	
				7	< 0.01	< 0.01	< 0.01	
				10	< 0.01	< 0.01	< 0.01	
				13	< 0.01	< 0.01	< 0.01	

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Table 70. Residues in protected cucumbers from foliar applications of pirimicarb (50% WG formulation) in supervised trials from France, Italy, Spain and the UK.

CUCUMBERS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
UK, 2001 (Frieda)	0.34	0.025	1	-0	0.03	0.03	< 0.01	PP62/1228 (AF/5965/SY/5)
			2	0	0.10	0.02	< 0.01	
				3	0.08	0.04	< 0.01	
				7	0.04	0.04	< 0.01	
UK, 2001 (Frieda)	0.37	0.025	2	-0	0.05	0.04	< 0.01	PP62/1228 (AF/5965/SY/6)
				0	0.17	0.04	< 0.01	
				3	0.12	0.05	< 0.01	
				7	0.04	0.04	< 0.01	
Italy, 1998 (Cherokee)	0.6	0.036	2	7	0.02	0.03	< 0.01	PP62/0384 (AF/4169/CL/3)
France (S), 1992 (Early)	0.21+ 0.38	0.021+ 0.038	1+ 3	-0	< 0.01	< 0.01		PP62/0405 (S323-92)
				0	0.06	0.01		
				3	0.09	0.05		
				7	0.03	0.03		
				14	< 0.01	0.03		
France (N), 1992 (Leen de Mos 804)	0.38	0.038	2	-0	0.04	0.04		PP62/0405 (S209-92)
			3	0	0.15	0.03		
				3	0.11	0.05		
				7	0.04	0.07		
				14	< 0.01	0.02		
France (N), 1992 (Pandorex)	0.38	0.038	3	-0	0.01	0.03		PP62/0405 (S210-92)
			4	0	0.14	0.04		
				3	0.04	0.03		
				7	0.03	0.03		
				14	< 0.01	0.01		
France (S), 1992 (Girola)	0.38	0.038	4	-0	0.04	0.03		PP62/0405 (S342-92)
			5	0	0.13	0.03		
				7	0.03	0.03		
				14	< 0.01	< 0.01		

CUCUMBERS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Italy, 1997 (Turner)	0.4	0.05	2	0 3 7 10 14	0.24 0.04 < 0.01 < 0.01 < 0.01	0.03 0.03 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0380 (IT41-97-E382)
Spain, 1997 (Almeria)	0.46	0.05	2	0 3 7 10 14	0.29 0.22 0.13 0.05 0.02	0.04 0.05 0.06 0.04 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0394 (ES10-97- SE116)
Spain, 1998 (Dasher II)	0.6	0.05	2	7	< 0.01	< 0.01	< 0.01	PP62/0384 (AF/4169/CL/2)
Italy, 1998 (Jasser)	0.6	0.05	2	7	< 0.01	< 0.01	< 0.01	PP62/0384 (AF/4169/CL/4)
Spain, 1998 (Darina)	0.61	0.05	2	7	< 0.01	< 0.01	< 0.01	PP62/0384 (AF/4169/CL/1)
Spain, 1997 (Almeria)	0.44+ 0.49	0.05	2	0 3 7 10 14	0.19 0.16 0.07 0.07 0.02	0.02 0.05 0.04 0.05 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0394 (ES10-97- SE016)
Italy, 1997 (Green Fall)	0.4+ 0.45	0.05+ 0.05	1+ 1	0 3 7 10 14	0.41 0.17 0.06 < 0.01 < 0.01	0.03 0.05 0.04 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0380 (IT51-97-E385)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Summer squash

In trials on outdoor and protected summer squash (courgettes) from France and Italy, 2 foliar applications of pirimicarb (50% WG formulations) were made at 10–12 day intervals to unreplicated 76–100 square metre plots, using knapsack or small plot mini-boom sprayers to obtain full foliar coverage of plants 30–70 cm in height. Samples (2 kg or 12 units) were analysed using Method RAM 265/04 with HPLC-MS-MS detection to measure residues of pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 84–90% at fortification levels of 0.01–1.0 mg/kg.

Table 71. Residues in outdoor summer squash (courgettes) from foliar applications of pirimicarb (50% WG formulation) in supervised trials from France and Italy.

SUMMER SQUASH Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (S), 2001 (Tolka)	0.47	0.05	1 2	-0 0 3 7	< 0.01 0.28 0.06 < 0.01	< 0.01 0.02 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1191 (AF/5982/SY/2)
France (S), 2001 (Cora)	0.49	0.05	1 2	-0 0 3 7	< 0.01 0.06 0.04 0.01	< 0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1191 (AF/5982/SY/1)
Italy, 2001 (Carisma)	0.5	0.05	1 2	-0 0 3 7	< 0.01 0.23 0.03 0.02	< 0.01 0.02 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1191 (AF/5982/SY/3)

SUMMER SQUASH Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Italy, 2001 (Pamela)	0.5	0.05	1	-0	< 0.01	< 0.01	< 0.01	PP62/1191 (AF/5982/SY/4)
			2	0	0.43	0.01	< 0.01	
				3	0.14	0.02	< 0.01	
				7	< 0.01	< 0.01	< 0.01	

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Table 72. Residues in protected summer squash (courgettes) from foliar applications of pirimicarb (50% WG formulation) in supervised trials from France.

SUMMER SQUASH Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (N), 2001 (Tosca)	0.38	0.047	1	-0	< 0.01	< 0.01	< 0.01	PP62/1228 (AF/5965/SY/1)
			2	0	0.11	< 0.01	< 0.01	
				3	0.05	< 0.01	< 0.01	
				7	0.01	< 0.01	< 0.01	
France (N), 2001 (Cora)	0.38+ 0.38	0.047+ 0.055	1+ 1	-0	< 0.01	< 0.01	< 0.01	PP62/1228 (AF/5965/SY/2)
				0	0.14	0.01	< 0.01	
				3	0.08	0.01	< 0.01	
				7	< 0.01	< 0.01	< 0.01	

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Melons

In trials on outdoor and protected melons from France, Italy and Spain, 2 foliar applications of pirimicarb (50% WG formulations) were made at 7−13 day intervals to unreplicated 40−120 square metre plots, using knapsack (hand lance or mini boom) sprayers to obtain full foliar coverage. Plants grown in the field ranged in height from 20–40 cm while those under protection were from 60–260cm in height. Samples (min 12 units) were analysed using Methods RAM 265/03 (with HPLC-MS-MS and GC-NPD detection) or RAM 265/04 with HPLC-MS-MS detection to measure residues of pirimicarb and its carbamate metabolites. In some trials, peel and pulp were analysed separately and the calculated whole fruit residues are reported below. The LOQ was 0.01 mg/kg and mean recovery rates were 77−100% at fortification levels of 0.01−5.0 mg/kg.

Table 73. Residues in outdoor melons from foliar applications of pirimicarb (50% WG formulation) in supervised trials in Italy and Spain.

MELONS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Italy, 2001 (Tamaris)	0.4	0.05	1	-0	< 0.01	0.02	< 0.01	PP62/1099 (AF/5966/SY/3)
			2	0	0.2	0.02	< 0.01	
				3	0.03	0.02	< 0.01	
				7	< 0.01	0.01	< 0.01	calculated[2]
Italy, 1997 (Soleado)	0.45	0.05	2	0	0.11	< 0.01	< 0.01	PP62/0928 (IT52-97-E397)
				3	0.06	0.04	< 0.01	
				7	0.01	0.02	< 0.01	
				10	< 0.01	< 0.01	< 0.01	
				14	< 0.01	< 0.01	< 0.01	
Italy, 2001 (Pamir)	0.45	0.05	1	-0	< 0.01	< 0.01	< 0.01	PP62/1099 (AF/5966/SY/4)
			2	0	0.05	0.01	< 0.01	
				3	0.06	0.02	< 0.01	
				7	< 0.01	0.01	< 0.01	calculated[2]

MELONS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (S), 2001 (Escrito)	0.45+ 0.5	0.05+ 0.05	1+ 1	-0 0 3 7	< 0.01 0.09 0.03 < 0.01	< 0.01 0.02 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1099 (AF/5966/SY/1) calculated[2]
France (S), 2001 (Matisse)	0.46	0.05	1 2	-0 0 3 7	< 0.01 0.12 <u>0.11</u> 0.04	< 0.01 0.01 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1099 (AF/5966/SY/2) calculated[2]
Spain, 1997 (Braco)	0.47+ 0.52	0.05+ 0.05	1+ 1	0 3 8 10 14	0.06 0.02 < 0.01 < 0.01 < 0.01	0.01 0.02 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0928 (ES10-97-SE305)
Spain, 1997 (Braco)	0.51	0.05	2	0 3 8 10 14	0.05 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 < 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0928 (ES10-97-SE205)
Italy, 1997 (Calypso)	0.5+ 0.56	0.05+ 0.05	1+ 1	0 3 7 10 14	0.09 0.03 0.01 0.01 < 0.01	0.01 0.02 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0928 (IT24-97-E399)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

[2]) calculated from peel and pulp residues

Table 74. Residues in protected melons from foliar applications of pirimicarb (50% WG formulation) in supervised trials from Italy and Spain.

MELONS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (N), 1998 (Talma)	0.21	0.03	2	3	0.01	0.01	< 0.01	PP62/0398 (AF/4170/CL1) calculated[2]
France (N), 2001 (Buffalo)	0.37	0.03	1 2	-0 0 3 7	0.01 0.06 <u>0.04</u> 0.03	0.01 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1181 (AF/5967/SY/2) calculated[2]
France (N), 1998 (Ontario)	0.38	0.03	2	3	<u>0.13</u>	0.03	< 0.01	PP62/0398 (AF/4170/CL3) calculated[2]
France (N), 2002 (Cezanne)	0.34+ 0.41	0.03+ 0.03	1+ 1	-0 0 3 7	< 0.01 0.09 <u>0.04</u> 0.02	0.01 0.01 0.2 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1301 (AF/6541/SY/1) calculated[2]
Italy, 1997 (Soleado)	0.45	0.05	2	0 3 7 10 14	0.08 <u>0.02</u> 0.02 < 0.01 < 0.01	0.01 < 0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0928 (IT51-97-E398)
Italy, 1997 (Harper)	0.5	0.05	2	0 3 7 10 14	0.11 0.04 0.03 0.03 0.02	0.02 0.02 0.02 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0928 (IT22-97-E396)

MELONS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Italy, 1998 (Greso)	0.53+ 0.62	0.05+ 0.05	1+ 1	3	0.11	0.04	< 0.01	PP62/0392 (AF/4272/CL/4) calculated[2]
Spain, 1998 (Galia)	0.59	0.05	2	3	0.05	0.02	< 0.01	PP62/0392 (AF/4272/CL/2) calculated[2]
Italy, 1998 (Drake)	0.60	0.05	2	3	0.06	0.02	< 0.01	PP62/0392 (AF/4272/CL/3) calculated[2]
Spain, 1998 (Galia)	0.64+ 0.57	0.05+ 0.05	1+ 1	3	0.05	0.02	< 0.01	PP62/0392 (AF/4272/CL/1) calculated[2]
Spain, 1997 (Lunabel) Cantaloupe melon	0.74	0.05	2	0 3 7 10 14	0.13 0.04 0.03 0.02 0.01	0.03 0.03 0.03 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0928 (ES10-97-SE005)
Spain, 1997 (Lunabel) Cantaloupe melon	0.78+ 0.72	0.05+ 0.05	1+ 1	0 3 7 10 14	0.08 0.05 0.05 0.02 0.02	< 0.01 0.02 0.03 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0928 (ES10-97-SE105)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

[1]) calculated from peel and pulp residues

Table 75. Residues in melon (pulp) from foliar applications of pirimicarb (50% WG formulation) in supervised trials from France, Italy and Spain.

MELONS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Spain, 1998 (Galia)		0.05	2	3	0.02	≤ 0.01	< 0.01	PP62/0392 (AF/4272/CL/1) protected crop
Spain, 1998 (Galia)		0.05	2	3	0.01	≤ 0.01	< 0.01	PP62/0392 (AF/4272/CL/2) protected crop
Italy, 1998 (Drake)		0.05	2	3	0.02	≤ 0.01	< 0.01	PP62/0392 (AF/4272/CL/3) protected crop
Italy, 1998 (Greso)		0.05	2	3	0.03	0.01	< 0.01	PP62/0392 (AF/4272/CL/4) protected crop
France (N), 1998 (Talma)		0.025	2	3	< 0.01	< 0.01	< 0.01	PP62/0398 (AF/4170/CL1) protected crop
France (N), 1998 (Ontario)		0.025	2	3	0.06	0.02	< 0.01	PP62/0398 (AF/4170/CL1) protected crop

MELONS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (S), 2001 (Escrito)		0.05	1 2	-0 0 3 7	< 0.01 0.02 <u>0.02</u> < 0.01	< 0.01 < 0.01 <u>≤ 0.01</u> < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1099 (AF/5966/SY/1) field crop
France (S), 2001 (Matisse)		0.05	1 2	-0 0 3 7	< 0.01 0.03 <u>0.08</u> 0.04	< 0.01 < 0.01 <u>0.01</u> 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1099 (AF/5966/SY/2) field crop
Italy, 2001 (Tamaris)		0.05	1 2	-0 0 3 7	0.01 0.05 <u>0.02</u> < 0.01	0.02 < 0.01 <u>0.01</u> < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1099 (AF/5966/SY/3) field crop
Italy, 2001 (Pamir)		0.05	1 2	-0 0 3 7	< 0.01 < 0.01 <u>0.05</u> < 0.01	< 0.01 < 0.01 <u>≤ 0.01</u> < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1099 (AF/5966/SY/4) field crop
France (N), 2001 (Buffalo)		0.025	1 2	-0 0 3 7	< 0.01 0.01 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1181 (AF/5967/SY/2) protected crop
France (N), 2002 (Cezanne)		0.025	1 2	-0 0 3 7	< 0.01 0.03 0.03 0.02	0.01 < 0.01 0.26 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1301 (AF/6541/SY/1) protected crop

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Tomatoes

In trials on outdoor and protected tomatoes from France, Italy and Spain, 2 foliar applications of pirimicarb (50% WG formulations) were made at 7–12 day intervals to unreplicated 20–140 square metre plots, using small plot boom or knapsack (hand lance or mini boom) sprayers to obtain full foliar coverage. Plants grown in the field ranged in height from 30–70 cm (up to 90 cm for staked tomatoes) while those grown under protection were from 150–220cm in height. Samples (min 12 units or 2 kg) were analysed using Methods RAM 265/03 (GC-NPD detection) or RAM 265/04 (with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 83–100% at fortification levels of 0.01–0.5 mg/kg.

Table 76. Residues in outdoor tomatoes from foliar applications of pirimicarb (50% WG formulation) in supervised trials from France, Italy and Spain.

TOMATO Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (N), 2001 (Paola)	0.25	0.031	1 2	-0 0 3 7	< 0.01 0.31 0.02 0.02	< 0.01 0.02 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1201 (AF/5961/SY/4)
France (N), 2001 (Picnic)	0.25	0.032	1 2	-0 0 3 7	< 0.01 0.07 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1201 (AF/5961/SY/1)
France (N), 2001 (Valina)	0.25	0.032	1 2	-0 0 3 7	< 0.01 0.15 0.05 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1201 (AF/5961/SY/2)

TOMATO Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (N), 2001 (Daisy)	0.25	0.032	1 2	-0 0 3 7	< 0.01 0.04 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1201 (AF/5961/SY/3)
France (N), 2001 (Daisy)	0.38	0.046	1 2	-0 0 3 7	< 0.01 0.06 0.05 0.03	< 0.01 0.02 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1201 (AF/5961/SY/3)
France (N), 2000 (Picnic)	0.38	0.047	2	0 3 7 10 14	0.16 0.07 0.02 0.02 < 0.01	0.02 0.02 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0936 (AF/5080/ZE/1)
France (N), 2000 (Montfavet)	0.38	0.047	2	0 3 7 10 14	0.24 0.09 0.06 0.05 0.01	0.03 0.02 0.02 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0936 (AF/5080/ZE/3)
France (N), 2000 (Fournaise)	0.38	0.047	2	0 3 7 10 14	0.09 0.04 0.03 0.03 < 0.01	0.03 0.02 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0936 (AF/5080/ZE/4)
France (N), 2001 (Picnic)	0.38	0.047	1 2	-0 0 3 7	< 0.01 0.17 0.04 0.02	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1201 (AF/5961/SY/1)
France (N), 2001 (Valina)	0.38	0.047	1 2	-0 0 3 7	0.03 0.1 0.07 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1201 (AF/5961/SY/2)
France (N), 2000 (Fernova)	0.38	0.048	1 2	-0 3 7 10 14	0.11 0.02 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0936 (AF/5080/ZE/2)
France (N), 2001 (Paola)	0.38	0.049	1 2	-0 0 3 7	< 0.01 0.28 0.03 0.03	< 0.01 0.03 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1201 (AF/5961/SY/4)
Italy, 2001 (UC 82)	0.41	0.05	1 2	-0 0 3 7	0.02 0.15 0.03 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1202 (AF/5962/SY/3)
France (S), 2001 (Tokapi)	0.49	0.05	1 2	-0 0 3 7	0.07 0.11 0.16 0.08	0.01 0.01 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1202 (AF/5962/SY/2)
Italy, 1997 (Snob)	0.5	0.05	2	0 3 7 10 13	0.16 0.09 0.06 0.03 0.01	0.03 0.02 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0396 (IT41-97-E375)
France (S), 2001 (Lenor)	0.5	0.05	1 2	-0 0 3 7	0.01 0.06 0.08 0.03	< 0.01 0.01 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1202 (AF/5962/SY/1)
Italy, 1997 (Brigade)	0.6	0.05	2	0 3 7 10 13	0.12 0.1 0.06 0.03 0.02	0.02 0.02 0.02 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0396 (IT22-97-E376)

TOMATO Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Italy, 1997 (Red River)	0.6	0.05	2	3	0.1	0.04	< 0.01	PP62/0525 (IT33-97-E379) Processing study
Spain, 1997 (Royestar)	0.65	0.05	2	0	0.27	0.03	< 0.01	PP62/0396 (ES10-97-SE211)
				3	0.07	0.03	< 0.01	
				7	0.03	0.02	< 0.01	
				10	0.01	0.01	< 0.01	
				14	< 0.01	< 0.01	< 0.01	
Spain, 1997 (Bodar)	0.67	0.05	2	0	0.16	0.03	0.01	PP62/0396 (ES10-97-SE311)
				3	0.02	0.01	0.02	
				7	< 0.01	< 0.01	< 0.01	
				10	< 0.01	< 0.01	< 0.01	
				14	< 0.01	< 0.01	< 0.01	

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Table 77. Residues in protected tomatoes from foliar applications of pirimicarb (50% WG formulation) in supervised trials from France, Italy, Spain and the UK.

TOMATO Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
UK, 2001 (Jewel)	0.33	0.025	1	-0	< 0.01	< 0.01	< 0.01	PP62/1232 (AF/5963/SY/6)
			2	0	0.2	≤ 0.01	< 0.01	
				4	0.05	0.01	0.01	
				7	0.05	0.01	0.01	
France (N), 2001 (Recento)	0.35+ 0.38	0.025+ 0.025	1+ 1	-0	< 0.01	< 0.01	< 0.01	PP62/1232 (AF/5963/SY/3)
				0	0.1	≤ 0.01	< 0.01	
				3	0.05	< 0.01	< 0.01	
				7	0.03	< 0.01	< 0.01	
UK, 2001 (Solution)	0.36+ 0.33	0.025+ 0.025	1+ 1	-0	< 0.01	< 0.01	< 0.01	PP62/1232 (AF/5963/SY/5)
				0	0.1	≤ 0.01	< 0.01	
				3	0.1	0.02	0.01	
				7	0.01	< 0.01	< 0.01	
France (N), 2001 (Servanne)	0.37	0.025	1	-0	< 0.01	< 0.01	< 0.01	PP62/1232 (AF/5963/SY/2)
			2	0	0.07	≤ 0.01	< 0.01	
				3	0.04	< 0.01	< 0.01	
				7	< 0.01	< 0.01	< 0.01	
France (N), 2001 (Cindel)	0.37	0.025	1	-0	< 0.01	< 0.01	< 0.01	PP62/1232 (AF/5963/SY/4)
			2	0	0.1	≤ 0.01	< 0.01	
				3	0.07	< 0.01	< 0.01	
				7	0.05	< 0.01	< 0.01	
UK, 2000 (Espero)	0.39	0.025	2	0	0.2	≤ 0.01	< 0.01	PP62/0945 (GB07-00-S096)
				3	0.15	< 0.01	< 0.01	
				7	0.06	< 0.01	< 0.01	
				10	0.06	< 0.01	< 0.01	
				14	0.02	< 0.01	< 0.01	
UK, 2000 (Eloisa)	0.39	0.025	2	0	0.17	≤ 0.01	< 0.01	PP62/0945 (GB07-00-S097)
				3	0.12	< 0.01	< 0.01	
				7	0.02	< 0.01	< 0.01	
				10	0.01	< 0.01	< 0.01	
				14	< 0.01	< 0.01	< 0.01	
France (N), 2001 (Sympathie)	0.39	0.025	1	-0	< 0.01	< 0.01	< 0.01	PP62/1232 (AF/5963/SY/1)
			2	0	0.08	≤ 0.01	< 0.01	
				3	0.04	< 0.01	< 0.01	
				7	0.02	< 0.01	< 0.01	
Spain, 1997 (Royestar)	0.59+ 0.71	0.05+ 0.05	1+ 1	0	0.37	0.02	0.01	PP62/0396 (ES10-97-SE011)
				3	0.22	0.03	0.02	
				7	0.06	0.02	0.01	
				10	0.04	< 0.01	< 0.01	
				14	< 0.01	< 0.01	< 0.01	

TOMATO Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Spain, 1997 (Royestar)	0.59+ 0.71	0.05+ 0.05	1+ 1	0 3 7 10 14	0.3 0.21 0.11 0.1 0.01	0.02 0.02 0.02 0.01 < 0.01	0.01 0.01 < 0.01 < 0.01 < 0.01	PP62/0396 (ES10-97- SE111)
Italy, 1997 (Galaxy)	0.9	0.05	2	0 3 7 10 13	0.14 0.05 0.03 < 0.01 < 0.01	0.04 0.02 0.02 < 0.01 < 0.01	< 0.01 0.01 0.01 < 0.01 < 0.01	PP62/0396 (IT51-97-E378)
Italy, 1997 (ES200)	1.25	0.05	2	0 3 7 10 13	0.56 0.11 0.03 0.01 < 0.01	0.02 0.02 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0396 (IT29-97-E377)
France (S), 2003 (Quest)	1.3	0.10	2	3	0.43	0.03	0.01	PP62/1392 (AF/7363/SY/1) Processing study

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Peppers

In trials on outdoor and protected peppers from France, Italy, Spain and the UK, 2 foliar applications of pirimicarb (50% WG formulations) were made at 6–13 day intervals to unreplicated 18–60 square metre plots as broadcast, band or directed sprays, using knapsack (hand lance) sprayers to obtain full foliar coverage. Plants grown in the field ranged in height from 40–70 cm while those grown under protection were from 70–300cm in height. Samples (min 12 units or 2 kg) were analysed using Methods RAM 265/03 with GC-NPD or HPLC-MS-MS detection or RAM 265/04 (with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 81–96% at fortification levels of 0.01–0.5 mg/kg.

Table 78. Residues in outdoor peppers from foliar applications of pirimicarb (50% WG formulation) in supervised trials in Italy and Spain.

PEPPERS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Italy, 1997 (Antares)	0.4	0.05	2	0 3 7 10 13	0.13 0.01 < 0.01 < 0.01 < 0.01	< 0.01 ≤ 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0382 (IT52-97-E390)
Italy, 2001 (Ecolo) Peppers, sweet	0.45	0.05	1 2	-0 0 3 7	< 0.01 0.08 0.03 0.02	< 0.01 < 0.01 ≤ 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1206 (AF/5947/SY/3)
Spain, 1997 (Piquilo)	0.4+ 0.49	0.05+ 0.05	1+ 1	0 3 7 10 14	0.3 0.07 0.02 < 0.01 < 0.01	0.02 0.02 0.01 0.01 < 0.01	0.02 0.04 0.03 < 0.01 < 0.01	PP62/0382 (ES10-97- SE204)
Spain, 1997 (Piquilo)	0.4+ 0.49	0.05+ 0.05	1+ 1	0 3 7 10 14	0.2 0.03 0.01 < 0.01 < 0.01	0.02 0.01 < 0.01 < 0.01 < 0.01	0.02 0.03 0.02 < 0.01 < 0.01	PP62/0382 (ES10-97- SE304)

PEPPERS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Italy, 2001 (Ronaldo) Peppers, sweet	0.5	0.05	1 2	-0 0 3 7	< 0.01 0.14 <u>0.07</u> 0.03	< 0.01 < 0.01 <u>0.01</u> < 0.01	< 0.01 < 0.01 < 0.01 0.02	PP62/1206 (AF/5947/SY/4)
Spain, 2001 (Dulci Italico) Peppers, sweet	0.52	0.05	1 2	-0 0 3 7	< 0.01 0.36 <u>0.04</u> < 0.01	< 0.01 0.04 <u>0.02</u> 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1206 (AF/5947/SY/1)
Spain, 2001 (Dulci Italico) Peppers, sweet	0.52	0.05	1 2	-0 0 3 7	< 0.01 0.32 <u>0.05</u> 0.02	< 0.01 0.02 <u>0.02</u> 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1206 (AF/5947/SY/2)
Italy, 1997 (Lipari)	0.63	0.05	2	0 3 7 10 13	0.02 <u>0.02</u> 0.01 < 0.01 < 0.01	0.01 <u>0.01</u> < 0.01 < 0.01 < 0.01	0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0382 (IT41-97-E388)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Table 79. Residues in protected peppers from foliar applications of pirimicarb (50% WG formulation) in supervised trials from France, Italy, Spain and the UK.

PEPPERS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (N), 1998 (Argo) Peppers, sweet	0.19+ 0.24	0.025+ 0.025	1+ 1	3	0.02	< 0.01	< 0.01	PP62/0388 (AF/4171/CL/1)
France (N), 1998 (Elisa) Peppers, sweet	0.31+ 0.34	0.025+ 0.025	1+ 1	3	0.13	< 0.01	0.04	PP62/0388 (AF/4171/CL/2)
France (N), 2001 (Spartakus)	0.5	0.034	1 2	-0 0 3 7	< 0.01 0.04 0.04 0.03	< 0.01 < 0.01 < 0.01 < 0.01	0.01 < 0.01 0.01 0.01	PP62/1209 (AF/5964/SY/2)
France (N), 2001 (Vidi)	0.5	0.035	1 2	-0 0 3 6	< 0.01 0.15 <u>0.05</u> 0.01	< 0.01 < 0.01 <u>≤ 0.01</u> < 0.01	< 0.01 < 0.01 0.02 < 0.01	PP62/1209 (AF/5964/SY/1)
UK, 2001 (Cardio)	0.5	0.036	1 2	-0 0 3 7	< 0.01 0.13 <u>0.04</u> 0.02	< 0.01 < 0.01 <u>≤ 0.01</u> < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1209 (AF/5964/SY/4)
Italy, 1997 (Pathos)	0.4	0.05	2	0 3 7 10 13	0.04 <u>≤ 0.01</u> < 0.01 < 0.01 < 0.01	< 0.01 <u>≤ 0.01</u> < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0382 (IT32-97-E389)
Italy, 1997 (Argo)	0.5	0.05	2	0 3 7 10 13	0.04 <u>0.01</u> < 0.01 < 0.01 < 0.01	< 0.01 <u>≤ 0.01</u> < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0382 (IT51-97-E391)
Spain, 1998 (Cuerno de Cabra) Peppers, sweet	0.6	0.05	2	3	<u>0.05</u>	<u>0.01</u>	0.04	PP62/0390 (AF/4273/CL/2)
Italy, 1998 (Clause 1588) Peppers, sweet	0.6	0.05	2	3	<u>0.08</u>	<u>≤ 0.01</u>	0.02	PP62/0390 (AF/4273/CL/3)

PEPPERS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Spain, 1997 (Italiano Star F-1)	0.61	0.05	2	0 3 7 10 14	0.24 0.15 0.13 0.12 0.04	0.04 0.04 0.04 0.06 0.02	0.01 < 0.01 < 0.01 0.01 < 0.01	PP62/0382 (ES10-97-SE104)
Spain, 1998 (Dulce Italico) Peppers, sweet	0.61	0.05	2	3	0.18	0.04	0.03	PP62/0390 (AF/4273/CL/5)
Spain, 1997 (Italiano Star F-1)	0.6+ 0.65	0.05+ 0.05	1+ 1	0 3 7 10 14	0.24 0.14 0.06 0.04 < 0.01	0.05 0.03 0.03 0.02 0.02	< 0.01 0.01 < 0.01 < 0.01 < 0.01	PP62/0382 (ES10-97-SE004)
Italy, 1998 (Seinor) Peppers, sweet	0.62+ 0.57	0.05+ 0.05	2	3	0.08	≤ 0.01	0.02	PP62/0390 (AF/4273/CL/4)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Sweetcorn (kernels)

In trials on sweetcorn from France, 2 foliar applications of pirimicarb (50% WG formulations) were made at 7 day intervals to unreplicated 35–165 square metre plots, using knapsack and mini-booms to obtain full foliar coverage. Mature cobs (12 units) were sampled and kernals removed for analysis using Method RAM 15/02 with GC-NPD detection to measure residues of pirimicarb and the combined residues of demethyl pirimicarb (R34836) and demethylformamido pirimicarb (R34885). The LOQ was 0.01 mg/kg and the mean recovery rates were 88–98% at a fortification level of 0.1 mg/kg.

Table 80. Residues in sweet corn (kernels) from foliar applications of pirimicarb (50% WG formulation) to sweet corn in supervised trials from France.

SWEETCORN Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (S), 1992 (Jubilee)	0.24	0.05	2	7	≤ 0.01	≤ 0.01		PP62/0485 (S 551.92)
France (S), 1992 (Jubilee)	0.25	0.05	2	7	≤ 0.01	≤ 0.01		PP62/0485 (S 552.92)
France (S), 1992 (Reward)	0.2	0.05	2	7	≤ 0.01	≤ 0.01		PP62/0485 (S 321.92)
France (S), 1992 (Reward)	0.2	0.07	2	7	0.02	≤ 0.01		PP62/0485 (S 340.92)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Lettuce

The Meeting received information on trials conducted on outdoor lettuce from France and the UK and on protected lettuce in France, Italy, Spain and the UK. Lettuce types used in these trials included 'Iceberg-type' head lettuce, the less compact crisphead or butterhead lettuce and leaf lettuce. Where possible, these lettuce types have been identified in the following tables.

In these trials, two foliar applications of pirimicarb (50% WG formulations) were made at 7–12 day intervals to unreplicated 10–120 square metre plots, either as broadcast or band sprays using small plot boom or knapsack (hand lance or mini boom) sprayers to obtain full foliar coverage. Plants grown in the field received the second application between 39 and 51 days after planting while those

grown under protection were last treated between 28 and 53 days after planting (with a longer interval of 115 days in one UK trial). A minimum of 12 heads or whole plants (excluding roots) were taken for analysis and in most tials were trimmed to remove damaged outer leaves before analysis using Methods RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 79–91% at fortification levels of 0.01–20 mg/kg.

Table 81. Residues in lettuce (outdoor) from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France and UK.

LETTUCE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (N), 2002 (Triathlon) Leaf lettuce	0.25	0.04	1 2	-0 0 3 7 10 14	< 0.01 1.7 0.17 0.01 < 0.01 < 0.01	< 0.01 0.6 0.33 0.09 0.02 < 0.01	< 0.01 0.04 < 0.01 ≤ 0.01 < 0.01 < 0.01	PP62/1302 (AF/6540/SY/1)
UK, 2000 (Tozeas) Butterhead (Little gem)	0.25	0.06	2	3 7 14	0.09 ≤ 0.01 < 0.01	0.25 0.07 < 0.01	< 0.01 ≤ 0.01 < 0.01	PP62/1079 (GB02-00-S087)
UK, 2000 (Robinson) Iceberg	0.25	0.06	2	3 7 14	< 0.01 ≤ 0.01 < 0.01	< 0.01 ≤ 0.01 < 0.01	< 0.01 ≤ 0.01 < 0.01	PP62/1079 (GB02-00-S088)
France (N), 2000 (Panida)	0.25	0.06	2	3 7 14	0.36 0.02 < 0.01	0.79 0.29 0.04	< 0.01 ≤ 0.01 < 0.01	PP62/1079 (FR81-00-S750)
France (N), 2002 (Triathlon) Leaf lettuce	0.38	0.06	1 2	-0 0 3 7 10 14	< 0.01 2.6 0.29 0.02 < 0.01 < 0.01	< 0.01 1.27 0.52 0.16 0.05 < 0.01	< 0.01 0.08 0.01 < 0.01 < 0.01 < 0.01	PP62/1302 (AF/6540/SY/1)
UK, 2000 (Tozeas) Butterhead (Little gem)	0.38	0.09	2	3 7 14	0.13 0.02 < 0.01	0.43 0.15 < 0.01	< 0.01 < 0.01 < 0.01	PP62/1079 (GB02-00-S087)
UK, 2000 (Robinson) Iceberg	0.38	0.09	2	3 7 14	< 0.01 < 0.01 < 0.01	0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	PP62/1079 (GB02-00-S088)
France (N), 2000 (Panida)	0.38	0.09	2	3 7 14	0.63 0.06 < 0.01	0.97 0.52 0.05	< 0.01 < 0.01 < 0.01	PP62/1079 (FR81-00-S750)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Table 82. Residues in lettuce (protected) from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France, Italy, Spain and the UK.

LETTUCE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
France (N), 2002 (Batavia) Iceberg	0.15	0.02	1 2	-0 0 3 7 10 14 17 21	< 0.01 4.8 1.2 0.28 0.14 0.04 0.04 0.01	0.14 0.41 0.57 (c=0.02) 0.45 (c=0.02) 0.54 (c=0.02) 0.37 (c=0.01) 0.33 (c=0.01) 0.2	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/1)
France (N), 2002 (Batavia Angie) Iceberg	0.15	0.02	1 2	-0 0 3 7 10 14 17 21	0.02 2.2 0.44 0.1 0.02 < 0.01 < 0.01 < 0.01	0.11 0.31 0.55 0.4 0.22 0.14 0.11 0.1	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/3)
France (N), 2002 (Batavia Destinie) Iceberg	0.15	0.02	1 2	-0 0 3 7 10 14 17 21	0.02 2.3 0.68 0.23 0.05 0.03 0.01 < 0.01	0.17 0.43 0.54 0.65 0.31 0.27 0.18 0.18	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/4)
UK, 2002 (Emerald) Butterhead	0.15	0.03	1 2	-0 0 3 7 10 14 17 20	0.06 7.9 0.77 0.25 0.04 0.03 < 0.01 0.01	0.17 0.19 0.41 0.38 0.18 0.11 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/8)
France (N), 2002 (Noemi)	0.15+ 0.15	0.03+ 0.02	1+ 1	-0 0 3 7 10 14 17 21	0.02 1.1 0.35 0.1 0.04 0.02 0.02 0.01	0.08 0.18 0.31 0.26 0.19 0.18 0.17 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/2)
France (N), 2002 (Batavia) Iceberg	0.25	0.03	1 2	-0 0 3 7 10 14 17 21	< 0.01 3.9 1.5 0.59 0.19 0.12 0.04 < 0.01	0.08 0.38 0.4 (c=0.02) 0.45 (c=0.02) 0.41 (c=0.02) 0.42 (c=0.01) 0.27 (c=0.01) 0.12	< 0.01 < 0.01 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/1)
France (N), 2002 (Batavia Angie) Iceberg	0.25	0.03	1 2	-0 0 3 7 10 14 17 21	< 0.01 1.5 0.27 0.07 0.02 < 0.01 < 0.01 < 0.01	0.26 0.43 0.5 0.37 0.17 0.06 0.03 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/3)

LETTUCE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
UK, 2002 (Emerald) Butterhead	0.25	0.05	1 2	-0 0 3 7 10 14 17 20	0.37 12 2.3 1.1 0.36 0.11 0.03 0.03	0.38 0.39 0.63 0.85 0.46 0.2 0.05 0.06	< 0.01 0.01 0.02 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/8)
France (N), 2002 (Batavia Destinie) Iceberg	0.25+ 0.25	0.04+ 0.03	1+ 1	-0 0 3 7 10 14 17 21	0.02 1.3 0.6 0.21 0.11 0.03 0.01 < 0.01	0.15 0.36 0.64 0.71 0.62 0.28 0.21 0.21	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/4)
France (N), 2002 (Noemi)	0.25+ 0.25	0.05+ 0.04	1+ 1	-0 0 3 7 10 14 17 21	0.05 3.2 0.86 0.45 0.14 0.06 0.05 0.04	0.28 0.61 1.1 1.3 0.79 0.67 0.69 0.58	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/2)
France (N), 2002 (Batavia) Iceberg	0.38	0.04	1 2	-0 0 3 7 10 14 17 21	0.04 5.0 1.7 0.96 0.4 0.16 0.04 0.02	0.09 0.2 0.57 (c=0.02) 0.53 (c=0.02) 0.64 (c=0.02) 0.64 (c=0.01) 0.25 (c=0.01) 0.12	< 0.01 < 0.01 0.01 0.02 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/1)
France (N), 2002 (Batavia Angie) Iceberg	0.38	0.04	1 2	-0 0 3 7 10 14 17 21	0.18 6.1 1.2 0.31 0.06 0.02 0.02 0.02	0.49 0.91 1.0 0.88 0.26 0.25 0.22 0.25	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/3)
UK, 2002 (Emerald) Butterhead	0.38	0.07	1 2	-0 0 3 7 10 14 17 20	0.13 19 2.0 0.89 0.14 0.06 0.03 0.03	0.21 0.28 0.83 0.84 0.4 0.12 0.08 0.04	< 0.01 < 0.01 0.02 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/8)
Italy, 1998 (Manita) ”Round-headed”	0.38	0.08	1 2	-0 0 3 7 10 14	6.6 (c=0.02) 16.3 12.7 10.1 6.1 6.5	1.6 1.7 1.8 2.8 1.7 2.6	0.03 0.04 0.03 0.04 0.02 0.03	PP62/0323 (IT30-98-E363)
Italy, 1998 (Flandria) ”Round-headed”	0.38	0.09	1 2	-0 0 3 7 10 14	< 0.01 1.5 0.17 0.02 < 0.01 < 0.01	0.01 0.33 0.64 0.29 0.08 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0323 (IT30-98-E362) 5 min water dip at harvest

LETTUCE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
France (N), 2002 (Batavia Destinie) Iceberg	0.38+ 0.38	0.06+ 0.04	1+ 1	-0 0 3 7 10 14 17 21	0.13 6.1 0.84 0.19 0.16 0.21 0.05 0.05	0.67 1.4 1.8 0.6 0.89 1.3 0.46 0.62	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/4)
France (N), 2002 (Noemi)	0.38+ 0.38	0.08+ 0.05	1+ 1	-0 0 3 7 10 14 17 21	0.02 4.4 1.4 0.97 0.68 0.39 0.2 0.15	0.18 0.71 0.91 1.8 2.2 1.8 1.7 1.7	< 0.01 < 0.01 < 0.01 0.01 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/2)
Spain, 1998 (Elisabeth)	0.41	0.05	1 2	-0 0 3 7 10 14	0.47 10.5 1.5 0.38 0.22 0.17	0.91 1.4 0.53 0.45 1.4 1.1	0.02 0.03 0.04 0.02 0.01 0.01	PP62/0323 (ES10-98-SE003)
Spain, 1998 (Elisabeth)	0.51	0.05	1 2	-0 0 3 7 10 14	1.6 8.5 1.9 1.7 0.74 (c=< 0.01)	1.4 4.4 2.8 2.6 1.8	0.04 0.13 0.05 0.04 0.02	PP62/0323 (ES10-98-SE103) Day0 & 1st control samples excluded – possibly cross-labelled
Spain, 2001 (Candela) Butterhead	0.36+ 0.49	0.05+ 0.05	1+ 1	-0 0 3 7 10 14 17	0.11 3.6 0.45 0.17 0.06 < 0.01 < 0.01	0.41 0.31 0.24 0.32 0.30 0.06 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1203 (AF/5977/SY/1)
Spain, 2001 (Bombita)	0.37+ 0.38	0.05+ 0.05	1+ 1	-0 0 3 7 10 14 17	0.14 4.0 1.7 0.77 0.55 0.21 0.22	0.69 0.84 1.3 1.3 1.2 0.83 0.93	< 0.01 < 0.01 0.02 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1203 (AF/5977/SY/2)
Italy, 2001 (Aldina)	0.39+ 0.43	0.05+ 0.05	1+ 1	-0 0 3 7 10 14 17 21	< 0.01 7.9 0.85 0.05 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.35 0.97 0.25 0.04 0.03 0.01 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1203 (AF/5977/SY/3)
Italy, 2001 (Planty)	0.37+ 0.39	0.05+ 0.05	1+ 1	-0 0 3 7 10 14 17 21	0.03 9.7 2.4 0.49 0.17 0.06 0.07 0.02	0.1 0.77 1.5 0.8 0.37 0.14 0.16 0.05	< 0.01 0.01 0.02 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1203 (AF/5977/SY/4)

LETTUCE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
UK, 2002 (Loveley)	0.15	0.03	1 2	-0 0 3 7 10 14	0.56 6.2 (c=0.03) 2.9 (c=0.04) 2.0 (c=0.02) 1.3 (c=0.01) 1.1	0.47 0.62 (c=0.1) 0.6 (c=0.08) 0.75 (c=0.06) 0.74 (c=0.05) 0.7 (c=0.03)	< 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/5) pre-trial spray contamination of control plots
UK, 2002 (Wynona) Butterhead	0.15	0.03	1 2	-0 0 3 7 10 14 17 20	0.19 3.2 (c=0.13) 0.33 (c=0.03) 0.12 (c=0.02) 0.06 (c=0.01) 0.03 (c=0.01) 0.02 0.01	0.44 0.55 (c=0.38) 0.41 (c=0.11) 0.33 (c=0.07) 0.22 (c=0.08) 0.15 (c=0.03) 0.09 (c=0.02) 0.1 (c=0.01)	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/6) pre-trial spray contamination of control plots
UK, 2002 (Hilary)	0.15	0.03	1 2	-0 0 3 7 10 14 17 21	0.02 4.9 (c=0.01) 0.52 (c=0.01) 0.05 (c=0.01) 0.02 < 0.01 < 0.01 < 0.01	0.46 1.6 (c=0.2) 1.6 (c=0.11) 0.46 (c=0.04) 0.27 (c=0.02) 0.08 0.07 0.02	< 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/9) pre-trial spray contamination of control plots
UK, 2002 (Loveley)	0.25	0.05	1 2	-0 0 3 7 10 14	1.7 5.1 (c=0.03) 2.9 (c=0.04) 2.7 (c=0.02) 2.8 (c=0.01) 0.8	0.69 0.72 (c=0.1) 0.84 (c=0.08) 0.87 (c=0.06) 0.83 (c=0.05) 0.5 (c=0.03)	< 0.01 < 0.01 0.01 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/5) pre-trial spray contamination of control plots
UK, 2002 (Wynona) Butterhead	0.25	0.05	1 2	-0 0 3 7 10 14 17 20	0.31 7.3 (c=0.13) 0.89 (c=0.03) 0.3 (c=0.02) 0.18 (c=0.01) 0.09 (c=0.01) 0.04 0.02	0.9 0.69 (c=0.38) 0.72 (c=0.11) 0.6 (c=0.07) 0.39 (c=0.08) 0.45 (c=0.03) 0.25 (c=0.02) 0.12 (c=0.01)	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/6) pre-trial spray contamination of control plots
UK, 2002 (Hilary)	0.25	0.05	1 2	-0 0 3 7 10 14 17 21	0.04 6.5 (c=0.01) 1.6 (c=0.01) 0.14 (c=0.01) 0.01 0.02 0.06 < 0.01	0.58 1.7 (c=0.2) 2.9 (c=0.11) 0.69 (c=0.04) 0.09 (c=0.02) 0.19 0.52 0.05	< 0.01 0.02 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/9) pre-trial spray contamination of control plots
UK, 2002 (Loveley)	0.38+ 0.38	0.08+ 0.07	1+ 1	-0 0 3 7 10 14	2.1 6.8 (c=0.03) 5.8 (c=0.04) 4.0 (c=0.02) 2.2 (c=0.01) 1.2	0.74 0.85 (c=0.1) 1.0 (c=0.08) 1.6 (c=0.06) 1.1 (c=0.05) 0.76 (c=0.03)	< 0.01 0.01 0.01 0.02 0.01 < 0.01	PP62/1292 (AF/5976/SY/5) pre-trial spray contamination of control plots
UK, 2002 (Wynona) Butterhead	0.38+ 0.38	0.08+ 0.07	1+ 1	-0 0 3 7 10 14 17 20	0.47 17 (c=0.13) 1.5 (c=0.03) 0.65 (c=0.02) 0.39 (c=0.01) 0.15 (c=0.01) 0.05 0.03	0.85 1.4 (c=0.38) 1.1 (c=0.11) 1.0 (c=0.07) 1.2 (c=0.08) 0.58 (c=0.03) 0.26 (c=0.02) 0.27 (c=0.01)	< 0.01 0.02 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1292 (AF/5976/SY/6) pre-trial spray contamination of control plots

LETTUCE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
UK, 2002	0.38+	0.07+	1+	-0	0.06	1.0	< 0.01	PP62/1292
(Hilary)	0.38	0.08	1	0	13 (c=0.01)	2.3 (c=0.2)	0.02	(AF/5976/SY/9)
				3	1.8 (c=0.01)	4.2 (c=0.11)	0.01	
				7	0.2 (c=0.01)	1.2 (c=0.04)	< 0.01	pre-trial spray
				10	0.08	0.78 (c=0.02)	< 0.01	contamination of
				14	0.02	0.32	< 0.01	control plots
				17	0.02	0.22	< 0.01	
				21	0.01	0.08	< 0.01	

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Beans

The Meeting received information on trials conducted on both common beans (i.e., with pods) and broad beans (without pods) in France, Germany, Greece, the Netherlands, Spain and the UK. In these trials, two foliar applications of pirimicarb (50% WG formulations) were made at 7−12 day intervals to unreplicated 30−120 square metre plots, either as broadcast or band sprays using small plot boom or knapsack (hand lance or mini boom) sprayers to obtain full foliar coverage. A minimum of 0.8-1.0 kg pods (with seeds) were taken for analysis using Methods RAM 265/02 (GC-NPD detection), RAM 265/03 (GC-NPD or HPLC-MS-MS detection) or RAM 265/04 (HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 79−102% at fortification levels of 0.01−5.0 mg/kg.

Table 83. Residues in beans (with pods) from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France, Germany, Greece, the Netherlands and Spain.

COMMON BEANS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
Netherlands, 1997	0.25	0.05	2	0	0.05	0.02	< 0.01	PP62/0364
(Montano)				3	0.02	0.02	< 0.01	(NL10-97-
				7	< 0.01	< 0.01	< 0.01	E303)
Snap bean				10	< 0.01	< 0.01	< 0.01	
				14	< 0.01	< 0.01	< 0.01	
Germany, 1997	0.25	0.06	1	-0	0.41	0.07	< 0.01	PP62/0360
(Magnum)			2	1	0.37	0.11	< 0.01	(RS-9723-H1)
				2	0.32	0.1	< 0.01	
French bean				3	0.25	0.12	< 0.01	
				6	0.21	0.11	< 0.01	
France, 2001	0.25	0.07	1	-0	0.01	< 0.01	< 0.01	PP62/1168
(Allegria)			2	0	0.21	0.02	< 0.01	(FR23-01-S774)
				3	0.26	0.05	< 0.01	
French bean				7	0.16	0.05	< 0.01	
Germany, 1997	0.25	0.08	2	0	0.13	0.04	< 0.01	PP62/0360
(Scuba)				1	0.07	0.05	< 0.01	(RS-9723-G1)
				2	0.05	0.05	< 0.01	
French bean				3	0.04	0.06	< 0.01	
				6	0.02	0.04	< 0.01	
France (N), 2001	0.25	0.08	1	-0	0.05	0.02	< 0.01	PP62/1168
(Skipper)			2	0	0.4	0.03	< 0.01	(FR81-01-S775)
				3	0.23	0.03	< 0.01	
French bean				7	0.16	0.03	< 0.01	
Netherlands, 1997	0.25+	0.05+	1+	0	0.31	0.12	< 0.01	PP62/0364
(Odessa)	0.22	0.05	1	3	0.24	0.16	< 0.01	(NL10-97-
				7	0.09	0.12	< 0.01	E203)
Snap bean				10	0.03	0.1	< 0.01	
				13	0.02	0.07	< 0.01	

COMMON BEANS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
Germany, 1996 (Maradona) French bean	0.38	0.08	2	0 3 7	0.57 0.31 0.1	0.06 0.13 0.08	< 0.01 < 0.01 < 0.01	PP62/0374 (RS-9615-K3)
Netherlands, 1996 (Montano) French bean	0.38	0.08	2	0 3 7	0.27 0.09 0.03	0.08 0.06 0.06	< 0.01 < 0.01 < 0.01	PP62/0376 (NL10-96-E301)
Netherlands, 1997 (Odessa) Snap bean	0.38	0.08	2	0 3 7 10 13	0..55 0.48 0.21 0.08 0.05	0.19 0.27 0.22 0.17 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0364 (NL10-97-E203)
Netherlands, 1997 (Montano) Snap bean	0.38	0.08	2	0 3 7 10 14	0.28 0.36 0.21 0.13 0.09	0.03 0.03 0.02 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0364 (NL10-97-E303)
Germany, 1997 (Magnum) French bean	0.38	0.09	2	0 1 4 7 10	0.28 0.36 0.21 0.13 0.09	0.09 0.11 0.16 0.11 0.11	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0360 (RS-9723-H1)
France (N), 2001 (Allegria) French bean	0.38	0.11	1 2	-0 0 3 7	0.02 0.46 0.5 0.31	0.01 0.05 0.11 0.1	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1168 (FR23-01-S774)
Germany, 1997 (Scuba) French bean	0.38	0.13	2	0 1 4 7 10	0.13 0.18 0.12 0.07 0.03	0.04 0.09 0.16 0.16 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0360 (RS-9723-G1)
France (S), 1997 (Landros)	0.38	0.13	2	0 3 7 9 14	0.41 0.38 0.28 0.2 0.1	0.06 0.1 0.09 0.08 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0362 (S902.97)
France (S), 1997 (Landros)	0.38	0.13	2	0 3 7 9 14	0.27 0.25 0.16 0.1 0.08	0.05 0.07 0.06 0.05 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0362 (S952.97)
France (N), 2001 (Skipper) French bean	0.38	0.13	1 2	-0 0 3 7	0.09 0.62 0.43 0.22	0.03 0.04 0.06 0.05	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1168 (FR81-01-S775)
Spain, 1997 (Boby) French bean	0.48	0.05	2	0 3 7 10 14	0.38 0.4 0.12 0.03 < 0.01	0.04 0.08 0.04 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0362 (ES10-97-SE114)
Spain, 1997 (Boby) French bean	0.49	0.05	2	0 3 7 10 14	0.25 0.22 0.1 0.07 < 0.01	0.03 0.05 0.04 0.04 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0362 (ES10-97-SE014)
Greece, 1996 (not specified) Black beans	0.5	0.05	2	0 3 7	0.35 0.09 0.06	0.08 0.06 0.04	< 0.01 < 0.01 < 0.01	PP62/0377 (GR-96-E201)

COMMON BEANS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
Spain, 1998 (Superba)	0.6	0.05	2	3 7	0.36 0.21	0.18 0.11	< 0.01 < 0.01	PP62/0366 (AF/4168/CL/1)
Spain, 1998 (Roma II Planta)	0.6	0.05	2	3 7	0.39 0.22	0.19 0.15	< 0.01 < 0.01	PP62/0366 (AF/4168/CL/2)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Table 84. Residues in broad beans (without pods) from foliar applications of pirimicarb (50% WG formulation) in supervised trials from the UK.

BROAD BEANS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
UK, 2001 (Drednort)	0.25	0.06	1 2	-0 0 3 7	0.02 0.07 0.04 0.02	0.01 0.01 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1178 (AF/5968/SY3)
UK, 2001 (Danko)	0.25	0.06	1 2	-0 0 3 7	0.05 0.13 0.02 0.02	0.02 0.02 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1178 (AF/5968/SY/4)
UK, 2001 (Drednort)	0.38	0.09	1 2	-0 0 3 7	0.01 0.16 0.06 0.02	< 0.01 0.02 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1178 (AF/5968/SY3)
UK, 2001 (Danko)	0.38	0.09	1 2	-0 0 3 7	0.06 0.23 0.04 0.02	0.02 0.04 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1178 (AF/5968/SY/4)
UK, 2002 (Wilkem major)	0.25	0.05	1 2	-0 0 3 7	< 0.01 0.02 0.03 0.02	0.01 0.01 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1299 & PP62/1346 (AF/6542/SY/1)
UK, 2002 (Wilkem major)	0.38	0.08	1 2	-0 0 3 7	0.01 0.04 < 0.01 0.02	0.02 0.03 < 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1299 & PP62/1346 (AF/6542/SY/1)
UK, 2002 (Listra)	0.25	0.05	1 2	-0 0 3 7	< 0.01 < 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1299 & PP62/1346 (AF/6542/SY/2)
UK, 2002 (Listra)	0.38	0.08	1 2	-0 0 3 7	< 0.01 0.01 0.02 0.01	< 0.01 < 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1299 & PP62/1346 (AF/6542/SY/2)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Peas

The Meeting received information on trials conducted in France, Italy, Germany, Netherlands and the UK on peas, both shelled and with pods. In these trials, 1–2 foliar applications of pirimicarb (50% WG formulations) were made at 7–12 day intervals to unreplicated 35–180 square metre plots, as broadcast sprays using small plot or knapsack mini boom sprayers to obtain full foliar coverage. A minimum of 0.8–1.0 kg peas were taken for analysis using either Methods RAM 265/02 (using GC-NPD detection to measure residues of pirimicarb and the combined residues of desmethyl pirimicarb (R34836) and desmethylformamido pirimicarb (R34885) or RAM 265/04 (with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 81–98% at fortification levels of 0.01–10.0 mg/kg.

Table 85. Residues in peas (with pods) from foliar applications of pirimicarb (50% WG formulation) in supervised trials in Germany.

PEAS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Germany, 1996 (Resal) Green peas	0.38	0.08	2	0 3 7	1.1 0.02 ≤ 0.01	0.05 0.01 ≤ 0.01	< 0.01 < 0.01 < 0.01	PP62/0374 (RS-9615-K3)
Netherlands, 1996 (Koka) Sugar peas	0.38	0.08	2	0 3 7	0.08 0.01 ≤ 0.01	0.05 0.02 ≤ 0.01	< 0.01 < 0.01 < 0.01	PP62/0376 (NL10-96-E302)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Table 86. Residues in fresh peas (without pods) from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France and the UK.

PEAS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[3]	R238177	
UK, 2001 (Waverex) Vining peas	0.38	0.09	1 2	-0 0 3 7	< 0.01 0.05 < 0.01[1] ≤ 0.01[1]	< 0.01 0.01 < 0.01[1] ≤ 0.01[1]	< 0.01 < 0.01 < 0.01[1] < 0.01[1]	PP62/1180 (AF/5971/SY/1)
UK, 2001 (Gallant) Vining peas	0.38	0.09	1 2	-0 0 3 7	< 0.01 0.07 0.01[1] ≤ 0.01[1]	< 0.01 0.01 0.01[1] ≤ 0.01[1]	< 0.01 < 0.01 < 0.01[1] < 0.01[1]	PP62/1180 (AF/5971/SY/2)
France (S), 2001 (Arabelle) Vining peas	0.38	0.09	1 2	-0 0 3 7	< 0.01 0.02 < 0.01[1] ≤ 0.01[1]	< 0.01 < 0.01 < 0.01[1] ≤ 0.01[1]	< 0.01 < 0.01 < 0.01[1] < 0.01[1]	PP62/1174 (AF/5970/SY/1)
France (S), 2001 (Ast) Vining peas	0.38	0.09	1 2	-0 0 3 7	< 0.01 0.02 < 0.01[1] ≤ 0.01[1]	< 0.01 < 0.01 < 0.01[1] ≤ 0.01[1]	< 0.01 < 0.01 < 0.01[1] < 0.01[1]	PP62/1174 (AF/5970/SY/2)
France (N), 1992 (Marlene)	0.38	0.13	1	7	≤ 0.01	≤ 0.01		PP62/0373 (S 322.92)
France (N), 1992 (Solara)	0.38	0.13	1	7	≤ 0.01	≤ 0.01		PP62/0373 (S 341.92)
France (N), 1992 (Carla)	0.38	0.13	1	7	≤ 0.01	≤ 0.01		PP62/0373 (S 201.92)
France (N), 1992 (Santon)	0.38	0.13	1	7	≤ 0.01	≤ 0.01		PP62/0373 (S 202.92)
Italy, 2001 (Resal)	0.5+ 0.5	0.1+ 0.07	1+ 1	-0 0 3 7 14	< 0.01 0.02 < 0.01 ≤ 0.01 < 0.01	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1164 (AF5716/SY/8)
Italy, 2001 (Lambado)	0.5+ 0.5	0.1+ 0.08	1+ 1	-0 0 3 7 14	< 0.01 0.02 < 0.01 ≤ 0.01 < 0.01	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1164 (AF5716/SY/6)

[1]) mean residues in pods or seeds after hand separation and mechanical separation

[2]) vines after removal of peas and pods

[3]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Beans (dry)

The Meeting received information on trials conducted in France beans (dry), where 2 foliar applications of pirimicarb (50% WG formulations) were made at 10–11 day intervals to unreplicated 120 square metre plots, as broadcast sprays using small plot mini boom sprayers to obtain full foliar coverage. A minimum of 1.0 kg dry beans were taken for analysis using Method RAM 265/04 with HPLC-MS-MS detection to measure residues of pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 93–94% at fortification levels of 0.01–15.0 mg/kg.

Table 87. Residues in dry beans (without pods) from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France.

BEANS (DRY) Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (S), 2001 (Linex)	0.5	0.06	1 2	-0 0 3 7	< 0.01 0.03 <u>0.04</u> 0.04	0.02 < 0.01 <u>0.04</u> 0.03	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1188 (AF5969/SY/1)
France (S), 2001 (Linex)	0.5	0.06	1 2	-0 0 3 7	0.06 0.15 <u>0.09</u> 0.07	0.04 0.04 <u>0.05</u> 0.03	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1188 (AF5969/SY/2)
France (S), 2001 (Linex)	0.5	0.06	1 2	-0 0 3 7	0.01 0.09 <u>0.03</u> 0.03	0.02 0.04 <u>0.03</u> 0.03	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1188 (AF5969/SY/3)
France (S), 2001 (Linex)	0.5+ 0.5	0.1+ 0.08	1+ 1	-0 0 3 7	0.06 0.12 0.13 0.06	0.09 0.11 0.12 0.06	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1188 (AF5969/SY/4)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Peas (dry)

The Meeting received information on trials conducted in France and Spain on peas (dry), where 2 foliar applications of pirimicarb (50% WG formulations) were made at 7–12 day intervals to unreplicated 60-240 square metre plots, as broadcast sprays using small plot boom sprayers to obtain full foliar coverage. A minimum of 1.0 kg dry peas (hand or machine threshed) were taken for analysis using Methods RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The limit of quantification was 0.01 mg/kg and mean recovery rates were 77–102% at fortification levels of 0.01–20.0 mg/kg.

Table 88. Residues in dry peas (without pods) from foliar applications of pirimicarb (50% WG formulation) to peas in supervised trials in France and Spain.

PEAS (DRY) Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
France (S), 2001 (Solara)	0.5	0.06	1 2	-0 0 3 7 14	< 0.01 0.05 <u>0.08</u> 0.05 0.03	< 0.01 < 0.01 <u>0.02</u> 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1164 (AF5716/SY/2)
Spain, 2001 (Calibra)	0.5	0.06	1 2	-0 0 3 7 14	< 0.01 0.02 <u>≤ 0.01</u> < 0.01 < 0.01	< 0.01 < 0.01 <u>≤ 0.01</u> < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1164 (AF5716/SY/3)

PEAS (DRY) Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
Spain, 2001 (Gracia)	0.5	0.06	1 2	-0 0 3 7 14	< 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1164 (AF5716/SY/4)
France (S), 2001 (Victor)	0.5+ 0.5	0.08+ 0.1	1+ 1	-0 0 3 7 14	0.06 0.22 0.1 0.03 0.05	< 0.01 0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1164 (AF5716/SY/7)
Spain, 1999 (Ballet) Field peas	0.53	0.05	2	3 7	0.05 0.05	0.02 0.02	< 0.01 < 0.01	PP62/0368 (AF/4127/CL/2)
Spain, 1999 (Ballet)	0.56	0.05	2	3 7	0.12 0.08	0.03 0.03	< 0.01 < 0.01	PP62/0368 (AF/4127/CL/1)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Carrots

The Meeting received information on trials conducted in France, Italy and Spain on carrots, where two foliar applications of pirimicarb (50% WG formulations) were made at 6–11 day intervals to unreplicated 30 square metre plots, as broadcast sprays using knapsack mini boom sprayers to obtain full foliar coverage. A minimum of 1.0 kg (or 12 units) were taken for analysis using Methods RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 76–90% at fortification levels of 0.01–1.0 mg/kg.

Table 89. Residues in carrots from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France, Italy and Spain.

CARROTS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Spain, 1998 (Nantesa)	0.35	0.062	1 2	-0 0 3 7 10 14	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0279 (ES10-98- SE001)
Spain, 1998 (Nantesa Coral)	0.37	0.075	1 2	-0 0 3 8 10 13	< 0.01 0.05 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0279 (ES10-98- SE101)
Italy, 1998 (Turbo)	0.38	0.063	1 2	-0 0 3 7 10 14	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0279 (IT40-98-E360)
Italy, 1998 (Efeso Hybrid)	0.38	0.075	1 2	-0 0 3 7 9 14	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0279 (IT50-98-E361)

CARROTS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (N), 2001 (Karotan)	0.38	0.09	1 2	-0 0 3 7	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1176 (FR41-01-S781)
France (N), 2001 (Maxima)	0.38	0.11	1 2	-0 0 3 6	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1176 (FR22-01-S779)
France (N), 2001 (Nerac)	0.38	0.11	1 2	-0 0 3 7	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1176 (FR23-01-S780)
France (N), 2001 (Puma)	0.38	0.13	1 2	-0 0 3 7	< 0.01 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	PP62/1176 (FR81-01-S782)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Sugar beet

The Meeting received information on sugar beet trials conducted in France, Italy, Spain and the UK, where 2–4 foliar applications of pirimicarb (50% WG formulations) were made at 7–14 day intervals to unreplicated 30–120 square metre plots, as broadcast sprays using small plot or knapsack mini boom sprayers to obtain full foliar coverage. A minimum of 1.0 kg roots (or 12 plants) were sampled, brushed and trimmed before analysis using either Methods RAM 15/02 (with GC-MSD detection to measure residues of pirimicarb and the combined residues of desmethyl pirimicarb (R34836) and desmethylformamido pirimicarb (R34885) or Methods RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 82–92% at fortification levels of 0.01–12.0 mg/kg.

Table 90. Residues in sugar beet (roots) from foliar applications of pirimicarb (50% WG formulation) to sugar beet in supervised trials in France, Italy, Spain and the UK.

SUGAR BEET (ROOTS) Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
UK, 1991 (Amethyst)	0.28	0.07	2	7	0.01	≤ 0.01		PP62/0268 (GB12-91-S061)
UK, 1991 (Amethyst)	0.28	0.07	4	7	0.01	≤ 0.01		PP62/0268 (GB12-91-S061)
UK, 1991 (Hilme)	0.28	0.07	2	7	≤ 0.01	≤ 0.01		PP62/0268 (GB12-91-S062)
UK, 1991 (Hilme)	0.28	0.07	4	7	≤ 0.01	≤ 0.01		PP62/0268 (GB12-91-S062)
UK, 1991 (Celt)	0.28	0.07	2	7	≤ 0.01	≤ 0.01		PP62/0268 (GB15-91-S062)
UK, 1991 (Celt)	0.28	0.07	4	7	0.02	≤ 0.01		PP62/0268 (GB15-91-S062)
UK, 1991 (Rex)	0.28	0.07	2	7	≤ 0.01	≤ 0.01		PP62/0268 (GB15-91-S063)
UK, 1991 (Rex)	0.28	0.07	4	7	≤ 0.01	≤ 0.01		PP62/0268 (GB15-91-S063)
UK, 1991 (Amethyst)	0.28	0.07	2	7	≤ 0.01	≤ 0.01		PP62/0268 (GB17-91-S061)
UK, 1991 (Amethyst)	0.28	0.07	4	7	≤ 0.01	≤ 0.01		PP62/0268 (GB17-91-S061)

SUGAR BEET (ROOTS) Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
UK, 1991 (Hilma)	0.28	0.07	2	7	≤ 0.01	≤ 0.01		PP62/0268 (GB17-91-S062)
UK, 1991 (Hilma)	0.28	0.07	4	7	≤ 0.01	≤ 0.01		PP62/0268 (GB17-91-S062)
UK, 1992 (Celt)	0.28	0.07	2	7	≤ 0.01	≤ 0.01		PP62/0269 (GB12-92-S071)
UK, 1992 (Saxon)	0.28	0.07	2	7	≤ 0.01	≤ 0.01		PP62/0269 (GB12-92-S072)
UK, 1992 (Regina)	0.28	0.07	2	7	≤ 0.01	≤ 0.01		PP62/0269 (GB12-92-S073)
UK, 1992 (Giselle)	0.28	0.07	2	7	≤ 0.01	≤ 0.01		PP62/0269 (GB12-92-S074)
UK, 1992 (Celt)	0.28	0.07	4	7	≤ 0.01	≤ 0.01		PP62/0269 (GB12-92-S071)
UK, 1992 (Saxon)	0.28	0.07	4	7	≤ 0.01	≤ 0.01		PP62/0269 (GB12-92-S072)
UK, 1992 (Regina)	0.28	0.07	4	7	≤ 0.01	≤ 0.01		PP62/0269 (GB12-92-S073)
UK, 1992 (Giselle)	0.28	0.07	4	7	≤ 0.01	≤ 0.01		PP62/0269 (GB12-92-S074)
Spain, 1998 (Korif)	0.36	0.075	1 2	-0 0 3 7 10 14	0.04 0.13 0.05 0.03 < 0.01 0.02	0.01 0.01 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0288 (ES10-98-SE102)
Spain, 1998 (Oryx)	0.37	0.075	1 2	-0 0 3 7 10 14	0.02 0.06 0.04 0.03 0.02 0.01	< 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0288 (ES10-98-SE002)
Italy, 2001 (Dorotea)	0.38	0.061	1 2	-0 0 3 8 10 14	< 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1229 (AF/5972/SY/3)
Italy, 2001 (Eko)	0.38	0.062	1 2	-0 0 3 7 10 14	< 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 ≤ 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1229 (AF/5972/SY/4)
France (S), 2001 (Sheriff)	0.38	0.073	1 2	-0 0 3 7 10 14	< 0.01 0.02 0.03 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1229 (AF/5972/SY/2)
Italy, 1998 (Asso)	0.38	0.075	1 2	-0 0 3 8 10 14	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0288 (IT30-98-E366)

SUGAR BEET (ROOTS) Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Italy, 1998 (Arma)	0.38	0.075	1	-0	< 0.01	< 0.01	< 0.01	PP62/0288 (IT20-98-E367)
			2	0	< 0.01	< 0.01	< 0.01	
				3	< 0.01	< 0.01	< 0.01	
				7	< 0.01	< 0.01	< 0.01	
				10	< 0.01	< 0.01	< 0.01	
				14	< 0.01	< 0.01	< 0.01	
France (S), 2001 (Sheriff)	0.38	0.079	1	-0	< 0.01	< 0.01	< 0.01	PP62/1229 (AF/5972/SY/1)
			2	0	0.06	< 0.01	< 0.01	
				3	0.04	0.01	< 0.01	
				7	0.03	0.02	< 0.01	
				10	< 0.01	< 0.01	< 0.01	
				14	< 0.01	< 0.01	< 0.01	

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Potato

The Meeting received information on potato trials conducted in France, Germany, Spain and the UK, where 2–5 foliar applications of pirimicarb (50% WG formulations) were made at 6–14 day intervals to unreplicated 37–50 square metre plots, as broadcast sprays using knapsack mini boom or hand lance sprayers to obtain full foliar coverage. A minimum of 2 kg or 24 tubers were taken for analysis using either Method RAM 15 to measuring residues of pirimicarb and the combined residues of desmethyl pirimicarb (R34836) and desmethylformamido pirimicarb (R34885) or Methods RAM 265/01 (with GC-NPD detection), RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 77–102% at fortification levels of 0.01–20.0 mg/kg.

Table 91. Residues in potatoes from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France, Germany, Spain and the UK.

POTATOES Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Germany, 1976 (Not stated)	0.23+ 0.2+ 0.18	0.038+ 0.033+ 0.03	1+ 1+ 2	0	< 0.01	< 0.01		PP62/1233 (7631-R-V-3)
				1	< 0.01	< 0.01		
				3	< 0.01	< 0.01		
				7	≤ 0.01	≤ 0.01		
				14	< 0.01	< 0.01		
				20	< 0.01	< 0.01		
Germany, 1976 (Not stated)	0.23+ 0.2+ 0.18	0.038+ 0.033+ 0.03	1+ 1+ 3	0	< 0.01	< 0.01		PP62/1233 (7631-R-V-1)
				3	< 0.01	< 0.01		
				7	≤ 0.01	≤ 0.01		
				14	< 0.01	< 0.01		
				49	< 0.01	< 0.01		
Germany, 1976 (Not stated)	0.23+ 0.2+ 0.18	0.038+ 0.033+ 0.03	1+ 1+ 3	0	< 0.01	< 0.01		PP62/1233 (7631-R-V-2)
				1	< 0.01	< 0.01		
				3	< 0.01	< 0.01		
				8	≤ 0.01	≤ 0.01		
				15	< 0.01	< 0.01		
				40	< 0.01	< 0.01		
Spain, 1998 (Kennebec)	0.24	0.063	3	6	< 0.01	< 0.01	< 0.01	PP62/0285 (ES10-98-SE005)
UK, 2000 (Romano)	0.25	0.063	1	-0	< 0.01	< 0.01	< 0.01	PP62/1080 (GB01-00-S094)
			2	0	< 0.01	< 0.01	< 0.01	
				3	< 0.01	< 0.01	< 0.01	
				7	≤ 0.01	≤ 0.01	< 0.01	
				14	< 0.01	< 0.01	< 0.01	
				21	< 0.01	< 0.01	< 0.01	

POTATOES Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
UK, 2000 (Wilja)	0.25	0.083	1 2	-0 0 3 7 14 20	< 0.01 < 0.01 < 0.01 <u>≤ 0.01</u> < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 <u>≤ 0.01</u> < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1080 (GB01-00-S093)
France (S), 2000 (Adora)	0.25	0.083	1 2	-0 0 3 8 14 21	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1078 (FR52-01-S751)
France (S), 2000 (Bintje)	0.25	0.13	1 2	-0 0 3 4 7 13 21	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1078 (FR17-01-S754)
Spain, 1998 (Jaeria)	0.27	0.05	3	7	< 0.01	< 0.01	< 0.01	PP62/0285 (ES10-98-SE105)
Spain, 1995 (Marfond)	0.38	0.064	3	3 7	<u>≤ 0.01</u> < 0.01	<u>≤ 0.01</u> < 0.01	< 0.01 < 0.01	PP62/1001 (ES10-98-SE105)
Spain, 1995 (Jaerla)	0.33+ 0.4	0.064+ 0.064	2+ 1	3 7	<u>≤ 0.01</u> < 0.01	<u>≤ 0.01</u> < 0.01	< 0.01 < 0.01	PP62/1001 (ES10-95-SE001)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Artichoke, Globe

The Meeting received information on globe artichoke trials conducted in France, Italy and Spain, where 2 foliar applications of pirimicarb (50% WG formulations) were made at 7–9 day intervals to unreplicated 30–225 square metre plots, as broadcast sprays using knapsack or small plot mini boom or hand lance sprayers to obtain full foliar coverage. A minimum of 2 kg or 12 units were taken for analysis using Method RAM 265/03 with HPLC-MS-MS detection to measure residues of pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 77–103% at fortification levels of 0.01–5.0 mg/kg.

Table 92. Residues in globe artichokes from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France, Italy and Spain.

ARTICHOKE, GLOBE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (N), 1997 (Camus)	0.36+ 0.38	0.09+ 0.08	1+ 1	-0 0 3 7 10 14	0.11 0.61 0.25 <u>0.16</u> 0.1 0.03	0.02 0.04 0.04 <u>0.03</u> 0.02 0.01	< 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0275 (97 I CL SA P30)
France (N), 1997 (Camus)	0.38	0.08	1 2	-0 0 3 7 10 14	0.09 0.6 0.41 <u>0.18</u> 0.13 0.08	0.02 0.06 0.07 <u>0.04</u> 0.03 0.02	< 0.01 < 0.01 0.01 < 0.01 < 0.01 < 0.01	PP62/0287 (AF/4167/CL/1)

ARTICHOKE, GLOBE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (N), 1997 (Petit Violet)	0.38	0.08	1 2	-0 0 3 7 10 14	0.05 0.61 0.28 0.07 0.06 0.05	0.01 0.05 0.06 0.02 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.02	PP62/0287 (AF/4167/CL/2)
France (N), 1999 (Castel)	0.38	0.08	2	7	0.23	0.04	< 0.01	PP62/0464 (AF/4754/ZE/1)
France (N), 1999 (Camus)	0.38	0.08	2	7	0.46	0.09	< 0.01	PP62/0464 (AF/4754/ZE/2)
France (N), 1997 (Camus)	0.38	0.09	1 2	-0 0 3 7 10 14	0.25 1.0 0.58 0.3 0.15 0.03	0.03 0.07 0.06 0.1 0.04 0.02	< 0.01 0.01 < 0.01 0.01 < 0.01 < 0.01	PP62/0275 (97 I CL SA P29)
Spain, 1999 (Blanca de Tudela)	0.52	0.05	1 2	-0 0 3 7 10 14	0.06 0.56 0.33 0.14 0.09 0.02	0.03 0.05 0.1 0.06 0.05 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0289 (AF/4323/CL/1)
Spain, 1999 (Blanca de Tudela)	0.54+ 0.57	0.05+ 0.05	1+ 1	-0 0 3 7 10 14	0.07 0.59 0.42 0.17 0.08 0.02	0.03 0.04 0.1 0.06 0.03 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0289 (AF/4323/CL/2)
Spain, 1998 (Blanca de Tudela)	0.75+ 0.79	0.05+ 0.05	1+ 1	0 3 7 10 14	1.4 0.44 0.2 0.11 0.04	0.1 0.07 0.04 0.02 < 0.01	0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0277 (AF/3960/ZE/1)
Italy, 1998 (Moro di Corneto)	0.76	0.05	2	0 3 7 10 14	5.3 2.6 0.92 0.21 0.05	0.2 0.15 0.09 0.02 < 0.01	0.02 0.02 0.01 < 0.01 < 0.01	PP62/0277 (AF/3960/ZE/4)
Spain, 1998 (Blanca de Tudela)	0.9+ 1.2	0.05+ 0.05	1+ 1	0 3 7 10 14	1.4 0.73 0.3 0.15 0.03	0.11 0.11 0.06 0.03 0.01	0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0277 (AF/3960/ZE/2)
Italy, 1998 (Teramo)	1.3+ 1.2	0.05+ 0.05	1+ 1	0 3 7 10 14	3.0 1.9 0.81 0.37 0.24	0.16 0.18 0.11 0.05 0.04	0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0277 (AF/3960/ZE/3)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Asparagus

The Meeting received information on asparagus trials conducted in Germany and Greece, where 2 foliar applications of pirimicarb (50% WG formulations) were made to asparagus ferns (120-190 cm in height) at 7 day interval (Greece) and 23–31 day intervals (Germany) to unreplicated 46–100 square metre plots, as broadcast sprays using knapsack mini boom or hand lance sprayers to obtain full foliar coverage. A minimum of 24 spears (washed in the German trials), were taken for analysis using Method RAM 265/02 with GC-NPD and HPLC-MS-MS detection to measure residues of

pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 77–89% at a fortification level of 0.1 mg/kg.

Table 93. Residues in asparagus from foliar applications of pirimicarb (50% WG formulation) in supervised trials in Germany and Greece.

ASPARAGUS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Germany, 1997 (Schneewittchen) Green asparagus	0.5	0.1	2	257	≤ 0.01	≤ 0.01	≤ 0.01	PP62/0302 (RS-9617-G1) Fern treatment
Germany, 1997 (Lukullus) White asparagus	0.5	0.1	2	266	≤ 0.01	≤ 0.01	≤ 0.01	PP62/0302 (RS-9617-K1) Fern treatment
Greece, 1997 (Larac)	0.54+ 0.57	0.05+ 0.05	1+ 1	195[2]	≤ 0.01	≤ 0.01	≤ 0.01	PP62/0271 (GR-96-E106) Fern treatment
Greece, 1997 (Svetsinger)	0.55+ 0.5	0.05+ 0.05	1+ 1	195[1]	≤ 0.01	≤ 0.01	≤ 0.01	PP62/0271 (GR-96-E107) Fern treatment

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

[2]) samples taken over a 3 day period

Barley

In trials on winter barley in France and the UK, 2 foliar applications of pirimicarb (50% WG formulation) were made at 6–23 day intervals to unreplicated 45–460 square metre plots, using knapsack sprayers and mini-booms or precision boom sprayers to obtain full foliar coverage. Mature grain samples (0.5–1.0 kg, hand or mechanically threshed) were taken for analysis using Methods RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ of the methods was 0.01 mg/kg for all analytes and the mean recovery rates were 75–94% at fortification levels of 0.01–1.0 mg/kg.

Table 94. Residues in barley grain from foliar applications of pirimicarb (50% WG formulation) to barley in supervised trials in France and the UK.

BARLEY Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
UK, 1998 (Hanna) winter barley	0.15	0.08	2	up to BBCH85	≤ 0.01	≤ 0.01	< 0.01	PP62/0459 (AK/4165/CL/1) sampled 21 days after last treatment
UK, 1998 (Intro) winter barley	0.15	0.08	2	up to BBCH85	0.03	0.02	< 0.01	PP62/0459 (AK/4165/CL/2) sampled 20 days after last treatment
France (N), 1998 (Clarine) winter barley	0.15	0.08	2	up to BBCH83	≤ 0.01	< 0.01	< 0.01	PP62/0459 (AK/4165/CL/3) sampled 29 days after last treatment

BARLEY Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
UK, 2000 (Intro) winter barley	0.15	0.08	2	up to BBCH83	≤ 0.01	≤ 0.01	< 0.01	PP62/0942 (GB02-00-S082) sampled 24 days after last treatment
UK, 2000 (Regina) winter barley	0.15	0.08	2	up to BBCH83	≤ 0.01	≤ 0.01	< 0.01	PP62/0942 (GB02-00-S083) sampled 24 days after last treatment
UK, 2000 (Fanfare) winter barley	0.15	0.08	2	up to BBCH85	0.01	≤ 0.01	< 0.01	PP62/0942 (GB02-00-S084) sampled 24 days after last treatment
UK, 2000 (Regina) winter barley	0.15	0.08	2	up to BBCH83	≤ 0.01	< 0.01	< 0.01	PP62/0942 (GB02-00-S085) sampled 24 days after last treatment
France (N), 1998 (Sunrise) winter barley	0.16	0.08	2	up to BBCH 85-87	≤ 0.01	≤ 0.01	< 0.01	PP62/0459 (AK/4165/CL/4) sampled 21 days after last treatment
UK, 2003 (Pearl) winter barley	0.38	0.13	2	21	0.03	0.02	< 0.01	PP62/1391 (AF/7360/SY/1)
UK, 2003 (Pearl) winter barley	0.75	0.25	2	21	0.14	0.05	< 0.01 (2)	PP62/1391 (AF/7360/SY/1) Processing study average residue single plot??

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Maize

In trials on maize in France, Germany and Italy, 2 foliar applications of pirimicarb (50% WG formulation) were made at 5−14 day intervals to unreplicated 45−120 square metre plots, using small plot or knapsack mini boom sprayers to obtain full foliar coverage. Samples of young plants (12 plants without roots), straw (0.5kg or 12 plants) and cobs, with husks from at least 12 plants (4 kg) were taken for analysis using Methods RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQs of the methods was 0.01 mg/kg for all analytes and mean recovery rates were 75−105% at fortification levels of 0.01−5.0 mg/kg.

Table 95. Residues in maize cobs and kernels from foliar applications of pirimicarb (50% WG formulation) to maize in supervised trials in France, Germany and Italy.

MAIZE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Germany, 2000 (Domineco)	0.25	0.04	2	112 112	cobs < 0.01 kernals < 0.01	cobs < 0.01 kernals < 0.01	cobs < 0.01 kernals < 0.01	PP62/0979 (DE16-00-S167)
Germany, 2000 (Benecia)	0.25	0.04	2	126 126	cobs < 0.01 kernals < 0.01	cobs < 0.01 kernals < 0.01	cobs < 0.01 kernals < 0.01	PP62/0979 (DE15-00-S167)
France (S), 2001 Cecilia)	0.25	0.04	2	64 64	cobs < 0.01 kernals ≤ 0.01	cobs < 0.01 kernals ≤ 0.01	cobs < 0.01 kernals < 0.01	PP62/1139 (AF/5980/SY/1)
Germany, 1998 (Helix)	0.25	0.06	2	88	kernals 0.02	kernals 0.02	kernals 0.02	PP62/0452 (RS-9826-K1)
Italy, 1998 (Alired LG)	0.25	0.06	2	81	kernals ≤ 0.01	kernals ≤ 0.01	kernals < 0.01	PP62/0462 (IT20-98-E364)
Italy, 1998 (Gitana)	0.25	0.06	2	81	kernals ≤ 0.01	kernals ≤ 0.01	kernals < 0.01	PP62/0462 (IT20-98-E365)
Germany, 1998 (Turkis)	0.25	0.08	2	103	kernals ≤ 0.01	kernals ≤ 0.01	kernals < 0.01	PP62/0452 (RS-9826-G1)
France (N), 1998 (Nobilis)	0.25	0.08	2	87	kernals ≤ 0.01	kernals ≤ 0.01	kernals < 0.01	PP62/0461 (S104.98)
France (N), 1998 (Anjou 285)	0.25	0.08	2	77	kernals ≤ 0.01	kernals ≤ 0.01	kernals < 0.01	PP62/0461 (S218.98)
France (N), 1999 (DK 312)	0.25	0.08	2	98 98	cobs < 0.01 kernals ≤ 0.01	cobs < 0.01 kernals ≤ 0.01	cobs < 0.01 kernals < 0.01	PP62/0476 (AF/4756/ZE/1)
France (N), 1999 (Magister)	0.25	0.08	2	92 92	cobs < 0.01 kernals ≤ 0.01	cobs < 0.01 kernals ≤ 0.01	cobs < 0.01 kernals < 0.01	PP62/0476 (AF/4756/ZE/1)
France (S), 1998 (Cecilia)	0.25	0.08	2	100	kernals ≤ 0.01	kernals ≤ 0.01	kernals < 0.01	PP62/0462 (S564.98)
France (S), 1998 (Furio)	0.25	0.08	2	124	kernals < 0.01	kernals < 0.01	kernals < 0.01	PP62/0462 (S751.98)
France (S), 1999 (Cecilia)	0.25	0.08	2	97 97	cobs < 0.01 kernals ≤ 0.01	cobs < 0.01 kernals ≤ 0.01	cobs < 0.01 kernals < 0.01	PP62/0474 (AF/4752/ZE/1)
France (S), 1999 (DK 604)	0.25	0.08	2	97 97	cobs < 0.01 kernals ≤ 0.01	cobs < 0.01 kernals ≤ 0.01	cobs < 0.01 kernals < 0.01	PP62/0474 (AF/4752/ZE/2)
Italy, 1999 (Orange)	0.25	0.08	2	78 78	cobs < 0.01 kernals ≤ 0.01	cobs < 0.01 kernals ≤ 0.01	cobs < 0.01 kernals < 0.01	PP62/0474 (AF/4752/ZE/4)

'cobs' includes kernals

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Wheat

In trials on winter wheat in France and the UK, 2 foliar applications of pirimicarb (50% WG formulation) were made at 7−16 day intervals to unreplicated 39−70 square metre plots, using small plot boom sprayers to obtain full foliar coverage. Grain samples (0.5−1.0 kg, combine harvested) were taken for analysis using Methods RAM 265/01 (with GC-NPD detection), RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQs of the methods was 0.01 mg/kg and mean recovery rates were 74−116% at fortification levels of 0.01−1.0 mg/kg.

Table 96. Residues in wheat grain from foliar applications of pirimicarb (50% WG formulation) to wheat in supervised trials in France and the UK.

WHEAT Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France(N), 2000 (Versailles) winter wheat	0.15	0.05	2	up to BBCH 83	≤ 0.01	≤ 0.01	< 0.01	PP62/0926 (FR22-00-S761) sampled 46 days after last treatment
France(N), 2000 (Shango) winter wheat	0.15	0.05	2	up to BBCH 83	≤ 0.01	≤ 0.01	< 0.01	PP62/0926 (FR22-00-S771) sampled 37 days after last treatment
UK, 1994 (Apollo) winter wheat	0.15	0.08	3	up to BBCH 77-83	≤ 0.01	≤ 0.01	< 0.01	PP62/0487 (GB15-94-S211) sampled 33 days after last treatment
UK, 1994 (Lynx) winter wheat	0.15	0.08	3	up to BBCH 77-83	≤ 0.01	≤ 0.01	< 0.01	PP62/0487 (GB15-94-S212) sampled 39 days after last treatment
UK, 2001 (Charger) winter wheat	0.17+ 0.15	0.08+ 0.07	1+ 1	up to BBCH 77-83	≤ 0.01	≤ 0.01	< 0.01	PP62/1096 (AF/591/SY/1) sampled 35 days after last treatment
UK, 2001 (Savannah) winter wheat	0.15	0.07	2	up to BBCH83	≤ 0.01	≤ 0.01	< 0.01	PP62/1096 (AF/591/SY/2) sampled 30 days after last treatment
France (N), 1998 (Bourbon) winter wheat	0.15	0.08	2	up to BBCH83	≤ 0.01	≤ 0.01	< 0.01	PP62/0455 (AF/4166/CL/1) sampled 38 days after last treatment
France (N), 1998 (Altria) winter wheat	0.15	0.08	2	up to BBCH85	≤ 0.01	≤ 0.01	< 0.01	PP62/0455 (AF/4166/CL/2) sampled 21 days after last treatment

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Rape seed

In trials on oil seed rape in France, Spain and the UK, 1–2 foliar applications of pirimicarb (50% WG formulation) were made at 6–9 day intervals to unreplicated 50–144 square metre plots, using knapsack or small plot boom sprayers to obtain full foliar coverage. Samples of pods (1–2 kg) were taken and hand threshed or machine harvested, with both full pods and seeds being analysed using Method RAM 265/03 with HPLC-MS-MS detection to measure residues of pirimicarb and its carbamate metabolites. The LOQs of the methods was 0.01 mg/kg for all analytes and mean recovery rates were 87–100% at fortification levels of 0.01–1.0 mg/kg.

Table 97. Residues in oil seed rape from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France, Spain and the UK.

RAPE SEED Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
France (N), 1997 (Capitole)	0.25	0.08	1		seed+pod	seed+pod	seed+pod	PP62/0491 (S 211.97)
				0	2.6	0.59	0.09	
				3	0.53	0.41	0.01	
				7	0.3	0.26	0.01	
					seeds	seeds	seeds	
				15	≤ 0.01	≤ 0.01	< 0.01	
				21	< 0.01	< 0.01	< 0.01	
France (N), 1997 (Ascona)	0.25	0.08	1		seed+pod	seed+pod	seed+pod	PP62/0491 (S 408.97)
				0	3.5	0.3	0.07	
				3	0.95	0.54	0.04	
				8	0.72	0.58	0.05	
					seeds	seeds	seeds	
				15	≤ 0.01	≤ 0.01	< 0.01	
				22	< 0.01	< 0.01	< 0.01	
UK, 1997 (Falcon)	0.25	0.05	1		seed+pod	seed+pod	seed+pod	PP62/0491 (AK/3740/ZE/1)
				0	2.9	0.21	0.04	
				3	0.34	0.17	0.01	
				7	0.33	0.23	0.02	
					seeds	seeds	seeds	
				14	≤ 0.01	≤ 0.01	< 0.01	
				21	< 0.01	< 0.01	< 0.01	
UK, 1997 (Martina)	0.25	0.04	1		seed+pod	seed+pod	seed+pod	PP62/0491 (AK/3740/ZE/2)
				0	2.8	0.21	0.04	
				3	0.19	0.18	< 0.01	
				7	0.08	0.11	< 0.01	
					seeds	seeds	seeds	
				14	≤ 0.01	≤ 0.01	< 0.01	
				21	< 0.01	< 0.01	< 0.01	
UK, 1998 (Lipton)	0.25	0.04	2	19	seeds 0.02	seeds < 0.01	seeds < 0.01	PP62/0493 (AK/4172/CL/1)
UK, 1998 (Artus)	0.25	0.04	2	21	seeds 0.01	seeds < 0.01	seeds < 0.01	PP62/0493 (AK/4172/CL/2)
France (N), 1998 (Navajo)	0.24	0.05	2	15	seeds ≤ 0.01	seeds ≤ 0.01	seeds < 0.01	PP62/0493 (AK/4172/CL/3)
France (N), 1998 (Navajo)	0.25	0.04	2	17	seeds 0.02	seeds ≤ 0.01	seeds < 0.01	PP62/0493 (AK/4172/CL/4)
Spain, 1998 (Kreta)	0.25	0.06	1		seeds	seeds	seeds	PP62/0495 (ES10-98-SE004)
			1	-0	0.03	0.03	< 0.01	
			2	0	0.15	0.13	< 0.01	
				7	0.08	0.07	< 0.01	
				13	0.03	0.03	< 0.01	
				21	≤ 0.01	≤ 0.01	< 0.01	

RAPE SEED Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Spain, 1998 (Bristol)	0.25	0.05	1	-0	seeds < 0.01	seeds 0.02	seeds < 0.01	PP62/0495 (ES10-98-SE104)
			2	0	0.11	0.07	0.01	
				7	< 0.01	0.02	< 0.01	
				14	< 0.01	< 0.01	< 0.01	
				22	≤ 0.01	≤ 0.01	< 0.01	
France (S), 2001 (Kolosse)	0.25	0.08	2	14	seeds 0.02	seeds 0.02	seeds < 0.01	PP62/0501 (AF4751/ZE/1)
				48	< 0.01	< 0.01	< 0.01	
Spain, 2001 (Kabel)	0.25	0.08	2	11	seeds 0.2	seeds 0.12	seeds < 0.01	PP62/0501 (AF4751/ZE/2)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Sunflower seed

In trials on sunflowers in France, Italy and Spain, 3 foliar applications of pirimicarb (50% WG formulation) were made at 7–8 day intervals to unreplicated 45–180 square metre plots, using small plot or knapsack boom sprayers to obtain full foliar coverage. Samples of seed (1 kg) from at least 12 flower heads were taken for analysis using Method RAM 265/03 with HPLC-MS-MS detection to measure residues of pirimicarb and its carbamate metabolites. The LOQs of the method was 0.01 mg/kg and mean recovery rates were 83–93% at fortification levels of 0.01–0.5 mg/kg.

Table 98. Residues in sunflower seed from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France, Italy and Spain.

SUNFLOWER SEED Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
France (N), 1998 (Rigasol)	0.25	0.06	3	21	0.01	≤ 0.01	< 0.01	PP62/0499 (AF/4173/CL1)
France (N), 1998 (Flores)	0.25	0.06	3	21	≤ 0.01	≤ 0.01	< 0.01	PP62/0499 (AF/4173/CL2)
Spain, 1998 (Sambro)	0.26	0.06	3	21	0.03	0.01	< 0.01	PP62/0499 (AF/4173/CL3)
Spain, 1998 (Coronil)	0.25	0.06	3	21	≤ 0.01	≤ 0.01	< 0.01	PP62/0499 (AF/4173/CL4)
Italy, 1998 (Oilbaril)	0.26	0.06	3	21	≤ 0.01	≤ 0.01	< 0.01	PP62/0499 (AF/4173/CL5)
Italy, 1998 (Ramona)	0.25	0.06	3	21	0.01	≤ 0.01	< 0.01	PP62/0499 (AF/4173/CL6)
Spain, 1997 (Tesoro)	0.25	0.09	3		seed head	seed head	seed head	PP62/0497 (AF/3550/ZE1)
				0	1.2	0.19	< 0.01	
				3	0.15	0.16	< 0.01	
				7	0.28	0.17	< 0.01	
					seed	seed	seed	
				14	0.03	0.03	< 0.01	
				21	0.01	0.01	< 0.01	
Spain, 1997 (Poblon)	0.25	0.09	3		seed head	seed head	seed head	PP62/0497 (AF/3550/ZE2)
				0	2.0	0.19	< 0.01	
				3	1.4	0.27	< 0.01	
				7	0.85	0.18	< 0.01	
					seed	seed	seed	
				14	0.05	0.02	< 0.01	
				21	0.05	0.02	< 0.01	

SUNFLOWER SEED Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
Italy, 1997 (Solbel)	0.25	0.04	3		seed head	seed head	seed head	PP62/0497 (AF/3550/ZE3)
				0	0.7	0.07	< 0.01	
				3	0.02	0.01	< 0.01	
				7	0.03	0.01	< 0.01	
					seed	seed	seed	
				14	< 0.01	< 0.01	< 0.01	
				21	0.03	0.01	< 0.01	
Italy, 1997 (Vidoc)	0.25	0.04	3		seed head	seed head	seed head	PP62/0497 (AF/3550/ZE4)
				0	0.66	0.06	< 0.01	
				3	0.12	0.05	< 0.01	
				7	0.11	0.05	< 0.01	
					seed	seed	seed	
				14	0.03	0.02	< 0.01	
				21	0.01	< 0.01	< 0.01	
France (N), 1997 (Challenger)	0.25	0.08	2		seed head	seed head	seed head	PP62/0489 (97 I TO SA P39)
			3	-0	0.1	0.05	< 0.01	
				0	0.52	0.13	< 0.01	
				6	0.09	0.04	< 0.01	
				13	0.04	0.04	< 0.01	
					seed	seed	seed	
				21	0.03	< 0.01	< 0.01	
				28	0.03	< 0.01	< 0.01	
France (N), 1997 (Rigasol)	0.26	0.08	2		seed head	seed head	seed head	PP62/0489 (97 I TO SA P40)
			3	-0	0.03	0.03	< 0.01	
				0	0.37	0.13	< 0.01	
				7	0.12	0.05	< 0.01	
				14	0.15	0.07	< 0.01	
					seed	seed	seed	
				14	0.04	< 0.01	< 0.01	
				21	0.03	< 0.01	< 0.01	

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Sugar beet (tops)

In sugar beet trials in France, Italy, Spain and the UK, 2–4 foliar applications of pirimicarb (50% WG formulations) were made at 7–14 day intervals to unreplicated 30–120 square metre plots, as broadcast sprays using small plot or knapsack mini boom sprayers to obtain full foliar coverage. A minimum of 1.0 kg leaves and tops (or from 12 plants) were sampled for analysis using either Methods RAM 15/02 (with GC-MSD detection to measure residues of pirimicarb and the combined residues of demethyl pirimicarb (R34836) and demethylformamido pirimicarb (R34885)) or Methods RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 82–92% at fortification levels of 0.01–12.0 mg/kg. Residues are expressed on a fresh weight basis.

Table 99. Residues in sugar beet (leaves) from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France, Italy, Spain and the UK.

SUGAR BEET (TOPS) Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
UK, 1991 (Amethyst)	0.28	0.07	2	7	0.23	0.4		PP62/0268 (GB12-91-S061)

SUGAR BEET (TOPS) Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
UK, 1991 (Hilme)	0.28	0.07	2	7	0.14	0.42		PP62/0268 (GB12-91-S062)
UK, 1991 (Celt)	0.28	0.07	2	7	0.7	0.46		PP62/0268 (GB15-91-S062)
UK, 1991 (Rex)	0.28	0.07	2	7	2.4	0.92		PP62/0268 (GB15-91-S063)
UK, 1991 (Amethyst)	0.28	0.07	2	7	0.66	0.56		PP62/0268 (GB17-91-S061)
UK, 1991 (Hilma)	0.28	0.07	2	7	0.37	0.46		PP62/0268 (GB17-91-S062)
UK, 1991 (Amethyst)	0.28	0.07	4	7	0.25	0.42		PP62/0268 (GB12-91-S061)
UK, 1991 (Hilme)	0.28	0.07	4	7	0.15	0.29		PP62/0268 (GB12-91-S062)
UK, 1991 (Celt)	0.28	0.07	4	7	1.4	1.0		PP62/0268 (GB15-91-S062)
UK, 1991 (Rex)	0.28	0.07	4	7	2.0	1.0		PP62/0268 (GB15-91-S063)
UK, 1991 (Amethyst)	0.28	0.07	4	7	0.52	0.53		PP62/0268 (GB17-91-S061)
UK, 1991 (Hilma)	0.28	0.07	4	7	0.55	0.7		PP62/0268 (GB17-91-S062)
UK, 1992 (Celt)	0.28	0.07	2	7	0.22	0.22		PP62/0269 (GB12-92-S071)
UK, 1992 (Saxon)	0.28	0.07	2	7	0.26	0.25		PP62/0269 (GB12-92-S072)
UK, 1992 (Regina)	0.28	0.07	2	7	0.09	0.05		PP62/0269 (GB12-92-S073)
UK, 1992 (Giselle)	0.28	0.07	2	7	0.21	0.27		PP62/0269 (GB12-92-S074)
UK, 1992 (Celt)	0.28	0.07	4	7	0.29	0.19		PP62/0269 (GB12-92-S071)
UK, 1992 (Saxon)	0.28	0.07	4	7	0.27	0.31		PP62/0269 (GB12-92-S072)
UK, 1992 (Regina)	0.28	0.07	4	7	0.15	0.12		PP62/0269 (GB12-92-S073)
UK, 1992 (Giselle)	0.28	0.07	4	7	0.24	0.48		PP62/0269 (GB12-92-S074)
Spain, 1998 (Korif)	0.36	0.075	1 2	-0 0 3 7 10 14	0.58 10 4 3.3 3.1 2.4	0.41 1.6 1.4 1.6 1.5 1.4	< 0.01 0.02 0.02 0.01 0.01 0.01	PP62/0288 (ES10-98-SE102)
Spain, 1998 (Oryx)	0.37	0.075	1 2	-0 0 3 7 10 14	0.67 5.7 2.2 2.2 2.2 1.1	0.84 0.89 1.2 1.2 1.6 0.99	< 0.01 0.01 < 0.01 0.01 0.01 < 0.01	PP62/0288 (ES10-98-SE002)
Italy, 2001 (Dorotea)	0.38	0.061	1 2	-0 0 3 8 10 14	0.14 7.5 2.7 0.93 0.74 0.38	0.45 0.7 1.5 1.5 1.2 0.7	< 0.01 0.02 0.01 < 0.01 < 0.01 < 0.01	PP62/1229 (AF/5972/SY/3)

SUGAR BEET (TOPS) Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
Italy, 2001 (Eko)	0.38	0.062	1 2	-0 0 3 7 10 14	0.03 3.4 <u>0.92</u> 0.52 0.29 0.18	0.04 1.3 <u>1.1</u> 0.58 0.56 0.32	< 0.01 0.02 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1229 (AF/5972/SY/4)
France (S), 2001 (Sheriff)	0.38	0.073	1 2	-0 0 3 7 10 14	< 0.01 5.8 1.6 0.79 0.22 0.07	0.06 1.5 1.4 1.2 0.39 0.19	< 0.01 0.05 0.01 0.01 < 0.01 < 0.01	PP62/1229 (AF/5972/SY/2)
Italy, 1998 (Asso)	0.38	0.075	1 2	-0 0 3 8 10 14	0.07 1.3 0.33 0.03 < 0.01 < 0.01	0.53 0.99 1.2 0.45 0.27 0.08	< 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0288 (IT30-98-E366)
Italy, 1998 (Arma)	0.38	0.075	1 2	-0 0 3 7 10 14	0.05 4.7 0.56 0.08 0.02 < 0.01	0.43 0.95 0.93 0.46 0.21 < 0.01 (c=0.05)	< 0.01 0.05 < 0.01 < 0.01 < 0.01 < 0.01	PP62/0288 (IT20-98-E367)
France (S), 2001 (Sheriff)	0.38	0.079	1 2	-0 0 3 7 10 14	0.01 6.1 0.87 0.94 0.35 0.35	0.14 1.8 1.3 1.3 0.6 0.46	< 0.01 0.07 < 0.01 < 0.01 < 0.01 < 0.01	PP62/1229 (AF/5972/SY/1)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Barley forage and straw

In the trials on winter barley in France and the UK, samples of whole plants and straw (1kg) were taken for analysis using Methods RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ of the methods was 0.01 mg/kg for all analytes and the mean recovery rates were 75–94% at fortification levels of 0.01–1.0 mg/kg. Residues are expressed on a fresh weight basis.

Table 100. Residues in barley straw and fodder from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France and the UK.

BARLEY Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
UK, 1998 (Hanna) winter barley	0.15	0.08	2	14 21	whole plant < 0.01 straw <u>≤ 0.01</u>	whole plant < 0.01 straw <u>≤ 0.01</u>	whole plant < 0.01 straw < 0.01	PP62/0459 (AK/4165/CL/1) up to BBCH 85
UK, 1998 (Intro) winter barley	0.15	0.08	2	14 20	whole plant 0.22 straw <u>0.13</u>	whole plant 0.1 straw <u>0.08</u>	whole plant < 0.01 straw < 0.01	PP62/0459 (AK/4165/CL/2) up to BBCH 85

BARLEY Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
France (N), 1998 (Clarine) winter barley	0.15	0.08	2	14 29	whole plant 0.03 straw 0.02	whole plant 0.04 straw 0.05	whole plant < 0.01 straw < 0.01	PP62/0459 (AK/4165/CL/3) up to BBCH 83
UK, 2000 (Intro) winter barley	0.15	0.08	2	24	straw 0.02	straw 0.02	straw < 0.01	PP62/0942 (GB02-00-S082) up to BBCH 83
UK, 2000 (Regina) winter barley	0.15	0.08	2	24	straw 0.03	straw 0.01	straw < 0.01	PP62/0942 (GB02-00-S083) up to BBCH 83
UK, 2000 (Fanfare) winter barley	0.15	0.08	2	24	straw 0.08	straw 0.03	straw < 0.01	PP62/0942 (GB02-00-S084) up to BBCH 85
UK, 2000 (Regina) winter barley	0.15	0.08	2	24	straw 0.02	straw 0.02	straw < 0.01	PP62/0942 (GB02-00-S085) up to BBCH 83
France (N), 1998 (Sunrise) winter barley	0.16	0.08	2	14 21	whole plant 0.05 straw ≤ 0.01	whole plant 0.05 straw ≤ 0.01	whole plant < 0.01 straw < 0.01	PP62/0459 (AK/4165/CL/4) up to BBCH 85-87
UK, 2003 (Pearl) winter barley	0.38	0.13	2	21	straw 0.25	straw 0.12	straw < 0.01	PP62/1391 (AF/7360/SY/1) Processing study
UK, 2003 (Pearl) winter barley	0.75	0.25	2	21	straw 0.83	straw 0.3	straw 0.02	PP62/1391 (AF/7360/SY/1) Processing study

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Maize forage and fodder

In the trials on maize in France, Germany and Italy, samples of plants (12 plants without roots), straw (0.5kg or 12 plants) and cobs, with husks from at least 12 plants (4 kg) were taken for analysis using Methods RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQs of the methods was 0.01 mg/kg for all analytes and mean recovery rates were 75−105% at fortification levels of 0.01−5.0 mg/kg. Residues are expressed on a fresh weight basis.

Table 101. Residues in maize forage and fodder from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France, Germany and Italy.

MAIZE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[2]	R238177	
Germany, 2000 (Domineco)	0.25	0.04	2	8 112	whole plant 0.01 straw & husks < 0.01	whole plant 0.02 straw & husks < 0.01	whole plant < 0.01 straw & husks < 0.01	PP62/0979 (DE16-00-S167)

MAIZE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[2]	R238177	
Germany, 2000 (Benecia)	0.25	0.04	2	8 126	whole plant < 0.01 straw & husks < 0.01	whole plant 0.03 straw & husks < 0.01	whole plant < 0.01 straw & husks < 0.01	PP62/0979 (DE15-00-S167)
France (S), 2001 Cecilia)	0.25	0.04	2	7 35 64	whole plant 0.02 < 0.01 straw & husks ≤ 0.01	whole plant 0.01 < 0.01 straw & husks ≤ 0.01	whole plant < 0.01 < 0.01 straw & husks < 0.01	PP62/1139 (AF/5980/SY/1)
Germany, 1998 (Helix)	0.25	0.06	2	0 8 16 23 88	whole plant 1.9 0.02 < 0.01 < 0.01 straw[1] 0.02	whole plant 0.21 0.04 0.01 < 0.01 straw[1] ≤ 0.01	whole plant 0.04 < 0.01 < 0.01 < 0.01 straw[1] < 0.01	PP62/0452 (RS-9826-K1)
Italy, 1998 (Alired LG)	0.25	0.06	2	0 7 14 50 81	whole plant 1.2 0.06 0.01 ≤ 0.01 straw & husks ≤ 0.01	whole plant 0.25 0.06 < 0.01 ≤ 0.01 straw & husks ≤ 0.01	whole plant 0.03 < 0.01 < 0.01 < 0.01 straw & husks < 0.01	PP62/0462 (IT20-98-E364)
Italy, 1998 (Gitana)	0.25	0.06	2	0 7 14 50 81	whole plant 1.8 < 0.01 < 0.01 ≤ 0.01 straw & husks ≤ 0.01	whole plant 0.4 < 0.01 < 0.01 ≤ 0.01 straw & husks ≤ 0.01	whole plant 0.05 < 0.01 < 0.01 < 0.01 straw & husks < 0.01	PP62/0462 (IT20-98-E365)
Germany, 1998 (Turkis)	0.25	0.08	2	0 8 14 20 103	whole plant 2.3 0.02 < 0.01 < 0.01 straw[1] 0.02	whole plant 0.44 0.01 < 0.01 < 0.01 straw[1] ≤ 0.01	whole plant 0.07 < 0.01 < 0.01 < 0.01 straw[1] < 0.01	PP62/0452 (RS-9826-G1)
France (N), 1998 (Nobilis)	0.25	0.08	2	0 7 14 67 87	whole plant 4.2 0.02 < 0.01 ≤ 0.01 straw&husk ≤ 0.01	whole plant 0.36 0.07 < 0.01 ≤ 0.01 straw&husk ≤ 0.01	whole plant 0.07 < 0.01 < 0.01 < 0.01 straw&husk < 0.01	PP62/0461 (S104.98)
France (N), 1998 (Anjou 285)	0.25	0.08	2	0 7 14 50 77	whole plant 3.4 0.15 0.04 ≤ 0.01 straw & husk 0.02	whole plant 0.17 0.06 0.03 ≤ 0.01 straw&husk ≤ 0.01	whole plant 0.01 < 0.01 < 0.01 < 0.01 straw&husk < 0.01	PP62/0461 (S218.98)

MAIZE Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[2]	R238177	
France (N), 1999 (DK 312)	0.25	0.08	2	7 98	whole plant 0.02 straw & husks ≤ 0.01	whole plant < 0.01 straw & husks ≤ 0.01	whole plant < 0.01 straw & husks < 0.01	PP62/0476 (AF/4756/ZE/1)
France (N), 1999 (Magister)	0.25	0.08	2	7 92	whole plant < 0.01 straw & husks ≤ 0.01	whole plant 0.01 straw & husks ≤ 0.01	whole plant < 0.01 straw & husks < 0.01	PP62/0476 (AF/4756/ZE/1)
France (S), 1998 (Cecilia)	0.25	0.08	2	0 7 14 70 100	whole plant 4.3 < 0.01 < 0.01 ≤ 0.01 straw & husks ≤ 0.01	whole plant 0.3 < 0.01 < 0.01 ≤ 0.01 straw & husks ≤ 0.01	whole plant 0.04 < 0.01 < 0.01 < 0.01 straw & husks < 0.01	PP62/0462 (S564.98)
France (S), 1998 (Furio)	0.25	0.08	2	0 7 13 49 124	whole plant 1.6 0.03 < 0.01 ≤ 0.01 straw & husks < 0.01	whole plant 0.26 0.09 0.03 ≤ 0.01 straw & husks < 0.01	whole plant 0.04 < 0.01 < 0.01 < 0.01 straw & husks < 0.01	PP62/0462 (S751.98)
France (S), 1999 (Cecilia)	0.25	0.08	2	7 97	whole plant 0.02 straw & husks ≤ 0.01	whole plant 0.02 straw & husks ≤ 0.01	whole plant < 0.01 straw & husks < 0.01	PP62/0474 (AF/4752/ZE/1)
France (S), 1999 (DK 604)	0.25	0.08	2	7 97	whole plant < 0.01 straw & husks ≤ 0.01	whole plant 0.01 straw & husks ≤ 0.01	whole plant < 0.01 straw & husks < 0.01	PP62/0474 (AF/4752/ZE/2)
Italy, 1999 (Orange)	0.25	0.08	2	7 78	whole plant < 0.01 straw & husks ≤ 0.01	whole plant < 0.01 straw & husks ≤ 0.01	whole plant < 0.01 straw & husks < 0.01	PP62/0474 (AF/4752/ZE/4)

[1]) 'straw' means whole plants (without roots), after removal of mature cobs and husks.

[2]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Wheat straw and fodder

In the trials on winter wheat in France and the UK, samples of young plants (1 kg) and straw (0.5kg) were taken for analysis using Methods RAM 265/01 with GC-NPD detection, RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQs of the methods was 0.01 mg/kg for all analytes except in the 1994 UK trials, where the LOQ in straw was 0.05 mg/kg (RAM 265/01). Mean recovery rates were 74–116% at fortification levels of 0.01–1.0 mg/kg. Residues are expressed on a fresh weight basis.

Table 102. Residues in wheat straw and fodder from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France and the UK

WHEAT Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
France(N), 2000 (Versailles) winter wheat	0.15	0.05	2	46	straw 0.02 (c=0.02)	straw 0.02	straw < 0.01	PP62/0926 (FR22-00-S761) up to BBCH 83
France(N), 2000 (Shango) winter wheat	0.15	0.05	2	37	straw ≤ 0.01	straw ≤ 0.01	straw < 0.01	PP62/0926 (FR22-00-S771) up to BBCH 83
UK, 1994 (Apollo) winter wheat	0.15	0.08	3	33	straw 0.07[2]	straw 0.15[2]	straw < 0.05	PP62/0487 (GB15-94-S211) up to BBCH 83
UK, 1994 (Lynx) winter wheat	0.15	0.08	3	39	straw ≤ 0.05[2]	straw 0.08[2]	straw < 0.05	PP62/0487 (GB15-94-S212) up to BBCH 83
UK, 2001 (Charger) winter wheat	0.17+ 0.15	0.08+ 0.07	1+ 1	35	straw 0.02	straw 0.03	straw < 0.01	PP62/1096 (AF/591/SY/1) up to BBCH 83
UK, 2001 (Savannah) winter wheat	0.15	0.07	2	30	straw 0.16	straw 0.16	straw < 0.01	PP62/1096 (AF/591/SY/2) up to BBCH 83
France (N), 1998 (Bourbon) winter wheat	0.15	0.08	2	14 38	whole plant 0.04 straw 0.02	whole plant 0.06 straw 0.06	whole plant < 0.01 straw < 0.01	PP62/0455 (AF/4166/CL/1) up to BBCH 83
France (N), 1998 (Altria) winter wheat	0.15	0.08	2	14 21	whole plant 0.03 straw ≤ 0.01	whole plant 0.03 straw ≤ 0.01	whole plant < 0.01 straw < 0.01	PP62/0455 (AF/4166/CL/2) up to BBCH 85

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

[2]) Limit of determination is 0.05 mg/kg

Bean forage

Samples of bean foliage were taken from two of the common bean trials in Spain, the broad bean trials in the UK and the dry bean trials in France. In these trials, 2 foliar applications of pirimicarb (50% WG formulations) were made at 7−12 day intervals to unreplicated 30−120 square metre plots, either as broadcast or band sprays using small plot boom or knapsack (hand lance or mini boom) sprayers to obtain full foliar coverage. The samples (1.0 kg) were analysed using Methods RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 79−102% at fortification levels of 0.01−5.0 mg/kg. Residues are expressed on a fresh weight basis.

Table 103. Residues in common bean vines from foliar applications of pirimicarb (50% WG formulation) in supervised trials in Spain.

COMMON BEANS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+R34885[1]	R238177	
Spain, 1998 (Superba)	0.6	0.05	2	3	plants[2] 1.4	plants[2] 2.0	plants[2] 0.01	PP62/0366 (AF/4168/CL/1)
				7	0.72	1.6	< 0.01	
Spain, 1998 (Roma II Planta)	0.6	0.05	2	3	plants[1] 3.6	plants[1] 3.2	plants[2] 0.02	PP62/0366 (AF/4168/CL/2)
				7	1.3	2.4	< 0.01	

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

[2]) plants without pods

Table 104. Residues in broad bean vines from foliar applications of pirimicarb (50% WG formulation) in supervised trials in the UK.

BROAD BEANS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[3]	R238177	
UK, 2001 (Drednort)	0.25	0.06	1	-0	empty pods < 0.01	empty pods < 0.01	empty pods < 0.01	PP62/1178 (AF/5968/SY3)
			2	0	0.78	0.09	< 0.01	
					foliage[1]	foliage[1]	foliage[1]	
			1	-0	0.13	0.42	< 0.01	
			2	0	5.7	1.2	0.03	
				3	0.25	0.45	< 0.01	
				7	0.06	0.15	< 0.01	
UK, 2001 (Danko)	0.25	0.06	1	-0	empty pods 0.01	empty pods 0.03	empty pods < 0.01	PP62/1178 (AF/5968/SY/4)
			2	0	1.1	0.07	< 0.01	
					foliage[1]	foliage[1]	foliage[1]	
			1	-0	0.06	0.23	< 0.01	
			2	0	9.1	0.84	0.05	
				3	0.11	0.29	< 0.01	
				7	0.05	0.06	< 0.01	
UK, 2001 (Drednort)	0.38	0.09	1	-0	empty pods < 0.01	empty pods < 0.01	empty pods < 0.01	PP62/1178 (AF/5968/SY3)
			2	0	1.1	0.13	< 0.01	
					foliage[1]	foliage[2]	foliage[1]	
			1	-0	0.17	0.55	< 0.01	
			2	0	8.1	1.8	0.06	
				3	0.43	0.77	< 0.01	
				7	0.07	0.19	< 0.01	
UK, 2001 (Danko)	0.38	0.09	1	-0	empty pods 0.13	empty pods 0.04	empty pods < 0.01	PP62/1178 (AF/5968/SY/4)
			2	0	1.2	0.08	< 0.01	
					foliage[1]	foliage[1]	foliage[1]	
			1	-0	0.15	0.39	< 0.01	
			2	0	16	1.7	0.14	
				3	0.27	0.34	< 0.01	
				7	0.08	0.07	< 0.01	
UK, 2002 (Wilkem major)	0.25	0.05	1	-0	plants[2] 0.02	plants[2] 0.05	plants[2] < 0.01	PP62/1299 & PP62/1346 (AF/6542/SY/1)
			2	0	0.83	0.13	0.01	
				3	0.08	0.12	< 0.01	
				7	0.07	0.09	< 0.01	

BROAD BEANS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[3]	R238177	
UK, 2002 (Wilkem major)	0.38	0.08	1	-0	plants[2] 0.04	plants[2] 0.07	plants[2] < 0.01	PP62/1299 & PP62/1346 (AF/6542/SY/1)
			2	0	1.5	0.4	0.04	
				3	0.13	0.19	< 0.01	
				7	0.09	0.18	< 0.01	
UK, 2002 (Listra)	0.25	0.05	1	-0	plants[2] 0.02	plants[2] 0.04	plants[2] < 0.01	PP62/1299 & PP62/1346 (AF/6542/SY/2)
			2	0	2.0	0.39	0.01	
				3	0.19	0.3	< 0.01	
				7	0.07	0.12	< 0.01	
UK, 2002 (Listra)	0.38	0.08	1	-0	plants[2] 0.03	plants[2] 0.03	plants[2] < 0.01	PP62/1299 & PP62/1346 (AF/6542/SY/2)
			2	0	4.3	0.87	0.03	
				3	0.12	0.06	< 0.01	
				7	0.05	0.1	< 0.01	

[1]) 'foliage' means the remaining vines after removal of beans and pods

[2]) 'plants' means empty pods and foliage (haulms).

[3]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Table 105. Residues in dry bean vines and empty pods from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France.

BEANS (DRY) Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[2]	R238177	
France (S), 2001 (Linex)	0.5	0.06	1	-0	empty pods 0.01	empty pods 0.03	empty pods < 0.01	PP62/1188 (AF5969/SY/1)
			2	0	1.4	0.24	< 0.01	
					plants[1]	plants[1]	plants[1]	
			1	-0	0.05	0.25	< 0.01	
			2	0	9.9	1.6	0.04	
				3	0.21	0.22	< 0.01	
				7	0.06	0.09	< 0.01	
France (S), 2001 (Linex)	0.5	0.06	1	-0	empty pods 0.1	empty pods 0.08	empty pods < 0.01	PP62/1188 (AF5969/SY/2)
			2	0	3.8	0.53	0.02	
					plants[1]	plants[1]	plants[1]	
			1	-0	0.02	0.08	< 0.01	
			2	0	6.8	1.5	0.04	
				3	0.29	0.33	< 0.01	
				7	0.09	0.14	< 0.01	
France (S), 2001 (Linex)	0.5	0.06	1	-0	empty pods 0.04	empty pods 0.07	empty pods < 0.01	PP62/1188 (AF5969/SY/3)
			2	0	1.7	0.17	< 0.01	
					plants[1]	plants[1]	plants[1]	
			1	-0	0.07	0.21	< 0.01	
			2	0	10.0	1.3	0.04	
				3	0.19	0.14	< 0.01	
				7	0.1	0.13	< 0.01	
France (S), 2001 (Linex)	0.5+ 0.5	0.1+ 0.08	1+ 1	-0	empty pods 0.18	empty pods 0.26	empty pods < 0.01	PP62/1188 (AF5969/SY/4)
				0	3.6	0.59	0.03	
					plants[1]	plants[1]	plants[1]	
			1+	-0	0.09	0.29	< 0.01	
			1	0	8.9	2.5	0.14	
				3	0.33	0.3	< 0.01	
				7	0.08	0.07	< 0.01	

[1]) 'plants' means empty pods and foliage (haulms).

[2]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Pea vines, hay or fodder

Pea vines were sampled in the fresh and dry pea trials conducted in France, Italy, Spain and the UK where 1−2 foliar applications of pirimicarb (50% WG formulations) were made at 7−12 day intervals to unreplicated 35−240 square metre plots, as broadcast sprays using small plot or knapsack mini boom sprayers to obtain full foliar coverage. A minimum of 0.8-1.0 kg vines and empty pods were taken for analysis using either Methods RAM 265/02 (with GC-NPD detection to measure residues of pirimicarb and the combined residues of demethyl pirimicarb (R34836) and demethylformamido pirimicarb (R34885) or Methods RAM 265/03 or RAM 265/04 (both with HPLC-MS-MS detection) to measure residues of pirimicarb and its carbamate metabolites. The LOQ was 0.01 mg/kg and mean recovery rates were 77−102% at fortification levels of 0.01−20.0 mg/kg.

Table 106. Residues in peas vines and empty pods from foliar applications of pirimicarb (50% WG formulation) in supervised trials in France and the UK.

PEAS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[3]	R238177	
UK, 2001					empty pods	empty pods	empty pods	PP62/1180
(Waverex)	0.38	0.09	1	-0	< 0.01	0.02 (c=0.02)	< 0.01	(AF/5971/SY/1)
			2	0	1.7	0.41	0.05	
Vining peas				3	0.49[1]	0.33[1]	0.021	
				7	0.09[1]	0.09[1]	< 0.011	
					foliage[2]	foliage[2]	foliage[2]	
			1	-0	0.34	0.25	< 0.01	
			2	0	8.7 (c=0.08)	1.1 (c=0.07)	0.15	
UK, 2001					empty pods	empty pods	empty pods	PP62/1180
(Gallant)	0.38	0.09	1	-0	< 0.01	< 0.01	< 0.01	(AF/5971/SY/2)
			2	0	1.4	0.16	0.01	
Vining peas				3	0.6[1]	0.37[1]	0.021	
				7	0.37[1]	0.21[1]	< 0.011	
					foliage[2]	foliage[2]	foliage[2]	
			1	-0	0.09	0.04	< 0.01	
			2	0	7.4	0.58	0.02	
France (S), 2001					empty pods	empty pods	empty pods	PP62/1174
(Arabelle)	0.38	0.09	1	-0	< 0.01	< 0.01	< 0.01	(AF/5970/SY/1)
			2	0	0.53	0.16	0.01	
Vining peas								
					foliage[2]	foliage[2]	foliage[2]	
			1	-0	0.06	0.06	< 0.01	
			2	0	7.0	1.0	0.03	
				3	0.6[1]	0.35[1]	< 0.011	
				7	0.44[1]	0.17[1](c=0.01)	< 0.011	
France (S), 2001					empty pods	empty pods	empty pods	PP62/1174
(Ast)	0.38	0.09	1	-0	< 0.01	< 0.01	< 0.01	(AF/5970/SY/2)
			2	0	0.14	0.1	< 0.01	
Vining peas								
					foliage[2]	foliage[2]	foliage[2]	
			1	-0	0.02	0.06	< 0.01	
			2	0	2.5	1.1	< 0.01	
				3	0.11[1]	0.24[1]	< 0.011	
				7	0.02[1]	0.07[1]	< 0.011	
France (N), 1992					empty pods	empty pods		PP62/0373
(Marlene)	0.38	0.13	1	7	< 0.01	< 0.01		(S 322.92)
France (N), 1992					empty pods	empty pods		PP62/0373
(Solara)	0.38	0.13	1	7	< 0.01	< 0.01		(S 341.92)
France (N), 1992					empty pods	empty pods		PP62/0373
(Carla)	0.38	0.13	1	7	< 0.01	< 0.01		(S 201.92)
France (N), 1992					empty pods	empty pods		PP62/0373
(Santon)	0.38	0.13	1	7	< 0.01	< 0.01		(S 202.92)

PEAS Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[3]	R238177	
Italy, 2001 (Resal)	0.5+ 0.5	0.1+ 0.07	1+ 1	-0 0 3 7 14	straw 0.02 6.2 0.79 0.55 0.5	straw 0.08 1.4 0.63 0.48 0.29	straw < 0.01 0.04 0.01 0.02 0.03	PP62/1164 (AF5716/SY/8)
Italy, 2001 (Lambado)	0.5+ 0.5	0.1+ 0.08	1+ 1	-0 0 3 7 14	straw < 0.01 4.3 0.49 0.14 0.16	straw 0.04 1.2 0.44 0.17 0.14	straw < 0.01 0.06 0.01 < 0.01 0.01	PP62/1164 (AF5716/SY/6)

1) mean residues in vines or empty pods after hand separation and mechanical separation

2) vines after removal of peas and pods

[3]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

Table 107. Residues in vines and empty pods from foliar applications of pirimicarb (50% WG formulation) to peas (dry) in supervised trials in France, Italy and Spain.

PEAS (DRY) Country, year (variety)	Application			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836+ R34885[1]	R238177	
France (S), 2001 (Solara)	0.5	0.06	1 2 1 2	-0 0 -0 0 3 7 14	empty pods < 0.01 0.94 straw 0.31 7.1 2.7 2.2 1.7	empty pods < 0.01 0.53 straw 0.11 2.4 1.8 1.7 1.6 (c=0.02)	empty pods < 0.01 0.02 straw < 0.01 0.08 0.03 0.04 0.05	PP62/1164 (AF5716/SY/2)
Spain, 2001 (Calibra)	0.5	0.06	1 2	-0 0 3 7 14	straw < 0.01 4.9 0.34 0.15 0.34	straw 0.02 0.5 0.36 0.23 0.21 (c=0.02)	straw < 0.01 0.01 < 0.01 < 0.01 < 0.01	PP62/1164 (AF5716/SY/3)
Spain, 2001 (Gracia)	0.5	0.06	1 2	-0 0 3 7 14	straw 0.01 3.3 0.89 0.34 0.25	straw 0.03 1.4 0.63 0.28 0.21	straw < 0.01 0.05 0.01 < 0.01 < 0.01	PP62/1164 (AF5716/SY/4)
France (S), 2001 (Victor)	0.5+ 0.5	0.08+ 0.1	1+ 1	-0 0 3 7 14	straw 3.9 17 6.8 6.8 0.57	straw 0.65 0.95 1.2 1.3 0.08	straw 0.06 0.08 0.06 0.06 < 0.01	PP62/1164 (AF5716/SY/7)
Spain, 1999 (Ballet) Field peas	0.53	0.05	2	3 7	straw 17 14	straw 4.3 (c=0.01) 3.8 (c=0.01)	straw 0.09 0.06	PP62/0368 (AF/4127/CL/2)
Spain, 1999 (Ballet) Field peas	0.56	0.05	2	3 7	straw 8.4 2.9	straw 1.8 (c=0.02) 1.1 (c=0.02)	straw 0.04 0.03	PP62/0368 (AF/4127/CL/1)

[1]) combined carbamate metabolite residues (de-methyl + de-methylformamido pirimicarb, as de-methyl pirimicarb)

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

Pirimicarb is not registered for use in stored products.

In processing

The Meeting received information on the fate of residues of pirimicarb under simulated processing conditions and on the fate of incurred residues of pirimicarb during the processing of apples, plums, cherries, tomatoes, kale, lettuce, head cabbage, Brussels sprouts, potatoes and barley.

Fate of residues under simulated processing conditions

Study 1. An experiment was carried out to determine the stability of pirimarb and its carbamate metabolites in boiling salt water (Edwards *et al*., 1976: PP62/0523). The water was analysed for parent and R34836 using GC-NPD method PPRAM 15/1. The reported LOQ was 0.01 mg/kg for each analyte. Recovery values of 87–91% were found for pirimicarb and 84–89% for R34836.

Study 2. A hydrolysis study was performed to assess the possible production of breakdown or reaction products from pirimicarb residues in raw products during processing of crop commodities (Robertson, 2002: PP62/1197). For this purpose, pirimicarb was exposed to three sets of conditions: pasteurization, baking/brewing/boiling and sterilization.

Radioactivity was measured by LSC and solutions were analysed by 2D-TLC against standard reference compounds for parent, carbamates (R34836, R35140, R34885) and hydroxypyrimidines (R31805, R34865).

Total recovered radioactivity was 100–105% for all samples. Pirimicarb underwent minimal degradation under hydrolysis and the majority of the radioactivity was recovered unchanged (86% to 94% TAR).

Processing studies on pome fruit (apples)

Study 1. Apples from two supervised residue trials in Germany were used for a processing study (Specht, 1992: PP62/0408). Because no residue data on the raw agricultural commodity were available, processing factors and % transferred residues could not be calculated from this study and the study was therefore not summarized.

Study 2. Apples from a residue trial in Italy were used for a processing study (Mills *et al*., 2001: PP62/0982). Pirimicarb was applied as a 500 WG formulation, twice at a rate of 0.05 kg ai/hL with a spray interval of 7 days. Apples were harvested 7 days after final treatment. Apple samples were then stored to at 5–10°C for 1–2 days until processing. Samples were then processed into apple juice, pomace and apple sauce. Processed samples were stored at -18 °C for a maximum of 173 days. Extracts were stored at 4 °C for a maximum of 13 days prior to analysis (LOQ was 0.01 mg/kg)

The results of the processing study are summarised in Table 108.

Study 3. Apples from a supervised residue trial in Northern France were used for a processing study (Brice *et al*., 2004: PP62/1390). Pirimicarb was applied as a 500 WG formulation, twice at a rate of 0.0375 kg ai/hL with a spray interval of 7 days. Samples were taken 7 days after the final application. Three samples of 25 kg were taken from at least 12 points in the plot. Samples were processed into washed apples, apple juice, apple sauce and dry pomace.

Samples were stored at -18 °C for a maximum of 114 days. Extracts were stored at 4 °C for a maximum of 1 day. All samples were analysed for parent, R34885R34836 and R238177 using method RAM/265/04 with HPLC-MS-MS detection. The reported LOQ was 0.01 mg/kg.

The results of the processing study are summarised in Table 108. Results were not corrected for mean concurrent method recoveries (71–91%, nor for matrix interferences (< LOQ).

Table 108. Residues of pirimicarb after processing of apples.

Location, year, (variety)	Treat-ment	Commodity	parent mg/kg	R34836 mg/kg (a)	R238177mg/kg	Total mg/kg (b)	PF	MF	%T	reference
San Bonifacio, Piemonte, Italy, 2000, (Red Chief)	2x 0.05 kg ai/hL DAT = 7	RAC	0.04	0.02	< 0.01	0,06		-		Mills *et al*, 2001 PP62/0982 trial IT20-00-S391
		Washed apple	0.03	0.01	< 0.01	0,04	0,66	1.0	66	
		Wet pomace	0.07	0.03	< 0.01	0,10	1,66	0.23	38	
		Dry pomace	0.23	0.09	< 0.01	0,33	5,32	0.066	35	
		Raw juice	0.02	0.01	< 0.01	0,03	0,50	0.75	38	
		Apple juice	0.02	0.01	< 0.01	0,03	0,50	0.75	38	
		Wash water [d]	0.17 [c]	0.28 [c]	< 0.01 [c]	0,47	7,63	-		
		RAC	0.03	0.02	< 0.01	0,05		-		
		Washed apple	0.03	0.01	< 0.01	0,04	0,79	1.0	79	
		Chopped	0.02	< 0.01	< 0.01	0,02	0,39	0.71	28	
		Apple peel	0.10	0.04	< 0.01	0,14	2,78	0.12	33	
		Apple sauce	0.01	< 0.01	< 0.01	0,01	0,20	0.68	13	
		Wash water [d]	0.17 [c]	0.28 [c]	< 0.01 [c]	0,47	9,12	-		
St. Hillaire St Mesmin, Loiret, N France, 2003 (Golden)	2x 0.0375 kg ai/hL DAT = 7	RAC	0.07	0.01	< 0.01	0,08		-		Brice *et al*., 2004 PP62/1390 trial AF/7359/SY/1
		Washed apple	0.03	< 0.01	< 0.01	0,03	0,37	1.0	37	
		Dry pomace	0.34	0.06	< 0.01	0,40	5,01	0.063	32	
		Apple juice	0.06	< 0.01	< 0.01	0,06	0,74	0.54	40	
		Apple sauce	0.04	< 0.01	< 0.01	0,04	0,50	0.50	25	
		RAC	0.08	< 0.01	< 0.01	0,08		-		
		Washed apple	0.07	0.01	< 0.01	0,08	1,01	1.0	101	
		Dry pomace	0.39	0.05	< 0.01	0,44	5,54	0.067	37	
		Apple juice	0.06	< 0.01	< 0.01	0,06	0,75	0.60	45	
		Apple sauce	0.04	< 0.01	< 0.01	0,04	0,50	0.52	26	
		RAC	0.05	< 0.01	< 0.01	0,05		-		
		Washed apple	0.11	0.01	< 0.01	0,12	2,41	1.0	241	
		Dry pomace	0.34	0.04	< 0.01	0,38	7,65	0.067	51	
		Apple juice	0.05	< 0.01	< 0.01	0,05	1,00	0.57	57	
		Apple sauce	0.05	< 0.01	< 0.01	0,05	1,00	0.53	53	

- not analysed or not applicable

PF processing factor is total residue in the processed commodity : total residue in the RAC.

MF mass fraction is mass of processed product (corrected for subfractionation) : mass of starting material (RAC) %T percentage transference of residues is PF x MF x 100%.

a Residues of desmethyl formamido pirimicarb (R34885, measured as R34836)and desmethyl pirimicarb (R34836).

b Total pirimicarb residue = pirimicarb + 1.06x R34836. Because residues of the demethyl metabolites did not generally contribute significantly to the total residue, they are only included in the total where they were reported at levels above the LoQ

c Value in washing water given as µg/L. R34836 in wash water does not include R34885.

d For washing water, the percentage transference is calculated from absolute residue in water: absolute residue in RAC x 100%. The absolute amount of residue in the water (in mg) is calculated from A µg/L residue in water x B L water / 1000. The absolute residue in the RAC (in mg) is calculated from C mg/kg residue in RAC x D kg RAC.

Processing studies on stonefruit (cherries, plums)

Study 1. Cherries from three supervised residue trials in Germany were used for a processing study (Specht, 1993: PP62/0411). Because no residue data on the raw agricultural commodity were available, processing factors, mass fractions and % transferred values could not be calculated from and the study was therefore not summarized.

Study 2. Plums from a supervised residue trial in Southern France were used for a processing study (Brice *et al*., 2004: PP62/1389, Milhan, 2004 and Simmons, 2006). Pirimicarb was applied as a 500 WG formulation, twice at a rate of 0.05 kg ai/hL with a spray interval of 7 days. Samples were

taken 6 days after the final application. Samples were processed into washed plums, canned plums, plum jam, plum puree and prunes.

Samples were stored at -18 °C for a maximum of 170 days. Extracts were stored at 10 °C for a maximum of 6 days. Samples were analysed for parent, R34885, R34836 and R238177. The reported LOQ was 0.01 mg/kg.

The results of the process study are summarised in Table 109. Results were not corrected for mean concurrent method recoveries (84–92%) nor for matrix interferences (< LOQ).

Table 109. Residues of pirimicarb after processing of plums.

Location, year, (variety)	Treat-ment	Commodity	parent mg/kg	R34836 mg/kg (a)	R238177mg/kg	Total mg/kg (b)	PF	MF	%T	reference
Moissac, Tarn et Garonne, S. France, 2003, (plums: Denthes)	2× 0.050 kg ai/hL DAT = 6	RAC	0.24	0.02	0.05	0,26		-		Brice *et al*., 2004: PP62/1389 trial AF/7362/ SY/1
		Washed plum	0.25	0.02	0.05	0,27	1,04	1.0	104	
		Wash water [d]	2.3 [c]	0.66 [c]	-	3,00	11,5	-		
		Canned	0.16	0.01	0.02	0,17	0,65	1.2	78	
		plums	0.23	0.02	0.04	0,25	0,96	0.83	80	
		Jam	0.33	0.02	0.05	0,35	1,34	0.20	27	
		Wet pomace	0.28	0.02	0.05	0,30	1,15	0.54	62	
		Puree	0.40	0.04	0.10	0,44	1,69	0.25	42	
		Prunes								
		RAC	0.26	0.02	0.05	0,28		-		
		Washed plum	0.23	0.02	0.05	0,25	0,89	1.0	89	
		Canned	0.13	< 0.01	0.03	0,13	0,46	1.3	60	
		plums	0.18	0.01	0.04	0,19	0,68	0.78	53	
		Jam	0.27	0.02	0.05	0,29	1,04	0.63	65	
		Puree	0.74	0.05	0.11	0,79	2,82	0.27	76	
		Prunes								
		RAC	0.28	0.02	0.05	0,30		-		
		Washed plum	0.24	0.02	0.05	0,26	0,87	1.0	87	
		Canned	0.16	0.01	0.03	0,17	0,57	1.3	74	
		plums	0.23	0.01	0.03	0,24	0,80	0.77	62	
		Jam	0.26	0.02	0.04	0,28	0,93	0.44	41	
		Puree	0.57	0.05	0.11	0,62	2,07	0.29	60	
		Prunes								
		RAC	0.35	0.02	0.06	0,37		-		
		Washed plum	0.26	0.02	0.06	0,28	0,76	1.0	76	
		Canned	0.10	< 0.01	0.02	0,10	0,27	1.2	32	
		plums	0.23	0.02	0.04	0,25	0,68	0.73	49	
		Jam	0.24	0.02	0.04	0,26	0,70	0.58	41	
		Puree	0.66	0.05	0.11	0,71	1,92	0.29	56	
		Prunes								

- not analysed or not applicable

PF processing factor is total residue in the processed commodity : total residue in the RAC.

MF mass fraction is mass of processed product (corrected for subfractionation) : mass of starting material (RAC)

%T percentage transference of residues is PF x MF x 100%.

a Residues of R34885 (determined as R34836) and R34836

b Total pirimicarb residue = pirimicarb + 1.06x R34836. Because residues of the demethyl metabolites did not generally contribute significantly to the total residue, they are only included in the total where they were reported at levels above the LoQ.

c Value in washing water given as µg/L. R34836 in wash water does not include R34885.

d For washing water, the percentage transference is calculated from absolute residue in water: absolute residue in RAC x 100%. The absolute amount of residue in the water (in mg) is calculated from A µg/L residue in water x B L water / 1000. The absolute residue in the RAC (in mg) is calculated from C mg/kg residue in RAC x D kg RAC.

Processing studies on fruiting vegetables other than cucurbits (tomatoes)

Study 1. Tomatoes from a supervised residue trial in Italy were used for a processing study (Miles and Bonfanti, 1999: PP62/0525). Pirimicarb was applied as a 500 WG formulation, twice at a rate of 0.05 kg ai/hL with a spray interval of 7 days. Samples were taken 3 days after the final application and stored for 1 day at ambient temperature and 2 days at 3−5 °C until processed into washed tomatoes, peeled tomatoes, tomato puree, tomato ketchup, tomato juice and canned tomatoes.

Samples were stored at -18 °C for a maximum of 8 months. All samples were analysed for parent, R34885 R34836 and R238177 using method RAM/265/03 with GC-NPD detection. The reported LOQ was 0.01 mg/kg for each analyte.

The results of the processing study are summarised in Table 110. Results were not corrected for mean concurrent method recoveries (84%−86%), nor for matrix interferences (< LOQ).

Study 2. Tomatoes from a supervised residue trial in Southern France were used for a processing study (Brice *et al.*: PP62/1392). Pirimicarb was applied as a 500 WG formulation, twice at a rate of 0.10 kg ai/hL with a spray interval of 11 days. Samples were taken 3 days after the final application and were stored for 1 day at ambient temperatures until processed into washed tomatoes, tomato juice and pomace, tomato puree and canned tomatoes.

Samples were stored at -18 °C for a maximum of 158 days. Extracts were stored at 4°C for a maximum of 7 days. All samples were analysed for parent, R34885, R34836 and R238177. The reported LOQ was 0.01 mg/kg for each analyte.

The results of the processing study are summarised in Table 110. Results were not corrected for mean concurrent method recoveries (76−110%), nor for matrix interferences (< LOQ).

Table 110. Residues of pirimicarb after processing of tomatoes.

Location, year, (variety)	Treat-ment	Commodity	parent mg/kg	R34836 mg/kg (a)	R238177mg/kg	Total mg/kg (b)	PF	MF	%T	reference
Fiorenzuola d'Arda, Emilia Romagna, Italy, 1997 (Red River)	2x 0.050 kg ai/hL DAT = 3	RAC	0.10	0.03	< 0.01	0,13		-		Miles and Bonfanti, 1999: PP62/0525 trial IT33-97-E379
		washed	0.10	0.02	< 0.01	0,12	0,92	1.0	92	
		peeled	0.04	< 0.01	< 0.01	0,04	0,30	0.93	28	
		peels	0.23	0.01	< 0.01	0,24	1,83	0.066	12	
		puree	0.06	0.02	< 0.01	0,08	0,62	0.17	10	
		ketchup	0.02	0.01	< 0.01	0,03	0,23	0.76	18	
		juice	0.06	0.02	< 0.01	0,08	0,62	0.54	33	
		canned	0.02	< 0.01	< 0.01	0,02	0,15	1.4	21	
Campsas; Tarn et Garonne, S. France, 2003 (var. Quest)	2x 0.10 kg ai/hL DAT = 3	RAC	0.41	0.02	< 0.01	0,43		-		Brice *et al.*, 2004: PP62/1392 trial AF/7363/SY/1
		washed	0.31	0.02	< 0.01	0,33	0,77	1.0	77	
		wash water [d]	5.3 [c]	0.66 [c]	-	6,00	13,9	-		
		wet pomace	0.61	0.04	0.02	0,65	1,51	0.27	41	
		raw Juice	0.45	0.03	0.02	0,48	1,12	0.68	76	
		juice	0.34	0.03	0.02	0,37	0,86	0.68	59	
		puree	0.94	0.06	0.03	1,00	2,33	0.37	86	
		blanch water [d]	31 [c]	2.8 [c]	-	33,97	78,8	-		
			0.35	0.02	0.01	0,37	0,86	0.086	7	
		peels	0.41	0.03	0.01	0,44	1,02	0.90	92	
		peeled	0.37	0.02	0.01	0,39	0,91	0.81	73	
		canned								
		RAC	0.35	0.02	< 0.01	0,37		-		
		washed	0.47	0.03	0.02	0,50	1,35	1.0	135	
		juice	0.53	0.04	0.02	0,57	1,54	0.69	106	
		puree	0.76	0.05	0.02	0,81	2,19	0.35	77	
		canned	0.47	0.04	0.02	0,51	1,38	0.79	109	
		RAC	0.44	0.03	0.01	0,47		-		
		washed	0.25	0.03	0.02	0,28	0,60	1.0	60	
		juice	0.31	0.02	0.01	0,33	0,70	0.71	50	
		puree	0.66	0.04	0.02	0,70	1,49	0.40	60	
		canned	0.49	0.02	< 0.01	0,51	1,08	0.80	87	

Location, year, (variety)	Treat-ment	Commodity	parent mg/kg	R34836 mg/kg (a)	R238177mg/kg	Total mg/kg (b)	PF	MF	%T	reference
		RAC	0.54	0.02	0.01	0,56		-		
		washed	0.32	0.03	0.01	0,35	0,63	1.0	63	
		juice	0.25	0.03	0.01	0,28	0,50	0.73	37	
		puree	0.33	0.03	0.01	0,36	0,64	0.45	29	
		canned	0.35	0.02	0.01	0,37	0,66	0.80	53	

- not analysed or not applicable

PF processing factor is total residue in the processed commodity : total residue in the RAC. MF mass fraction is mass of processed product (corrected for subfractionation) : mass of starting material (RAC)

%T percentage transference of residues is PF x MF x 100%.

a Residues of R34885 (determined as R34836) and R34836

b Total pirimicarb residue = pirimicarb + 1.06x R34836 Because residues of the demethyl metabolites did not generally contribute significantly to the total residue, they are only included in the total where they were reported at levels above the LoQ

c Value in washing water given as µg/L. R34836 in wash water does not include R34885.

d For washing water, the percentage transference is calculated from absolute residue in water: absolute residue in RAC x 100%. The absolute amount of residue in the water (in mg) is calculated from A µg/L residue in water x B L water / 1000. The absolute residue in the RAC (in mg) is calculated from C mg/kg residue in RAC x D kg RAC.

Processing studies on Brassica vegetables (head cabbage, Brussels sprouts)

The effect of boiling was studied for head cabbage and Brussels sprouts (Edwards *et al*., 1976: PP62/0523). Brussels sprouts were treated with a 500 WP formulation with 5 applications of 0.56 kg ai/ha. Head cabbages were treated with one application of a 500 DG formulation at a rate of 0.025 kg ai/hL. Samples were harvested on the same day as the last treatment (DAT=0).

Samples of Brussels sprouts, head cabbage and wash water were analysed for parent, R34885, and R34836 using GC-NPD method PPRAM 15/1. The reported LOQ was 0.01 mg/kg for each analyte.

The results of the processing study are summarised in Table 111.

Table 111. Residues of pirimicarb after processing of Brussels sprouts and cabbage.

Location, year, (variety)	Treat-ment	Commodity	parent mg/kg	R34836 mg/kg (a)	R238177mg/kg	Total mg/kg (b)	PF	MF	%T	reference
1976, (Brussels sprouts)	5x 0.56 kg ai/ha DAT = 0	RAC Boiled sprouts Boil water	2.1 0.83 1.2	0.51 0.24 0.26	- - -	2,64 1,08 1,48	 0,41 0,56			Edwards *et al*., 1976: PP62/0523
1976, (Cabbage)	1x 0.025 kg ai/hL DAT = 0	RAC Boiled cabb Boil water	11 3.5 5.9	0.32 0.15 0.28	- - -	11,34 3,66 6,20	 0,32 0,55			Edwards *et al*., 1976: PP62/0523

- not analysed or not applicable

PF processing factor is total residue in the processed commodity : total residue in the RAC.

MF mass fraction is mass of processed product (corrected for subfractionation) : mass of starting material (RAC) %T percentage transference of residues is PF x MF x 100%.

a Residues of desmethyl formamido pirimicarb (R34885, measured as R34836)and desmethyl pirimicarb (R34836).

b Total pirimicarb residue = pirimicarb + 1.06x R34836 Because residues of the demethyl metabolites did not generally contribute significantly to the total residue, they are only included in the total where they were reported at levels above the LoQ.

Processing studies on leafy vegetables including Brassica leafy vegetables (lettuce, kale)

Study 1. The effect of washing was studied for lettuce (Edwards *et al*., 1976: PP62/0523). Lettuce was treated with a 500 DG formulation with 1 application of 0.025 kg ai/hL. Samples were harvested on the same day as the treatment (DAT=0).

Samples of lettuce and wash water were analysed for parent, R34885 and R34836 using GC-NPD method PPRAM 15/1. The reported LOQ was 0.01 mg/kg for each analyte.

The results of the processing study are summarised in Table 112. Results were not corrected for concurrent method recoveries (78–117%) nor for matrix interferences (< LOQ for parent, 0.06–0.17 mg/kg for demethylpirimicarb).

Study 2. Curly kale from two supervised residue trials in the UK were used for a processing study (McGill *et al*., 2003: PP62/1290 and Simmons, 2006). Pirimicarb was applied as a 500 WG formulation, 2 times at a rate of 0.38 kg ai/ha with a spray interval of 12 days. Samples (leaves and petioles) were taken and stored for 1 day at ambient temperatures and 4–5 days at 2–5 °C until processed into boiled and steamed kale following typical domestic cooking practices.

Samples were stored at -16 °C or lower for a maximum of 10 months. All samples were analysed for parent, R3488, R34836 and R238177. The reported LOQ was 0.01 mg/kg for each analyte.

The results of the processing study are summarised in Table 112. Results were not corrected for mean concurrent method recoveries (89–103%), nor for matrix interferences (< LOQ).

Study 3. Curly kale from a supervised residue trial in the UK was used for a processing study (Brice *et al*.: PP62/1450). Pirimicarb was applied as a 500 WG formulation, twice at a rate of 0.5 kg ai/ha with a spray interval of 8 days. Samples were taken 3 days after the final application and stored for 1 day at ambient temperatures and 2 days at 1–5 °C until processed into boiled and steamed kale following typical domestic cooking practices.

Samples were stored at -16 °C, or lower, for a maximum of 7 days and were analysed for parent, R34885, R34836 and R238177. The reported LOQ was 0.01 mg/kg for each analyte.

The results of the processing study are summarised in Table 112. Results were not corrected for mean concurrent method recoveries (88–92%), nor for matrix interferences (< LOQ).

Remark: The sum of all leaf fractions used for processing (weights before processing and washing) is larger (141%, 115%) than the initial weight of the leaves available.

Table 112 Residues of pirimicarb after processing of lettuce and kale.

Location, year, (variety)	Treat-ment	Commodity	parent mg/kg	R34836 mg/kg (a)	R238177mg/kg	Total mg/kg (b)	PF	MF	%T	reference
1976, (lettuce)	1x 0.025 kg ai/hL DAT = 0	RAC washed lett wash water	16 8.9 5.5	0.96 0.58 < 0.6	- - -	17,02 9,51 5,50	 0,56 0,32	- 1.0 -	 56	Edwards *et al*., 1976: PP62/0523
Chipping Campden, Gloucester-shire, UK, 2000 (kale, Winterbor)	2x 0.38 kg ai/ha; DAT = 4	RAC stalk/ribs wash water [e] washed leaves boil water [e] boiled kale steam water [e] steamed kale	0.48 [c] 0.02 6 [d] 0.48 60 [d] 0.10 10 [d] 0.42	0.82 [c] 0.02 < 10 [d] 0.78 100 [d] 0.12 20 [d] 0.61	< 0.01 < 0.01 < 1 [d] < 0.01 < 1 [d] < 0.01 < 1 [c] < 0.01	1,35 0,04 6,00 1,31 166,0 0,23 31,20 1,07	 0,03 4,45 0,97 123 0,17 23,1 0,79	 0.44 - 0.56 - 0.58 - -	 1 54 10	McGill *et al*., 2003: PP62/1290 trial GB05-00-S181

Location, year, (variety)	Treat-ment	Commodity	parent mg/kg	R34836 mg/kg (a)	R238177mg/kg	Total mg/kg (b)	PF	MF	%T	reference
Bretforton, Worcester-shire, UK, 2000 (kale, Winterbor)	2x 0.38 kg ai/ha; DAT = 4	RAC	0.28 [c]	0.57 [c]	< 0.01	0,88		-		McGill *et al*., 2003: PP62/1290 trial GB05-00-S182
		stalk/ribs	0.03	0.02	< 0.01	0,05	0,06	0.40	2	
		wash water [e]	2 [d]	< 10 [d]	< 1 [d]	2,00	2,26	-		
		washed leaves	0.20	0.40	< 0.01	0,62	0,71	0.60	42	
			20 [d]	15 [d]	< 1 [d]	35,90	40,6	-		
		boil water [e]	0.05	0.07	< 0.01	0,12	0,14	0.40	6	
		boiled kale	10 [d]	35 [d]	< 1 [d]	47,10	53,3	-		
		steam water [e]	0.15	0.29	< 0.01	0,46	0,52	-		
Moulton Marsh, Spalding, Lincoln-shire, UK, 2004, (kale, Reflex)	2x 0.50 kg ai/ha DAT = 3	RAC	0.84	2.9	< 0.01	3,91		-		Brice *et al*., 2004: PP62/1450 trial AF/8042/ SY/1
		washed leaves	0.75	2.8	< 0.01	3,72	0,95	-		
		boiled kale	0.19	0.42	< 0.01	0,64	0,16	-		
		steamed kale	1.0	3.1	< 0.01	4,29	1,10	-		
		RAC	1.3	3.6	< 0.01	5,12		-		
		washed leaves	0.86	3.2	< 0.01	4,25	0,83	-		
		boiled kale	0.18	0.44	< 0.01	0,65	0,13	-		
		steamed kale	0.88	2.6	< 0.01	3,64	0,71	-		

- not analysed or not applicable

PF processing factor is total residue in the processed commodity : total residue in the RAC.

MF mass fraction is mass of processed product (corrected for subfractionation) : mass of starting material (RAC)

%T percentage transference of residues is PF x MF x 100%.

a Residues of R34885 (determined as R34836) and R34836

b Total pirimicarb residue = pirimicarb + 1.06x R34836 Because residues of the demethyl metabolites did not generally contribute significantly to the total residue, they are only included in the total where they were reported at levels above the LoQ

b Value for kale differs from values in the residue trials section, because samples were measured again prior to cooking

d Value in washing water given as µg/L, values are mean of two analytical portions.

e For washing water, the percentage transference is calculated from absolute residue in water: absolute residue in RAC x 100%. The absolute amount of residue in the water (in mg) is calculated from A µg/L residue in water x B L water / 1000. The absolute residue in the RAC (in mg) is calculated from C mg/kg residue in RAC x D kg RAC.

Processing studies on root and tuber vegetables (potato)

Potatoes from a supervised residue trial in the USA were used for a processing study (Harradine and Barnes, 1996: PP62/0521). Pirimicarb was applied as a 500 DF formulation 4 times at a rate of 1.85 kg ai/ha with a spray interval of 8 days. Samples were taken 14 days after the final application and stored for 1 day at 7 °C until processed processed into peeled potatoes, wet peel, dry peels, chips and flakes.

Samples were stored at -18 °C or lower for a maximum of 7 months. All samples were analysed for parent, R34885, R34836 and R238177. The reported LOQ was 0.01 mg/kg for each analyte.

Because no residues were found in the raw agricultural commodity (< 0.01 mg/kg for each analyte), processing factors and percent transferred (%T) residues could not be calculated from this study. Results were therefore not summarized.

Processing studies on cereal grains (barley)

Barley from a supervised residue trials in the UK was used for a processing study (Brice *et al*., 2004: PP62/1391). Pirimicarb was applied as a 500 WG formulation twice at a rate of 0.75 kg ai/ha with a spray interval of 14 days. Samples were taken 21 days after the final application, stored for 78–113 days at ambient temperatures until processed into threshed grains, beer and pearl barley.

Samples were stored at -18 °C or lower for a maximum of 257 days. Extracts were stored at 4 °C for a maximum of 5 days. All samples were analysed for parent, R34885, R34836 and R238177. The reported LOQ was 0.01 mg/kg for each analyte.

The results of the processing study are summarised in Table 113. Results were not corrected for mean concurrent method recoveries (79–101%) nor for matrix interferences (< LOQ).

Table 113. Residues of pirimicarb after processing of barley.

Location, year, (variety)	Treat-ment	Commodity	parent mg/kg	R34836 mg/kg (a)	R23817 7mg/kg	Total mg/kg (b)	PF	MF	%T	reference
Wilson, Melbourne, Derbyshire,UK, 2003, (winter barley, var Pearl)	2x 0.750 kg ai/ha DAT = 21	RAC 2.5 mm grains dried malt sprouts malt spent grain sweet wort cooked wort spent hops young beer spent yeast beer pearl barley abrasion	0.10 0.09 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.03	0.05 0.03 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.15 0.12 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.05	0.8 < 0.07 < 0.07 < 0.07 < 0.07 < 0.07 < 0.07 < 0.07 < 0.07 < 0.07 < 0.07 0.33	- 0.93 0.78 0.03 0.75 0.55 5.24.6 - 3.8 - 3.6 0.69 0.33	 74 5 < 1 5 4 35 32 27 25 5 11	Brice at al., 2004: PP62/1391 trial AF/7360/SY/1

- not analysed or not applicable

PF processing factor is total residue in the processed commodity : total residue in the RAC.

MF mass fraction is mass of processed product (corrected for subfractionation) : mass of starting material (RAC)

%T percentage transference of residues is PF x MF x 100%.

a Residues of R34885 (determined as R34836) and R34836

b Total pirimicarb residue = pirimicarb + 1.06x R34836. Because residues of the demethyl metabolites did not generally contribute significantly to the total residue, they are only included in the total where they were reported at levels above the LoQ

In the table below, the relevant processing factors are summarised.

Commodity	Processing factors	Processing factor (median or best estimate)
Washed apples	0.66, 0.79, 0.37, 1.01, 2.41	0.79
Apple peel	2.78	
Wet apple pomace	1.66	
Dry apple pomace	5.32, 5.01, 5.54, 7.65	5.43
Apple juice	0.50, 0.74, 0.75, 1.00	0.75
Apple sauce	0.20, 0.50, 0.50, 1.00	0.50
Washed plums	1.04, 0.89, 0.87, 0.76	0.88
Prunes	1.69, 2.82, 2.07, 1.92	2.00
Washed tomatoes	0.92, 0.77, 1.35, 0.60, 0.63	0.77
Tomato puree	0.62, 2.33, 2.19, 1.49, 0.64	1.49
Tomato juice	0.62, 0.86, 1.54, 0.70, 0.50	0.70
Boiled Brussels sprouts	0.41	0.41
Boiled head cabbage	0.32	0.32
Washed lettuce	0.56	0.56
Boiled kale	0.17, 0.14, 0.16, 0.13	0.15
Steamed kale	0.79, 0.52, 1.1, 0.71	0.75
Beer	< 0.07	< 0.07

RESIDUES IN ANIMAL COMMODITIES

Direct animal treatments

Not applicable, because pirimicarb is not registered for use on animals.

Farm animal feeding studies

The Meeting received information on feeding studies with lactating cows and laying hens.

Cattle feeding studies

A residue transfer study in livestock (Edwards *et al*., 1978: PP62/0537) was conducted with 4 groups of 3 Friesian cows that were fed for 28 or 29 days with diets containing pirimicarb. Actual pirimicarb levels corresponded to actual feeding levels of 0, 24, 71 and 235 ppm.

One cow from each group was slaughtered on day 28 (A2, B4, C8, D30) and one cow on day 29 (A1, B5, C9, D37), each within 24 hours of the final dose. The remaining cow from each group (A3, B6, C7, D70) was maintained on a control diet for a further 7 days before slaughter. Milk was collected in themorning and afternoon at 2–3 day intervals throughout the study. Liver, kidney, muscle, fat (subcutaneous, peritoneal) were taken for analysis.

Pirimicarb and its carbamate metabolites R34836 and R34885 were analysed by method PPRAM 38. The reported LOQ was 0.005 mg/kg for milk or 0.01 mg/kg for tissues for each analyte. Milk samples from control animals were < 0.005 mg/kg for each analyte, except for the day 3 milk, where a value of 0.01 mg/kg was found for parent and day 17 and day 26 milk, where a value of 0.005 mg/kg was found for R34836. Tissue samples from control animals were < 0.01 mg/kg for each analyte, except for muscle where a value of 0.01 mg/kg was found for parent.

Results in milk and tissues are shown in Tables 114–5. No parent was found at any of the feeding levels (< 0.04 mg/kg). R34386 (including R34855) was only found at the highest feeding level (235 ppm) in the range < 0.02–0.088 mg/kg. Residues did not accumulate and declined rapidly when pirimicarb feeding ceased. No parent and no R34836 (including R34855) were found at any of the feeding levels in kidney and liver (< 0.01 mg/kg). Parent and metabolite R34386 (including R34855) were only occasionally found in muscle or fat at levels up to 0.02 mg/kg.

Table 114. Residues (mg/kg)[a] in milk (bulked AM and PM) from cows fed with pirimicarb for 28–29 days.

		235 ppm cow reference numbers				71 ppm cow reference numbers				24 ppm cow reference numbers			
Day	Analyte	A1	A2	A3	Mean	B4	B5	B6	Mean	C7	C8	C9	Mean
3	parent[e]	0.009	0.025	0.007	0.014	<	<	<	<	<	<	<	<
	R34836[e]	0.059	0.088	0.064	0.070	0.005	0.005	0.010	0.007	<	<	<	<
	Total												
5	parent[e]	0.006	0.026[b]	0.007	0.013	<	<	<	<	<	<[b]	<	<
	R34836[e]	0.038	0.075[b]	0.047	0.053	0.006	0.005	0.011	0.007	<	0.014[b]	<	0.008
	Total												
8	parent[e]	<	0.013	0.009	0.009	<	<	<	<	<[b]	<	<	<
	R34836[e]	0.023	0.044	0.073	0.047	0.006	<	<	0.005	0.006[b]	<	<	0.005
	Total												
10	parent[e]	<	0.016	0.005	0.009	<	<	<	<	<	<	<	<
	R34836[e]	0.040	0.064	0.062	0.055	<	<	0.009	0.006	<	0.005	<	0.005
	Total												
12	parent[e]	0.005	0.011	0.005[c]	0.007	<[b]	<	<	<	<	<	<	<

		235 ppm cow reference numbers				71 ppm cow reference numbers				24 ppm cow reference numbers			
Day	Analyte	A1	A2	A3	Mean	B4	B5	B6	Mean	C7	C8	C9	Mean
	R34836[e]	0.053	0.028	0.062[c]	0.048	0.010[b]	<	0.009	0.008	<	<	<	<
	Total												
15	parent[e]	<	0.009	<	0.006	<	<	<	<	<	<	<	<
	R34836[e]	0.038	0.036	0.059	0.044	0.010	<	0.007	0.007	<	<	<	<
	Total												
17	parent[e]	0.035	0.015	0.005	0.018	<	<	<	<	<	<	<	<
	R34836[e]	0.073	0.063	0.074	0.070	0.010	<	0.009	0.008	<	<	<	<
	Total												
24	parent[e]	0.015	0.036	0.006	0.019	<	<	<	<	<	<	<	<
	R34836[e]	0.069	0.054	0.058	0.060	0.009	<	0.016	0.010	<	<	<	<
	Total												
26	parent[e]	0.014	0.021	0.009	0.015	<	<	<	<	<	<	<	<
	R34836[e]	0.052	0.037	0.048	0.046	0.012	<	0.011	0.009	<	<	<	<
	Total												
29	parent[e]	0.006	0.012[d]	<	0.008	<[d]	<	<	<	<	<[d]	<	<
	R34836[e]	0.045	0.065[d]	0.011	0.040	0.007[d]	0.005	0.012	0.008	<	<[d]	<	<
	Total												
Pirimicarb feeding ceased													
31	parent[e]	-	-	<	-	-	-	<	-	<	-	-	-
	R34836[e]	-	-	<	-	-	-	<	-	<	-	-	-
	Total	-	-		-	-	-		-		-	-	-
33	parent[e]	-	-	<	-	-	-	<	-	<	-	-	-
	R34836[e]	-	-	<	-	-	-	<	-	<	-	-	-
	Total	-	-		-	-	-		-		-	-	-
36	parent[e]	-	-	<[b]	-	-	-	<	-	<	-	-	-
	R34836[e]	-	-	0.007[b]	-	-	-	<	-	<	-	-	-
	Total	-	-		-	-	-		-		-	-	-

- not applicable, cows were slaughtered at day 28 or 29

< < LOQ (0.005 mg/kg for each analyte), see also note e R34836 = R34836 + R34885 (measured as R34836), expressed as R34836

a Results were corrected for concurrent method recovery, uncorrected results are not available.

b Results from 2-3 replicate analyses.

c PM sample missing.

d AM sample not available, due to slaughter of cows A2, B4, C8 on day 28

e Because of matrix interferences the valid LOQ must be increased to 0.04 mg/kg for parent and to 0.02 mg/kg for R34836.

Table 115. Residues (mg/kg)[a] in tissues from cows fed with pirimicarb for 28–29 days.

Matrix	Analyte	235 ppm cow reference numbers				71 ppm cow reference numbers				24 ppm cow reference numbers			
		A1	A2	A3	Mean	B4	B5	B6	Mean	C7	C8	C9	Mean
liver	parent	<	<	<	<	<	<	<	<	<	<	<	<
	R34836	<	<	<	<	<	<	<	<	<	<	<	<
	Total												
kidney	parent	<	<	<	<	<	<	<	<	<	<	<	<
	R34836	<	<	<	<	<	<	<	<	<	<	<	<
	Total												
muscle cardiac	parent[c]	<	<	<	<	<	<	<	<	<	<	<	<
	R34836	<	<	<	<	<	<	<	<	<	<	<	<
	Total												
muscle adductor	parent[c]	<[b]	<[b]	<	<	<	<[b]	<[b]	<	0.01[b]	<	0.01[b]	0.010
	R34836	0.013[b]	0.01[b]	<	0.011	<	0.01[b]	0.01[b]	0.010	<[b]	<	<[b]	<
	Total												
muscle pectoral	parent[c]	<	0.020[b]	<	0.013	<	<	<	<	<	<	<	<
	R34836	<	<[b]	<	<	<	<	<	<	<	<	<	<
	Total												

Matrix	Analyte	235 ppm cow reference numbers				71 ppm cow reference numbers				24 ppm cow reference numbers			
		A1	A2	A3	Mean	B4	B5	B6	Mean	C7	C8	C9	Mean
fat subcutaneous	parent	<	0.013[b]	<	0.011	0.020[b]	<	<	0.013	<	<	<	<
	R34836	<	<[b]	<	<	<[b]	<	<	<	<	<	<	<
	Total												
fat peritoneal	parent	<	0.01[b]	<	0.01	<	<	<	<	<	0.01[b]	<	0.010
	R34836	<	<[b]	0.01	0.01	<	<	<	<	<	<	<	<
	Total												

< < LOQ (0.01 mg/kg for each analyte), see also note c R34836 = R34836 + R34885 (measured as R34836), expressed as mg/kg R34836

a Results were corrected for concurrent method recovery, uncorrected results are not available.

b Results from 2-3 replicate analyses.

c Because of matrix interferences the valid LOQ for muscle must be increased to 0.04 mg/kg for parent

Poultry feeding study

A residue transfer study in laying hens (Ross *et al*., 1978: PP62/0598 and Edwards and Dick, 1978: PP62/0536) was conducted with four groups of 40 laying hens + four cockerels. The hens were fed for up to 28 days with basal layers diet containing pirimicarb at nominal levels of 0, 2, 6 and 20 mg/kg feed, followed by a recovery period of 14 days on untreated feed.

Actual feed levels were considerably lower than the nominal levels. At the beginning of the trial, the prepared feed contained pirimicarb and a small fraction of R34836. Over the duration of the trials no change in levels of pirimicarb or metabolite in the diet was observed. Levels were 0.03, 1.3, 4.2, 13 ppm parent and 0.05, 0.22, 0.36, 1.2 ppm R34836. Low residues of pirimicarb (0.03 mg/kg) and R34836 (0.05 mg/kg) were also found in the control diet samples, resulting in actual feeding levels of 0.083, 1.5, 4.6 and 14.3 ppm parent eq instead of the nominal 0, 2, 6 and 20 ppm parent eq.

Eggs (10 per treatment group) were collected on days 1, 3, 7, 11, 15, 21, 25 and 27 (treatment period) and days 31, 35, 39 and 42 (post-treatment period). Eggs were separated into whites and yolks. On each day, the white and yolk samples from each group were pooled. Five hens from each group were sacrificed on days 21, 28, 35 and 42 of the trial. Muscle and skin with underlying fat were taken and the samples from each group were pooled. Liver samples were also pooled. The remaining hens from each group were used for post-mortem examinations. Feed samples, tissues and eggs (whites/yolks) were stored for 7–25 weeks at -14 °C until analysis.

Pirimicarb and its carbamate metabolites R34836 and R34885 were analysed and the reported LOQ was 0.01 mg/kg for eggs (whites/yolks) and tissues for each analyte, except for R34836 in muscle (0.02 mg/kg). Samples from control animals were < 0.01 mg/kg for each analyte, except for egg whites (where values of up to 0.02 mg/kg were found for parent and up to 0.03 mg/kg for R34836) and liver (where values of up to 0.01 mg/kg were found for R34836).

Residue levels for individual animals were not available as all the samples were pooled per group. No residues were found in the pooled samples of egg yolk and egg white (< LOQ for each analyte) at any feeding level. No residues were found in pooled composite tissue samples (muscle, skin with fat) at any feeding level (< LOQ for each analyte). Residues in pooled liver samples are shown in Table 116. Residues in liver were at or below the LOQ: < 0.01 to 0.01 mg/kg for parent and < 0.04 to 0.04 mg/kg for R34836 (including R34855).

Table 116. Residues (mg/kg)[a] in pooled liver samples from laying hens fed with pirimicarb for 21-28 days.

ppm parent eq	Treatment period						Post-treatment period					
	21 Day			28 D			35 D			42 D		
	parent	R3483 6[c]	Total	parent	R3483 6[c]	Total	parent	R3483 6[c]	Total	parent	R3483 6[c]	Total
0.083	< 0.01	< 0.01	< 0.01	< 0.01	0.01	0.02	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
1.5	< 0.01	0.02 [c]	0.03	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01

ppm parent eq	Treatment period						Post-treatment period					
	21 Day			28 D			35 D			42 D		
	parent	R34836[c]	Total	parent	R34836[c]	Total	parent	R34836[c]	Total	parent	R34836[c]	Total
					[c]			[c]			[c]	
4.6	< 0.01	0.02 [c]	0.03	< 0.01	< 0.01 [c]	< 0.01	< 0.01	0.02 [c]	0.03	< 0.01	< 0.05 [b]	< 0.01
14.3	< 0.01	0.04	0.05	< 0.01	0.02 [c]	0.03	0.01	0.01 [c]	0.02	< 0.01	0.01 [c]	0.02

R34836 = R34836 + R34885 (measured as R34836), expressed as mg/kg R34836

a Results were corrected for concurrent method recovery, uncorrected results are not available.

b. Due to high background levels a higher LOQ was set for this sample.

c Because of matrix interferences in the control sample (0.01 mg/kg), the valid LOQ needs to be increased to 0.04 mg/kg for R34836.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No data available.

NATIONAL MAXIMUM RESIDUE LIMITS

No longer required.

APPRAISAL

Residue and analytical aspects of pirimicarb were evaluated by the JMPR in 1976, 1978, 1979, 1981 and 1985. The compound was listed in the Periodic Re-Evaluation Programe at the 37th Session of the CCPR for periodic review by the 2006 JMPR. The toxicological review was conducted in 2004, when an ADI of 0–0.02 mg/kg bw and an ARfD of 0.1 mg/kg bw were established.

The Meeting received a full data package including animal and plant metabolism studies (goats, hens, apple trees, lettuce, potatoes and wheat), crop rotational studies, hydrolysis and photolysis studies in water, information on analytical methods, supervised residue trial data from use as a foliar spray on a range of fruit, vegetable, cereal and oil seed crops, processing studies and livestock feeding studies. GAP information was also submitted by the Netherlands.

Animal metabolism

The Meeting received information on the fate of orally dosed pirimicarb in the lactating goat and in laying hens. Experiments were carried out with pirimicarb ^{14}C labelled at the pyrimidinyl-2 position. Metabolism in laboratory animals (rats) was summarized and evaluated by the WHO panel of the JMPR in 2004.

Kinetic studies in rats have demonstrated that pirimicarb administered orally to male and female rats is rapidly and extensively absorbed (> 70% of the administered dose) and widely distributed. Radioactivity from [^{14}C]pyrimidinyl-labelled pirimicarb was excreted predominantly in the urine, while radioactivity from [^{14}C]carbamoyl-labelled pirimicarb was excreted predominantly in expired air. Tissue retention of radioactivity was low. Pirimicarb was extensively metabolized, giving rise to 24 metabolites, 17 of which were identified. The main metabolic pathway involves the loss of the carbamate moiety to produce a range of substituted hydroxypyrimidines, some of which are glucuronide conjugates.

One lactating Alpine goat orally treated twice daily for five consecutive days with ^{14}C-labelled pirimicarb at a calculated dose rate of 17 ppm feed, was sacrificed approximately 21 hours after the last dose. The largest amount of radioactivity was found in the urine and faeces, which contained around 63% and 11% of the total dose, respectively. The edible tissues (liver, kidney,

muscle and fat) contained 1.3%, while milk contained 0.29%. The goat was monitored for expired ^{14}C-volatiles, but none were found. The overall recovery of the radioactivity was 77.3%.

After 5 days of treatment, the highest concentration of radioactive residues was found in the liver (0.46 mg/kg eq). Kidney, muscle and fat contained 0.34, 0.07, and 0.02 mg/kg eq, respectively. The total residue in the pm milk was higher than the residue in the am milk, indicating that a plateau was reached within 24 hours after dosing. Residue levels in milk during the dosing period were on average 0.054 mg/kg eq in the pm milk, while residue levels in the am milk were on average 0.03 mg/kg eq.

Neither pirimicarb nor any carbamate containing metabolites were detected in goat tissues or milk. The metabolites identified in liver, kidney and muscle were the hydroxypyrimidines R31805, R34865 and R31680. Individually these metabolites did not exceed 0.03 mg/kg eq in any tissue. The same three hydroxypyrimidine metabolites, plus a fourth closely related compound of the same structural type, were identified at very low levels (up to 0.01 mg/kg) in milk. The major part of the radioactivity remained unidentified: 65% in liver, 75% in kidney, 68% in muscle and 34% in milk. Each individual compound was < 10% of the total recovered radioactivity, and it is very unlikely that they comprised significant levels of any carbamate containing metabolite.

Ten white leghorn laying hens were orally dosed once daily for ten consecutive days with ^{14}C-labelled pirimicarb at a calculated dose rate of 7.7 ppm feed. The hens were sacrificed approximately 21–24 hours after the final dose. The largest amount of radioactivity was found in the excreta, which contained around 88% of the total dose. The edible tissues (liver, kidney, muscle and fat) contained around 0.6%, while eggs contained around 0.3%. The hens were monitored for expired ^{14}C-volatiles, but none were found. The overall recovery of the radioactivity was around 89%.

After 10 days of treatment, the highest concentration of radioactive residues was found in the liver (0.3 mg/kg eq). Kidney, breast muscle and fat contained 0.11, 0.14, and 0.02 mg/kg eq, respectively. The residue levels in egg yolk and white reached a plateau at day 6 and day 3, respectively. The residue levels at the plateau level were on average 0.13 mg/kg eq (range 0.11–0.15 mg/kg eq) in egg yolks and on average 0.08 mg/kg eq (range 0.065–0.088 mg/kg eq) in egg whites.

Neither pirimicarb nor any carbamate containing metabolites were found in hen tissues or eggs. The metabolites identified were the hydroxypyrimidines R31805, R34865 and R31680. In tissues, only compound R31680 exceeded 0.01 mg/kg eq, whilst in eggs only compound R34865 exceeded 0.01 mg/kg eq. A substantial part of the radioactivity remained unidentified: 64% in liver, 24% in kidney, 24% in breast muscle, 34% in thigh muscle, 17% in fat, 27% in egg white and 49% in egg yolk. It is however very unlikely that these unidentified fractions contain significant levels of any carbamate.

In conclusion, the metabolism of pirimicarb in farm animals was similar to that in laboratory animals. Goats and laying hens dosed with pirimicarb quickly detoxify the compound. Neither parent nor any carbamate containing metabolites were found in edible tissues, milk and eggs.

Plant metabolism

The Meeting received new information on the fate of pirimicarb after foliar treatment of apple trees, lettuce, potatoes and wheat which superseded the old metabolism studies carried out during the 1970s and early 1980s. Experiments were carried out with pirimicarb ^{14}C labelled at the pyrimidinyl-2 position. In all experiments an extensive set of carbamates, hydroxypyrimidines and guanidines was used as reference compounds.

Two apple trees, grown in pots in a caged area open to normal weather conditions in the UK, were sprayed with a WG formulation containing ^{14}C-labelled pirimicarb. The first tree was treated three times with 1.2 kg ai/ha and the second tree three times with 1.1 kg ai/ha. The first interval was 70 days, the second 46 days. Apples were harvested at 21 days after treatment (DAT). The total radioactive residues in the apples were 2.4 mg/kg eq and 1.7 mg/kg eq for the first and second tree, respectively. The major residue found in apples was parent pirimicarb (30% TRR). Other carbamates,

hydroxypyrimidines, N,N-dimethylguanidine and urea were found at low levels (< 2% TRR). The polar residue (total 51% TRR) did not contain any significant levels of carbamates and comprised many different components including compounds with basic, strongly basic and neutral or acidic properties.

Lettuce plants were glasshouse grown in pots of sandy loam soil in the UK. Lettuce foliage was sprayed with a WG formulation containing ^{14}C- labelled pirimicarb. The first pot was treated three times with 0.255 kg ai/ha and the second pot three times with 0.265 kg ai/h at intervals of 7 days starting with 8 week old plants. The heads of mature lettuce plants were collected at 3 DAT (first pot) and 7 DAT (second pot). Total radioactive residues in lettuce leaves were 14 and 12 mg/kg eq at 3 and 7 days post-treatment, respectively. In the samples, 91% and 88% TRR could be extracted with MeOH, respectively. Pirimicarb and the carbamate metabolite demethyl pirimicarb together accounted for the majority of the radioactivity (68.7% and 59.3%). Three other carbamates and two hydroxypyrimidines were identified at much lower levels (≤ 2%).

Potato plants were grown in pots in sandy loam soil in a caged area open to normal weather conditions in the UK. Potato foliage was sprayed with a WG formulation containing ^{14}C- labelled pirimicarb. Two separate experiments were conducted: a low dose where 0.78 kg ai/ha was applied twice at an interval of 13 days, and a high dose where four applications were made at 2.8 kg ai/ha with an interval of 7, 6 and 8 days. The first spray application was 108 days after planting. Potato tubers were harvested at 17 and 18 days post-treatment from the low and high dose trials, respectively. The total radioactive residues found in tubers were 0.04 and 0.23 mg/kg eq for the low and high dose experiment, respectively. In the low dose experiment, 95.1% of the total recovered radioactivity could be extracted. Neither parent nor any metabolites containing the carbamate moiety were found. The majority of the residue (90.2%) was comprised of highly polar water-soluble components, e.g., 1,1-dimethylguanidine, methylguanidine, of which no single metabolite exceeded 0.01 mg/kg eq. In the high dose experiment, 95.0% of the total recovered radioactivity could be extracted. Trace amounts of parent (1.7%), carbamates demethyl pirimicarb (1.0%) and demethyl formamido pirimicarb (0.7%) and a hydroxypyrimidine R31805 (1.1%) were identified. The principal metabolites, identified in the polar water-soluble fractions (81.6%), were N,N-dimethylguanidine (15.8%) and N-methylguanidine (3.5%).

In the UK field grown wheat was transplanted into a circular tub placed in a caged area open to normal weather conditions. Transplanting was carried out 55 days prior to the first treatment. Wheat foliage was sprayed with a WG formulation containing ^{14}C- labelled pirimicarb. The crop was sprayed twice at rates of 0.28 and 0.29 kg ai/ha, respectively. The first treatment was at growth stage BBCH 70 - 80 (just after completion of the flowering stage) and the second treatment was 35 days later. Wheat was collected at 14 days post-treatment, with grain heads separated from the straw. Total radioactive residues found in wheat straw and grain were 16 mg/kg eq and 0.72 mg/kg eq, respectively. The metabolism of pirimicarb in straw and grain is very similar. For grain and straw 86.6% and 80.1% of the total recovered radioactivity was extracted, respectively. Pirimicarb itself was the major residue (25.2% in grain, 13.4% in straw). Other identified compounds were demethyl pirimicarb (2.8% in grain, 4.4% in straw) and demethyl formamido pirimicarb (1.3% in grain, 1.7% in straw) and hydroxypyrimidine R31805 (1.6% in grain, 1.2% in straw). The remaining radioactivity comprised highly polar, water soluble constituents, including guanidines.

In conclusion, studies on the nature of residues in primary crops have demonstrated that pirimicarb undergoes very extensive metabolism resulting in a diverse range of metabolites. The early stages of metabolism, as demonstrated by the lettuce study, but also exhibited in other crops studies, involve modification of the dimethylamino moiety on position 2 of the pyrimidine ring and loss of the carbamate moiety. Loss of the carbamate moiety produces hydroxypyrimidine metabolites. The main metabolic route involves degradation of pirimicarb to demethyl pirimicarb and further degradation of both these compounds to the corresponding hydroxypyrimidines R31805 and R34865.

Further degradation of the hydroxypyrimidines takes place, resulting in ring opening of the pyrimidine and further degradation to form low molecular weight polar molecules such as the guanidines.

Although the metabolism of pirimicarb is qualitatively comparable to that in animals, the turnover in plants is much lower and substantial amounts of parent and carbamate-containing metabolites can still be present at harvest.

Environmental fate in soil

The Meeting received confined and field rotational crop studies.

A confined rotational crop study was undertaken to determine the accumulation and metabolic fate of ^{14}C-labelled pirimicarb under field conditions. The crops used were lettuce, radish and millet. A single application of pirimicarb at a rate of 1.48 kg ai/ha was made to bare soil (sandy loam) in Visalia, California, USA. Crops were planted into the soil 29, 61, 119 days after treatment and were harvested at maturity. Millet was also harvested at the forage and hay stages. The total radioactive residues in the crops declined significantly as the plantback or rotation interval increased. Millet straw had the highest residues at all rotations (5 mg/kg eq at a plantback of 29 DAT), while the lowest residue was found in the radish root (0.029 mg/kg eq at a plantback of 119 DAT). Leafy, root and small grain crops all show comparable metabolic profiles. Low levels of pirimicarb (< 0.001–11.5% of the total recovered radioactivity) and carbamate metabolites (demethyl pirimicarb 0.213–14.5%, demethyl formamido pirimicarb < 0.001–4.14% and R35140 < 0.001–5.88%) were found in some samples and levels of carbamates decreased as the rotation interval increased. Other identified compounds were hydroxypyrimidines (R31805, R34865 and R31680) and guanidine (R12378).

Two supervised field trials were carried out in the USA (Whitakers, North Carolina (NC) and Visalia, California (CA)) to determine the magnitude of residues of pirimicarb in rotational crops (millet, mustard and turnip). At each site two plots were used with a primary crop of lettuce. Pirimicarb as a WG formulation was applied on the first plot four times at 0.56 kg ai/ha with 5 day intervals. On the second plot a combination of a single application at 0.37 kg ai/ha + two applications at 0.56 kg ai/ha with a 5 and 10 day interval were made. In NC the last application was at the vegetative growth stage, in CA at the full grown vegetative growth stage. The formulation was applied as a broadcast treatment using a tractor mounted sprayer. The soil type was USDA sandy loam. The lettuce was removed and separate plots of millet, mustard and turnip were planted back at 30, 60 and 120 days after the last application. The rotated crops were sampled at normal harvest for the rotational crop.

No residues of pirimicarb or its metabolites demethyl pirimicarb, demethyl formamido pirimicarb and R238177 were measured in any of the samples from North Carolina from any of the plant back intervals (< 0.01 mg/kg, each analyte). Low pirimicarb and demethyl pirimicarb residues were measured in some of samples from the Californian trials. The highest total pirimicarb residues were found in millet forage (0.05–0.07 mg/kg eq) and mustard leaves (0.03–0.04 mg/kg eq) from the 30 day planting interval. No residues of R238177 were measured in any of the samples (< 0.01 mg/kg).

Environmental fate in water-sediment systems

The Meeting received information on the hydrolysis and photolysis of pirimicarb in water. Pirimicarb was shown to be hydrolytically stable under acidic, neutral and alkaline conditions. However, pirimicarb was rapidly degraded by photolysis in aqueous solution with DT_{50} values of 3.2 hours and 2.28 hours at pH 5 and 7, respectively. After a period equivalent to 31 hours summer sunlight, only 1.2% and 1.4% of the total applied radioactive parent remained at pH 5 and 7. The major degradation products formed are demethyl formamido pirimicarb, hydroxypyrimidine R31805 and N,N-dimethylguanidine(sulfate), which accounted for up to 17.9%, 27.8% and 14.1% of the radioactivity applied, respectively at pH 5 and 16.4%, 25.5% and 26.9% at pH 7.

Methods of analysis

The Meeting received data on analytical methods for enforcement and monitoring of pirimicarb and its carbamate metabolites (demethyl pirimicarb, demethyl formamido pirimicarb, and R238177) in plant commodities and pirimicarb and demethyl pirimicarb in animal commodities. The Meeting also received information on analytical methods used in the study reports.

Analytical methods for enforcement and monitoring

Method RAM 265 is intended for use as an enforcement-monitoring method for the determination of pirimicarb and its carbamate metabolites (demethyl pirimicarb, demethyl formamido pirimicarb, and R238177) in plant commodities. The method is also used in study reports (see below). In general, LOQs of 0.01 mg/kg can be reached. The Meeting noted that method RAM 265 is considered a special method and cannot be included in a multi-residue method because of the acid treatment required for the conversion of demethyl formamido pirimicarb into demethyl pirimicarb.

Method DFG S19 is intended for use as an enforcement-monitoring method for the determination of pirimicarb and its carbamate metabolite (demethyl pirimicarb) in animal commodities. Method DFG S19 is a published German multi-residue method and results show that pirimicarb and its metabolite can be incorporated into this existing method. The published method consists of GC technology. Newer studies use HPLC-MS/MS with the S19 method. LOQs of 0.01 mg/kg were reported for milk, muscle, kidney, liver, fat and eggs.

Analytical methods used in study reports

In the course of time, numerous analytical methods to determine pirimicarb and its metabolites have been described. They include methods PPRAM 15 (1972-1997, several versions), PPRAM 38 (1978), RAM 265 (1995-2004, several versions), RAM 277 (2000-2004), RAM 319 (2000-2002) and RAM 360 (2001). Most methods were developed for the determination of pirimicarb and its carbamate metabolites demethyl pirimicarb and demethyl formamido pirimicarb, while some versions of method RAM 265 and methods RAM 319 and RAM 360 also measure the metabolite R238177.

In general, extracts were left to stand overnight or incubated for 1 hour at 50 °C to ensure the conversion of any demethyl formamido pirimicarb into demethyl pirimicarb. After an optional clean-up step (depending on commodity) pirimicarb and demethyl pirimicarb were analysed by GC-NPD, HPLC-MS/MS (APCI, positive ion mode or with fluorescence detection). Modifications of the methods mainly concerned the clean-up and changed GC or HPLC conditions. The reported LOQ for each analyte was usually 0.01 mg/kg. In some cases the LOQ had to be raised to 0.05 mg/kg because of matrix interferences, e.g., in cabbage, fodder and straw.

Stability of residues in stored analytical samples

The Meeting received data on the stability of residues in various crops and milk.

Storage stability studies on apple, cauliflower, cabbage, cucumber, tomato, Iceberg lettuce, snap beans, potato, artichoke, asparagus, wheat grain and straw and seeds from oilseed rape fortified with a mixture of pirimicarb, demethyl pirimicarb and R238177 show that residues are stable for up to 12 months when stored at -18 °C.

The storage stability of pirimicarb residues in stonefruit, berries and other small fruits, bulb vegetables, Brussels sprouts, courgettes and melons, peppers and sweet corn, kale, turnip tops, mustard leaves, beans without pods and peas with or without pods, carrots and turnip roots, pulses (dry peas, dry broad beans) and sunflower seeds, barley (straw and grains), maize (grain, forage, fodder), and millet (grain, forage, hay, straw) was not specifically investigated but the storage stability in these commodities can be extrapolated from other crops with high water content, e.g., apples and tomatoes, and other dry crops with starch and proteins, e.g., wheat grain and seeds from oilseed rape.

Residues of pirimicarb, demethyl pirimicarb and demethyl formamido pirimicarb are stable in milk for up to 24 months when stored at -14 °C. No storage stability data are available on meat, edible offal and fat; however metabolism studies show that it is unlikely that any carbamate containing residue will occur in animal tissues.

Definition of the residue

In animals, pirimicarb is quickly detoxified and neither parent nor any other carbamate containing metabolites were found in edible tissues, milk and eggs.

Because of the lack of a better indicator molecule the Meeting agreed that parent pirimicarb should be the compound of interest in animal commodities, both for enforcement and for dietary risk assessment.

It was concluded from the low magnitude of residues in animal fat and the log P_{ow} of 1.7 of the parent that the residue is not fat-soluble.

In plants, the major residue is the parent pirimicarb. Metabolites include carbamates, hydroxypirimidines and guanidines. The only metabolites of significance were demethyl pirimicarb and demethyl formamido pirimicarb. Hydroxypyrimidines and guanidines are not of toxicological concern. The 2004 JMPR concluded that demethyl pirimicarb and demethyl formamido pirimicarb have toxicological profiles similar to that of pirimicarb itself. Metabolite (2-dimethylamino-6-hydroxymethyl-5-methylpyrimidin-4-yl dimethylcarbamate) (R238177) is the 6-hydroxymethyl metabolite of pirimicarb. The current JMPR decided that in the absence of specific data, the toxicological properties of pirimicarb itself can be assumed for this 6-hydroxymethyl metabolite.

The Meeting noted that in the residue trials, this metabolite was virtually always below the LOQ except in some trials on currants and peppers, where measurable residues were found, in one instance up to 0.08 mg/kg. The Meeting decided that the 6-hydroxymethyl metabolite does not have to be included in the residue definition for dietary risk assessment.

Definition of the residue in plant commodities for compliance with MRLs: pirimicarb.

Definition of the residue in plant commodities for estimation of dietary intake: sum of pirimicarb, demethyl pirimicarb and demethyl formamido pirimicarb, expressed as pirimicarb.

Definition of the residue in animal commodities for compliance with MRLs and estimation of dietary intake: pirimicarb.

Results of supervised trials on crops

Supervised trials were available for the use of pirimicarb as a foliar spray on the following crops: citrus (mandarins, oranges), apples, stone fruit (cherries, peaches, nectarines and plums), berry fruit (currants, gooseberries, raspberries, blackberries and strawberries), onions, brassica vegetables (cabbage, cauliflower, broccoli, Brussels sprouts and kale), cucumber, summer squash, melons, tomatoes, peppers, sweetcorn, lettuce, legumes and pulses, carrots, sugar beet, potato, globe artichoke, asparagus, cereals (barley, wheat and maize), oil seed rape and sunflower.

Trial data or relevant GAP was not submitted for alfalfa (fodder); alfalfa forage (green); celery; cotton seed; endive; leek; parsley; pecan; spinach; sweet corn (corn-on-the-cob) and watercress, for which current recommendations for maximum residue levels exist.

The Meeting agreed to withdraw its previous maximum residue level recommendations for these commodities.

Analytical methods used in the trials measured residues of pirimicarb and also the combined residues of demethyl pirimicarb (R34836) and demethylformamido pirimicarb (R34885), the latter being converted to and measured as R34836. In most trials the methods were also able to measure 2-

dimethylamino-6-hydroxymethyl-5-methylpyrimidin-4-yl dimethylcarbamate), the 6-hydroxymethyl metabolite of pirimicarb (R238177).

The Meeting agreed to use residue results for pirimicarb for the estimation of maximum residue limits and to combine the results for pirimicarb and for demethyl pirimicarb plus demethylformamido pirimicarb, expressed as pirimicarb (adjustment factor of 1.06) for the estimation of STMRs and HRs. In this appraisal, the term 'total pirimicarb residues' refers to these combined residues of pirimicarb and the listed de-methyl metabolites, expressed as pirimicarb.

The ratio of the de-methyl metabolites and parent compound varied in different crops and in some cases this may lead to STMR and/or HR values (based on the total pirimicarb residues) being established at levels higher than the estimated maximum residue level, as this is based on the parent compound only. In addition, the Meeting agreed that because residues of the demethyl metabolites did not generally contribute significantly to the total residue, they would only be included in the total where they were reported at levels above the LOQ. This approach is shown in the following example:

Pirimicarb (mg/kg)	*Demethyl pirimicarb plus demethylformamido pirimicarb (mg/kg)*	*Total pirimicarb residues, expressed as pirimicarb (mg/kg)*
< 0.01	*< 0.01*	*< 0.01*
0.1	*< 0.01*	*0.1*
0.2	*0.1 [× 1.06]*	*0.31*

Oranges, sweet, sour

The results of residue trials in Italy and Spain on oranges were made available to the Meeting.

GAP for citrus in Spain is for foliar spray applications of 0.05 kg ai/hL (PHI of 7 days). In trials from Italy and Spain, matching this GAP, pirimicarb residues in whole fruit were: 0.11, 0.11, 0.25, 0.27, 0.37 and 0.40 mg/kg (n = 6). Total pirimicarb residues in orange pulp in these trials were: < 0.01 (5) and 0.01 mg/kg.

Mandarin

The results of residue trials in Italy and Spain on mandarins were made available to the Meeting.

GAP for citrus in Spain is for foliar spray applications of 0.05 kg ai/hL (PHI of 7 days). In trials from Italy and Spain, matching Spanish GAP, pirimicarb residues in whole fruit were: 0.35, 0.68, 0.77, 0.87, 1.2, 1.2, 1.8 and 2.2 mg/kg (n = 8). Total pirimicarb residues in mandarin pulp in these trials were: < 0.01, 0.01, 0.01, 0.01, 0.02, 0.03, 0.04 and 0.08 mg/kg (n = 8).

The Meeting agreed that the data for oranges and mandarins were sufficient to support a citrus fruit commodity group maximum residue level and estimated a maximum residue level of 3 mg/kg for pirimicarb on citrus fruit and based on the mandarin data, estimated an STMR of 0.015 mg/kg and HR of 0.08 mg/kg for total pirimicarb residues in the edible portion of citrus fruit.

The Meeting also agreed to withdraw its previous recommendations of 0.5 mg/kg for oranges (Sweet and Sour) and 0.05 (*) mg/kg for citrus fruit (except oranges, Sweet, Sour) as these were being replaced by the recommendation for citrus fruit.

Apples

The results of residue trials in France, Germany, Italy, Spain and the UK on apples were made available to the Meeting.

GAP for deciduous fruit crops in Spain is for foliar spray applications of 0.05 kg ai/hL (PHI of 7 days) and in trials from France, Italy and Spain matching this GAP, pirimicarb residues in whole fruit were: 0.03, 0.05, 0.12, 0.13, 0.15, 0.15 and 0.25 mg/kg (n = 7). Total pirimicarb residues in apples these trials were: 0.03, 0.07, 0.15, 0.18, 0.19, 0.21 and 0.30 mg/kg (n = 7).

In the Netherlands, GAP for apples and pears is for up to two applications of 0.025 kg ai/hL (PHI 7 days) and in trials from France and the UK matching this GAP, residues of pirimicarb were: 0.05, 0.14, 0.15, 0.16, 0.18, 0.28, 0.3 and 0.88 mg/kg (n = 8). Total pirimicarb residues in apples these trials were: 0.05, 0.16, 0.17, 0.17, 0.2, 0.3, 0.33 and 0.91 mg/kg (n = 8).

The Meeting noted that the two residue populations were similar and agreed to use a combined data set of: 0.03, 0.05, 0.05, 0.12, 0.13, 0.14, 0.15, 0.15, 0.15, 0.16, 0.18, 0.25, 0.28, 0.3 and 0.88 mg/kg (n = 15) for pirimicarb residues in apples and 0.03, 0.05, 0.07, 0.15, 0.16, 0.17, 0.17, 0.18, 0.19, 0.2, 0.21, 0.3, 0.3, 0.33 and 0.91 mg/kg for total pirimicarb residues.

The Meeting agreed that the data on apples could be used to support a pome fruit commodity group maximum residue level and estimated a maximum residue level of 1 mg/kg for pirimicarb on pome fruit (confirming the existing recommendation) and estimated an STMR of 0.18 mg/kg and HR of 0.91 mg/kg for total pirimicarb residues in pome fruit.

Cherries

The Meeting received results of residue trials in France, Germany, Italy, Spain and the UK on cherries.

GAP for deciduous fruit crops in Spain is for foliar spray applications of 0.05 kg ai/hL (PHI of 7 days) and in trials from France, Italy and Spain matching this GAP, pirimicarb residues in whole fruit were: 0.28, 1.1, 1.2, 1.4 and 1.9 mg/kg (n = 5). Total pirimicarb residues in flesh of cherries in these trials were: 0.36, 1.3, 1.3, 1.8 and 2.1 mg/kg (n = 5).

GAP for stone fruit in the Czech Republic is for foliar spray applications of up to 0.038 kg ai/hL (PHI 7 days) and in trials from Germany, France and UK matching this GAP, pirimicarb residues in whole fruit were: 0.43, 0.69, 0.71 and 0.89 mg/kg (n = 4). Total pirimicarb residues in flesh of cherries in these trials were: 0.49, 0.78, 0.82, and 0.99 mg/kg (n = 4).

The Meeting noted that the two residue populations appeared to from similar populations and agreed to use a combined data set of: 0.28, 0.43, 0.69, 0.71, 0.89, 1.1, 1.2, 1.4 and 1.9 mg/kg (n = 9) for pirimicarb residues in cherries and 0.36, 0.49, 0.78, 0.82, 0.99, 1.3, 1.3, 1.8 and 2.1 mg/kg (n = 9) for total pirimicarb residues.

Peaches (and nectarines)

The Meeting received results of residue trials in France, Italy and Spain on peaches and nectarines.

GAP for deciduous fruit crops in Spain is for foliar spray applications of 0.05 kg ai/hL (PHI 7 days) and eight trials on peaches and four trials on nectarines from France, Italy and Spain matched this GAP.

Pirimicarb residues in nectarines were 0.09, 0.17, 0.22 and 0.36 mg/kg and total pirimicarb residues in two of these trials were 0.27 and 0.41 mg/kg in nectarine flesh. In peaches, pirimicarb residues were 0.09, 0.15, 0.22, 0.25, 0.32, 0.34 0.39 and 1.2 mg/kg and in six of these trials, total pirimicarb residues in peach flesh were 0.13, 0.29, 0.37, 0.39, 0.46 and 1.4 mg/kg.

The Meeting noted that the residues from the nectarine and peach trials were from similar populations and agreed to combine the results. Pirimicarb residues in whole fruit were: 0.09, 0.09,

0.15, 0.17, 0.22, 0.22, 0.25, 0.32, 0.34, 0.36, 0.39 and 1.2 mg/kg (n = 12). Total pirimicarb residues in flesh of peaches and nectarines in eight of these trials were: 0.13, 0.27, 0.29, 0.37, 0.39, 0.41, 0.46 and 1.4 mg/kg (n = 8).

Plums

The Meeting received results of residue trials in France, Germany, Italy, Spain and the UK on plums.

GAP for deciduous fruit crops in Spain is for foliar spray applications of 0.05 kg ai/hL (PHI 7 days) and in trials from France, Italy and Spain matching this GAP, pirimicarb residues in whole fruit were: 0.1, 0.15, 0.17, 0.29 and 0.3 mg/kg (n = 5). Total pirimicarb residues in flesh of plums in four of these trials were: 0.11, 0.19, 0.22 and 0.36 mg/kg (n = 4).

In the Netherlands, GAP for plums is for up to two applications of 0.038 kg ai/hL (PHI 7 days) and in trials from Germany and the UK matching this GAP, residues of pirimicarb were: 0.08, 0.1, 0.15 and 0.27 mg/kg (n = 4). Total pirimicarb residues in flesh of plums in these trials were: 0.09, 0.11, 0.17 and 0.30 mg/kg (n = 4).

In the Czech Republic, GAP for plums is for up to two applications of 0.038 kg ai/hL (PHI 14 days) and in trials from Germany and the UK matching this GAP, residues of pirimicarb were: 0.12, 0.20, 0.21, 0.21, 0.24, 0.28, 0.32 and 0.34 mg/kg (n = 8). Total pirimicarb residues in flesh of plums in these trials were: 0.13, 0.21, 0.24, 0.28, 0.28, 0.31, 0.37 and 0.43 mg/kg (n = 8).

The Meeting noted that the three sets of residue results were from similar populations and agreed that they could be combined. Pirimicarb residues in whole fruit were: 0.08, 0.1, 0.1, 0.12, 0.15, 0.15, 0.17, 0.20, 0.21, 0.21, 0.24, 0.27, 0.28, 0.29, 0.3, 0.32 and 0.34 mg/kg (n = 17). Total pirimicarb residues in the flesh were: 0.09, 0.11, 0.11, 0.13, 0.17, 0.19, 0.21, 0.22, 0.24, 0.28, 0.28, 0.30, 0.31, 0.36, 0.37 and 0.43 mg/kg (n = 16).

The Meeting agreed that the data on peaches, nectarines, cherries and plums could be used to support a ‘stone fruit’ commodity group maximum residue level and estimated a maximum residue level of 3 mg/kg for pirimicarb on stone fruit and based on the cherry data, estimated an STMR of 0.99 mg/kg and HR of 2.1 mg/kg for total pirimicarb residues in the flesh of stone fruit.

The Meeting also agreed to withdraw its previous maximum residue level recommendations of 0.5 mg/kg for peaches and for plums (including prunes) because they were being replaced by the maximum residue level for stone fruit.

Currants (and gooseberries)

The Meeting received results of residue trials in Germany on currants and gooseberries.

In the Netherlands, GAP for currants and gooseberries is for up to two applications of 0.025 kg ai/hL (PHI 7 days) and seven currant trials and one gooseberry trial from Germany matched this GAP.

Pirimicarb residues in currants were 0.07, 0.08, 0.09, 0.14, 0.18, 0.23 and 0.28 mg/kg (n = 7) and in gooseberries, the pirimicarb residue was 0.13 mg/kg. The Meeting agreed to combine the currant and gooseberry results as mutually supporting data. The combined data set for currants and gooseberries were: 0.07, 0.08, 0.09, 0.13, 0.14, 0.18, 0.23 and 0.28 mg/kg (n = 8). Total pirimicarb residues were: 0.08, 0.08, 0.11, 0.14, 0.16, 0.18, 0.25 and 0.3 mg/kg (n = 8).

Raspberries

The Meeting received results of residue trials in Germany on raspberries and blackberries.

In the Netherlands, GAP for raspberries and blackberries is for up to 2 applications of 0.025 kg ai/hL (PHI 7 days) and three raspberry trials from Germany matched this GAP.

Pirimicarb residues in raspberries were 0.23, 0.34, and 0.76 mg/kg (n = 3). Total pirimicarb residues were: 0.24, 0.36 and 0.82 mg/kg (n = 3).

Strawberries

The Meeting received results of residue trials in Italy and Spain on outdoor strawberries and in France, Italy, Spain and the UK on protected strawberries.

GAP in the Czech Republic for berry fruit is for up to 0.25 kg ai/ha, PHI 7 days and in Belgium, GAP is for up to 0.2 kg ai/ha (PHI 7 days), no residue trials matched these GAPs. In two outdoor trials in Italy matching the GAP in France (up to 0.375 kg ai/ha (PHI 15 days), residues of pirimicarb were 0.08 and 0.12 mg/kg and total pirimicarb residues were: 0.09 and 0.14 mg/kg.

The Meeting agreed the data were not sufficient to estimate a maximum residue limit for strawberries and agreed to withdraw the previous recommendation of 0.5 mg/kg.

The Meeting agreed that the data on currants, gooseberries and raspberries could be used to support a 'berry fruit (except grapes and strawberries)' commodity group maximum residue level and estimated a maximum residue level of 1 mg/kg for pirimicarb on berries and other small fruits (except grapes and strawberries) and based on the raspberry data, estimated an STMR of 0.36 mg/kg and HR of 0.82 mg/kg for total pirimicarb residues in berries and other small fruits (except grapes and strawberries).

The Meeting also agreed to withdraw its previous maximum residue level recommendation of 0.5 mg/kg for blackberries; currant, Black and raspberries, Red, Black, because they were being replaced by maximum residue level for berries and other small fruits (except grapes and strawberries).

Onions, bulb

The Meeting received results of residue trials on bulb onions from France, Germany, Italy, Spain and the UK.

GAP for vegetables in Spain is for foliar spray applications of 0.05 kg ai/hL (PHI 3 days for vegetables except cucurbits) and in trials from Italy and Spain matching this GAP, pirimicarb residues in onion bulbs were: < 0.01 (4), 0.01, 0.02, 0.05 and 0.06 mg/kg (n = 8). Total pirimicarb residues in these trials were: < 0.01, < 0.01, < 0.01, < 0.01, 0.01, 0.02, 0.07 and 0.09 mg/kg (n = 8)

In the Czech Republic, GAP for onions and garlic is for up to two applications of 0.025 kg ai/ha (maximum) with a PHI of 14 days. In six trials from Germany, France and the UK matching this GAP, residues of pirimicarb were all < 0.01 (n = 6) and total pirimicarb residues were also < 0.01 (6).

The Meeting agreed to use the trials matching the GAP from Spain and estimated a maximum residue level of 0.1 mg/kg for pirimicarb in onion, bulb (to replace the existing recommendation of 0.5 mg/kg) and estimated an STMR of 0.01 mg/kg and an HR of 0.09 mg/kg for total pirimicarb residues.

Garlic

The Meeting also agreed to extrapolate the results for onion, bulb to garlic and estimated a maximum residue level of 0.1 mg/kg for pirimicarb in garlic (to replace the existing recommendation of 0.5 mg/kg) and estimated an STMR of 0.01 mg/kg and an HR of 0.09 mg/kg for total pirimicarb residues in garlic.

Cauliflower

The Meeting received results of residue trials in France and the UK on cauliflowers.

In the Czech Republic, GAP for brassica vegetables is for up to two applications of 0.25 kg ai/ha (PHI 3 days). Trials from the UK matched this GAP, except for the higher number of

applications. The Meeting noted that the residue half-life for pirimicarb in cauliflowers was less than 7 days, and that the residue contribution from treatments applied more than 14 days before harvest would not be significant.

The Meeting agreed to use the results of the UK trialsm matching the GAP of the Czech Republic, but with 2−5 applications (at 7−14 day intervals). Pirimicarb residues in these trials were: 0.01, 0.01, 0.01, 0.02, 0.02, 0.03, 0.04, 0.04, < 0.05, < 0.05, < 0.05, < 0.05, 0.05 and 0.05 mg/kg (n = 14). Total pirimicarb residues were: 0.01, 0.01, 0.01, 0.02, 0.02, 0.04, 0.04, < 0.05, < 0.05, < 0.05, < 0.05, 0.05, 0.05 and 0.06 mg/kg (n = 14).

Broccoli

The Meeting received results of residue trials in UK on broccoli.

In the Czech Republic, GAP for brassica vegetables is for up to 2 applications of 0.25 kg ai/ha (PHI 3 days). Trials from the UK matched this GAP except for the higher number of applications. The Meeting noted that the residue half-life for pirimicarb in broccoli was less than 7 days, and that the residue contribution from treatments applied more than 14 days before harvest would not be significant.

The Meeting agreed to use the results of the UK trials matching the GAP of the Czech Republic but with 2-5 applications (at 14 day intervals). Pirimicarb residues in these trials were: < 0.01, < 0.01, < 0.01, < 0.01, 0.01, 0.39 and 0.41 mg/kg (n = 7). The total pirimicarb residues were: < 0.01, < 0.01, < 0.01, < 0.01, 0.01, 0.48 and 0.5 mg/kg (n = 7).

Brussels sprouts

The Meeting received results of residue trials in Germany and UK on Brussels sprouts.

In the Czech Republic, GAP for brassica vegetables is for up to two applications of 0.25 kg ai/ha (PHI 3 days). Trials from the UK and Germany matched this GAP, except for the higher number of applications. The Meeting noted that the residue half-life for pirimicarb in Brussels sprouts was less than 7 days, and that the residue contribution from treatments applied more than 14 days before harvest would not be significant.

The Meeting agreed to use the results of the trials from Germany and the UK matching the GAP of the Czech Republic but with 2−5 applications (at 10-35 day intervals). Pirimicarb residues in these trials were: 0.04, 0.04 and 0.05 mg/kg (n = 3). The total pirimicarb residues were: 0.05, 0.05 and 0.06 mg/kg (n = 3).

In Germany, the GAP for brassica vegetables is for up to 3 applications of 0.125 kg ai/ha (PHI 7 days) and in two broccoli trials from Germany matching this GAP, pirimicarb residues for both were 0.02 mg/kg and the total pirimicarb residues were also 0.02 mg/kg.

The combined results from the trials matching the GAPs of the Czech Republic and Germany were: 0.02, 0.02, 0.04, 0.04 and 0.05 mg/kg (n = 5) for pirimicarb and the total pirimicarb residues were: 0.02, 0.02, 0.05, 0.05 and 0.06 mg/kg (n = 5).

Cabbage, head

The Meeting received results of residue trials in Germany and the UK on cabbage.

In France, GAP for cabbage is 0.375 kg ai/ha (PHI 7 days) and in trials from France, Germany and the UK, matching the GAP of France, pirimicarb residues were: 0.01, 0.03, < 0.05, < 0.05, < 0.05, < 0.05, < 0.05 and 0.06 mg/kg (n = 8). Total pirimicarb residues were: 0.02, < 0.05, < 0.05, < 0.05, < 0.05, < 0.05, 0.06 and 0.12 mg/kg (n = 8).

The Meeting agreed that the data on broccoli, Brussels sprouts, cauliflower and cabbage (head) could be used to support a 'brassica vegetables' commodity group maximum residue level and estimated a maximum residue level of 0.5 mg/kg for pirimicarb on brassica (cole or cabbage)

vegetables and estimated an STMR of 0.05 mg/kg (based on the cabbage data) and HR of 0.5 mg/kg (based on the broccoli data) for total pirimicarb residues in brassica (cole or cabbage) vegetables.

The Meeting also agreed to withdraw its previous maximum residue levels of 1 mg/kg for broccoli; Brussels sprouts; cabbages, head and cauliflower, and of 0.5 mg/kg for kohlrabi as they would be replaced by the maximum residue level for brassica (cole or cabbage) vegetables.

Cucumbers and squash, summer

The Meeting received results of residue trials in Italy and Spain on outdoor cucumbers and in France, Italy, Spain and the UK on protected cucumbers. Results from trials on protected courgettes in France and outdoor courgettes in Italy were also provided to the Meeting.

Cucumbers: GAP for cucumbers in the Netherlands is for foliar spray applications of 0.025 kg ai/hL, up to 0.37 kg ai/ha (PHI 1 day). While matching residue trials data for outdoor cucumbers were not available, pirimicarb residues on protected cucumbers from trials in France, Italy, Spain and the UK, matching the GAP of the Netherlands were: 0.09, 0.1, 0.13, 0.14, 0.15, 0.17, 0.24, 0.29 and 0.41 mg/kg. Total pirimicarb residues in these trials were: 0.12, 0.14, 0.16, 0.18, 0.18, 0.21, 0.27, 0.33 and 0.44 mg/kg.

In France, GAP for cucumbers is for up to two applications of 0.375 kg ai/ha (PHI 3 days) and two outdoor cucumber trials in Italy matched this GAP. Pirimicarb residues in these trials were < 0.01 and 0.22 mg/kg. Total pirimicarb residues were < 0.01 and 0.24 mg/kg.

In Spain, GAP for vegetables is 0.05 kg ai/hL (PHI 7 days) and two outdoor cucumber trials in Spain matched this GAP. Pirimicarb residues in these trials were 0.02 and 0.02 mg/kg. Total pirimicarb residues were 0.02 and 0.04 mg/kg.

Squash, summer: GAP for summer squash in the Netherlands is for foliar spray applications of 0.025 kg ai/hL, up to 0.37 kg ai/ha (PHI 1 day). While matching residue trials data for outdoor summer squash were not available, pirimicarb residues on protected summer squash (courgettes) from trials in France, matching the GAP of the Netherlands were 0.11 and 0.14 mg/kg, with total pirimicarb residues in these trials being 0.11 and 0.15 mg/kg.

In Spain, GAP for vegetables is 0.05 kg ai/hL (PHI 7 days) and outdoor summer squash trials in France and Italy matched this GAP. Pirimicarb residues in these trials were < 0.01, < 0.01, 0.01 and 0.02 mg/kg. Total pirimicarb residues in these trials were < 0.01, < 0.01, 0.01 and 0.03 mg/kg.

The Meeting noted that the residues in the protected cucumber and protected summer squash trials matching the Netherlands GAP were from similar populations and agreed to combine them as mutually supporting data for cucumbers and squash, summer. Pirimicarb residues in the combined data set were: 0.09, 0.1, 0.11, 0.13, 0.14, 0.14, 0.15, 0.17, 0.24, 0.29 and 0.41 mg/kg (n = 11). Total pirimicarb residues in these combined trials were: 0.11, 0.12, 0.14, 0.15, 0.16, 0.18, 0.18, 0.21, 0.27, 0.33 and 0.44 mg/kg (n = 11).

The Meeting agreed that the data on cucumbers and summer squash could be used to support a 'fruiting vegetables, cucurbits (except melons and watermelons)' commodity group maximum residue level and estimated a maximum residue level of 1 mg/kg for pirimicarb on fruiting vegetables, cucurbits (except melons and water melons) and estimated an STMR of 0.18 mg/kg and HR of 0.44 mg/kg.

The Meeting also agreed to withdraw its previous maximum residue levels of 1 mg/kg for cucumber and gherkin because they were being replaced by the recommendation for maximum residue levels for fruiting vegetables, cucurbits (except melons and water melons).

Melons, except watermelons

The Meeting received results of residue trials in Italy and Spain on outdoor and protected melons.

GAP for melons in France is for foliar spray applications of up to 0.375 kg ai/ha (PHI 3 days). Residue trials from Italy and France, for both outdoor melons (4) and protected melons (4) matched this GAP.

Pirimicarb residues in outdoor melons were 0.03, 0.06, 0.06 and 0.11 mg/kg (n = 4). Total pirimicarb residues in melon flesh were 0.02, 0.03, 0.05 and 0.09 mg/kg.

In protected melons, pirimicarb residues were: 0.02, 0.04, 0.04 and 0.13 mg/kg (n = 4), and total pirimicarb residues in melon flesh were: 0.01, 0.02, 0.02 and 0.04 mg/kg (n = 4).

The Meeting noted that the two data sets were from similar populations and agreed to use the results of the outdoor and protected melon trials to give a combined data set of: 0.02, 0.03, 0.04, 0.04, 0.06, 0.06, 0.11 and 0.13 mg/kg (n = 8) for pirimicarb in whole melons. Total pirimicarb residues in the flesh were: 0.01, 0.02, 0.02, 0.02, 0.03, 0.04, 0.05 and 0.09 mg/kg (n = 8).

The Meeting estimated a maximum residue level of 0.2 mg/kg for pirimicarb in melons, except watermelon and estimated an STMR of 0.025 mg/kg and an HR of 0.09 mg/kg for total pirimicarb residues in the pulp.

Tomato

The Meeting received results of residue trials in France, Italy and Spain on outdoor tomatoes and in France, Italy, Spain and the UK on protected tomatoes.

In Spain, GAP for vegetables is 0.05 kg ai/hL (PHI 3 days except cucurbits) and in outdoor tomato trials from France, Italy and Spain, matching this GAP, pirimicarb residues were: 0.02, 0.03, 0.07, 0.08, 0.09, 0.1, 0.1 and 0.16 mg/kg. Total pirimicarb residues in these trials were: 0.03, 0.03, 0.1, 0.1, 0.11, 0.12, 0.14 and 0.18 mg/kg.

In trials on protected tomatoes in France and the UK matching the GAP of the Netherlands (up to 0.37 kg ai/hL with a 1 day PHI), pirimicarb residues were: 0.07, 0.08, 0.1, 0.1, 0.1, 0.17, 0.2 and 0.2 mg/kg and total pirimicarb residues were 0.07, 0.08, 0.1, 0.1, 0.1, 0.17, 0.2 and 0.2 mg/kg.

In trials on protected tomatoes in Spain and Italy matching the GAP of Spain, pirimicarb residues were: 0.05, 0.11, 0.21 and 0.22 mg/kg and total pirimicarb residues were 0.07, 0.13, 0.23 and 0.25 mg/kg.

The Meeting noted that the results of the outdoor tomato trials, the protected tomato trials matching the GAP of Spain and the protected tomato trials matching the GAP of the Netherlands appeared to be from similar populations and the Meeting agreed to combine all the tomato residue results. The combined residues of pirimicarb were: 0.02, 0.03, 0.05, 0.07, 0.07, 0.08, 0.08, 0.09, 0.1, 0.1, 0.1, 0.1, 0.1, 0.11, 0.16, 0.17, 0.2, 0.2, 0.21 and 0.22 mg/kg (n = 20). Total pirimicarb residues in these trials were: 0.03, 0.03, 0.07, 0.07, 0.08, 0.1, 0.1, 0.1, 0.1, 0.1, 0.11, 0.12, 0.13, 0.14, 0.17, 0.18, 0.2, 0.2, 0.23 and 0.25 mg/kg (n = 20).

Peppers, sweet

The Meeting received results of residue trials in Italy and Spain on outdoor peppers and in France, Italy, Spain and the UK on protected peppers.

In Spain, GAP for vegetables is 0.05 kg ai/hL (PHI 3 days except cucurbits) and in outdoor pepper trials from Italy and Spain, matching this GAP, pirimicarb residues were: 0.01, 0.02, 0.03, 0.03, 0.04, 0.05, 0.07 and 0.07 mg/kg. Total pirimicarb residues in these trials were: 0.01, 0.03, 0.03, 0.04, 0.06, 0.07, 0.08 and 0.09 mg/kg.

In trials on protected peppers in France, Italy, Spain and the UK matching the GAP of Spain, pirimicarb residues were: < 0.01, 0.01, 0.04, 0.05, 0.05, 0.08, 0.08, 0.14, 0.15 and 0.18 mg/kg. Total pirimicarb residues in these trials were: < 0.01, 0.01, 0.04, 0.05, 0.06, 0.08, 0.08, 0.17, 0.19 and 0.22 mg/kg.

The Meeting noted that the combined results of the outdoor and protected sweet pepper trials appeared to be from the same population and the Meeting agreed to combine all the sweet pepper residue results. The combined residues of pirimicarb were: < 0.01, 0.01, 0.01, 0.02, 0.03, 0.03, 0.04, 0.04, 0.05, 0.05, 0.05, 0.07, 0.07, 0.08, 0.08, 0.14, 0.15 and 0.18 mg/kg (n = 18). Total pirimicarb residues in these trials were: < 0.01, 0.01, 0.01, 0.03, 0.03, 0.04, 0.04, 0.05, 0.06, 0.06, 0.07, 0.08, 0.08, 0.08, 0.09, 0.17, 0.19 and 0.22 mg/kg (n = 18).

The Meeting agreed that the data on tomatoes and sweet peppers could be used to support a 'fruiting vegetables, other than cucurbits, mushrooms, edible fungi and sweet corn (kernels and corn-on-the-cob)' commodity group maximum residue level and estimated a maximum residue level of 0.5 mg/kg for pirimicarb on fruiting vegetables, other than cucurbits (except mushrooms, fungi, edible (not including mushrooms), sweet corn (kernels) and sweet corn (corn-on-the-cob)) and estimated an STMR of 0.105 mg/kg and HR of 0.25 mg/kg, based on the tomato data.

The Meeting also agreed to withdraw its previous maximum residue levels for eggplant (1 mg/kg); peppers, chili (2 mg/kg); peppers, Sweet (1 mg/kg) and tomatoes because they were being replaced by the maximum residue level for fruiting vegetables, other than cucurbits (except mushrooms, fungi, edible (not including mushrooms), sweet corn (kernels) and sweet corn (corn-on-the-cob)).

Dried chili peppers

The Meeting agreed to apply a default dehydration factor of 10 (in the absence of specific processing information) to the above estimated maximum residue level, STMR and HR and estimated a maximum residue level of 5 mg/kg for pirimicarb in dried chili peppers and an STMR of 1.05 mg/kg and HR of 2.5 mg/kg for total pirimicarb residues.

Sweetcorn

The Meeting received results of residue trials in France on sweetcorn.

In France, GAP is 0.2 kg ai/ha (PHI 7 days) and in trials from France matching this GAP, pirimicarb residues in sweetcorn kernels were: < 0.01, < 0.01, < 0.01 and 0.02 mg/kg (n = 4). Total pirimicarb residues in these trials were: < 0.01, $\underline{< 0.01}$, $\underline{< 0.01}$ and 0.02 mg/kg (n = 4).

The Meeting estimated a maximum residue level of 0.05 mg/kg for pirimicarb in sweet corn (kernels) and estimated an STMR of 0.01 mg/kg and an HR of 0.02 mg/kg for total pirimicarb residues in sweet corn kernels. The Meeting agreed to withdraw the previous recommendation for sweetcorn (corn on the cob) of 0.05* mg/kg.

Lettuce

The Meeting received results of residue trials on outdoor lettuce from France and the UK and on protected lettuce from France, Italy, Spain and the UK. These trials were conducted using a range of different lettuce types, most of which (where identified) were the 'Iceberg' type head lettuce or the more loosely packed 'Butterhead' type of leafy lettuce.

In outdoor lettuce trials from France and the UK matching the GAP of the Netherlands of 0.25 kg ai/ha (PHI 7 days), pirimicarb residues were: < 0.01, < 0.01, 0.01 and 0.02 mg/kg and total pirimicarb residues were < 0.01, 0.07, 0.11 and 0.33 mg/kg.

In Spain, GAP for vegetables is 0.05 kg ai/hL (PHI 3 days except cucurbits) and while no outdoor lettuce trials matching this GAP were available, in protected lettuce trials in France, Spain and UK matching the GAP of Spain , pirimicarb residues were: 0.6, 0.84, 0.86, 1.2, 1.4, 1.5, 1.7 and 2.3 mg/kg (n = 8). Total pirimicarb residues in these trials were: 1.3, 2.0, 2.1, 2.3, 2.3, 2.4, 2.8 and 3.0 mg/kg (n-8).

Five trials on protected lettuce in France matched the German GAP of 0.125 kg ai/ha (PHI 7 days) but with two applications at 7−12 day intervals rather than the GAP maximum of 3 applications

at least 10 days apart. The Meeting noted that the residue contribution from treatments made more than 21 days before harvest would be insignificant, and agreed the use these trials. Pirimicarb residues were: 0.1, 0.1, 0.23, 0.25 and 0.28 mg/kg. Total pirimicarb residues were 0.38, 0.53, 0.65, 0.76 and 0.92 mg/kg.

The Meeting noted that the residues in the protected lettuce trials were higher than in the outdoor crops and agreed to use the results of the protected lettuce trials. The combined residues of pirimicarb were: 0.1, 0.1, 0.23, 0.25, 0.28, 0.6, 0.84, 0.86, 1.2, 1.4, 1.5, 1.7 and 2.3 mg/kg (n = 13). Total pirimicarb residues in these trials were: 0.38, 0.53, 0.65, 0.76, 0.92, 1.3, 2.0, 2.1, 2.3, 2.3, 2.4, 2.8 and 3.0 mg/kg (n = 13).

The Meeting estimated a maximum residue level of 5 mg/kg for pirimicarb in lettuce, head (replacing the existing recommendation of 1 mg/kg) and estimated an STMR of 2 mg/kg and an HR of 3 mg/kg for total pirimicarb residues.

The Meeting also noted that in the nine protected lettuce trials where the lettuce type could be classified as either head lettuce (seven trials) or leaf lettuce (two trials), the results appeared to be from similar populations, and agreed to use the results to estimate a maximum residue level of 5 mg/kg for pirimicarb in lettuce, leaf and estimated an STMR of 2 mg/kg and an HR of 3 mg/kg for total pirimicarb residues.

Kale

The Meeting received results of residue trials in UK on kale.

In the Czech Republic, GAP for brassica vegetables is for up to 2 applications of 0.25 kg ai/ha (PHI 3 days). Six trials on kale from UK matched this GAP but in three trials an additional application was made 16-17 days before sampling. The Meeting noted that the residue half-life for pirimicarb in kale was less than 3 days and that the residue contribution from treatments applied more than 14 days before harvest would not be significant.

The Meeting agreed to use the results of these trials from the UK, matching the GAP of the Czech Republic, but with 2 or 3 applications. Pirimicarb residues in kale leaves and petioles from these trials were: < 0.05, 0.05, 0.07, 0.08, 0.09 and 0.15 mg/kg (n = 6). Total pirimicarb residues were: 0.20, 0.26, 0.27, 0.34, 0.35 and 0.60 mg/kg (n = 6).

The Meeting estimated a maximum residue level of 0.3 mg/kg for pirimicarb in kale and estimated an STMR of 0.31 mg/kg and an HR of 0.6 mg/kg for total pirimicarb residues.

Beans (except broad bean and soya bean)

The Meeting received results of residue trials in France, Germany, Greece, the Netherlands and Spain on fresh beans.

In Spain, GAP for vegetables is 0.05 kg ai/hL (PHI 3 days except cucurbits) and in trials from Greece and Spain, matching this GAP, pirimicarb residues were: 0.09, 0.22, 0.36, 0.39 and 0.4 mg/kg. Total pirimicarb residues in these trials were: 0.15, 0.27, 0.49, 0.55 and 0.59 mg/kg.

GAP for legume vegetables in Germany is for up to 3 applications of 0.25 kg ai/ha, at least 10 days apart (PHI 3 days). The Meeting agreed to use matching residue trials data with 2 applications (7-12 days apart) from France and Germany as the final applications contributed the majority of the residues. In these trials, pirimicarb residues were: 0.04, 0.23, 0.25 and 0.26 mg/kg and total pirimicarb residues were 0.1, 0.26, 0.31 and 0.38 mg/kg.

In France, GAP for green beans is 0.375 kg ai/ha (PHI 7 days) and in trials from France, Germany and the Netherlands, matching this GAP, pirimicarb residues were: 0.03, 0.07, 0.1, 0.13, 0.16, 0.21, 0.21, 0.22, 0.28 and 0.31 mg/kg. Total pirimicarb residues in these trials were: 0.09, 0.19, 0.22, 0.23, 0.24, 0.25, 0.27, 0.38, 0.42 and 0.44 mg/kg.

The Meeting noted that the results of these trials appeared to be from the same population and agreed to combine all of the bean (with pods) residue results. The combined residues of pirimicarb were: 0.03, 0.04, 0.07, 0.09, 0.1, 0.13, 0.16, 0.21, 0.21, 0.22, 0.22, 0.23, 0.25, 0.26, 0.28, 0.31, 0.36, 0.39 and 0.4 mg/kg (n = 19). Total pirimicarb residues in these trials were: 0.09, 0.1, 0.15, 0.19, 0.22, 0.23, 0.24, 0.25, 0.26, 0.27, 0.27, 0.31, 0.38, 0.38, 0.42, 0.44, 0.49, 0.55 and 0.59 mg/kg (n = 19).

Broad bean, shelled (succulent)

The Meeting received results of residue trials in the UK on broad beans.

In Germany, GAP is for up to 3 applications of 0.25 kg ai/ha, at least 10 days apart (PHI 3 days). The Meeting considered that the residue contribution from applications made more than 20 days before harvest would be negligible and agreed to use matching residue trials data with 2 applications (7-12 days apart) from the UK. In these trials, pirimicarb residues were: 0.01, 0.02, 0.03 and 0.04 mg/kg and total pirimicarb residues were 0.01, 0.03, 0.05 and 0.06 mg/kg.

Peas, shelled

The Meeting received results of residue trials in France, Italy and the UK on fresh peas (without pods).

In France, GAP for peas is 0.375 kg ai/ha (PHI 7 days) and in ten trials from France, Italy and UK, matching this GAP, pirimicarb residues in peas (without pods) were all < 0.01 (n = 10).

Peas

The Meeting received results of residue trials in Germany and the Netherlands on peas (with pods).

In two trials from Germany and the Netherlands, matching the GAP in France (0.375 kg ai/ha, PHI 7 days), pirimicarb residues were < 0.01 and < 0.01 mg/kg and total pirimicarb residues were also < 0.01 and < 0.01 mg/kg.

The Meeting agreed that the data on peas and beans (with and without pods) could be used to support a 'legume vegetables (except soya beans)' commodity group maximum residue level and using the results for beans (with pods), estimated a maximum residue level of 0.7 mg/kg for pirimicarb on legume vegetables (except soya beans) and estimated an STMR of 0.27 mg/kg and HR of 0.59 mg/kg.

The Meeting also agreed to withdraw its previous maximum residue levels of 0.1 mg/kg for beans, shelled; 1 mg/kg for common bean (pods and/or immature seeds) and 0.2 mg/kg for peas (pods and succulent=immature seeds) because they were being replaced by the maximum residue level for legume vegetables (except soya beans).

Beans and peas (dry)

Residue results on beans (dry) and peas (dry) were made available to the Meeting from trials in France and Spain.

Beans (dry): In Spain, GAP for vegetables is 0.05 kg ai/hL (PHI 3 days except cucurbits). In three trials on beans grown in France for dry bean production, matching the GAP of Spain, pirimicarb residues in beans (dry) were: 0.03, 0.04 and 0.09 mg/kg and total pirimicarb residues were 0.06, 0.08 and 0.14 mg/kg.

Peas (dry): In Spain, GAP for vegetables is 0.05 kg ai/hL (PHI 3 days except cucurbits). In five trials on peas grown in France and Spain for dry pea production, matching the GAP of Spain, pirimicarb residues in peas (dry) were: < 0.01, < 0.01, 0.05, 0.08 and 0.12 mg/kg and total pirimicarb residues were < 0.01, < 0.01, 0.07, 0.1 and 0.15 mg/kg.

The Meeting noted that the results on dry peas and beans were mutually supportive as they appeared to be from similar populations. The combined results for pirimicarb were: < 0.01, < 0.01, 0.03, 0.04, 0.05, 0.08, 0.09 and 0.12 mg/kg (n = 8) and total pirimicarb residues were < 0.01, < 0.01, 0.06, 0.07, 0.08, 0.1, 0.14 and 0.15 mg/kg (n = 8).

The Meeting agreed that the data on peas, dry and beans, dry could be used to support a 'pulse (except soya beans)' commodity group maximum residue level and estimated a maximum residue level of 0.2 mg/kg for pirimicarb on pulses (except soya beans) and estimated an STMR of 0.075 mg/kg and HR of 0.15 mg/kg .

Carrots

The Meeting received results of residue trials in France, Italy and Spain on carrots.

In France, GAP for carrots is 0.375 kg ai/ha (PHI 7 days). In eight trials from France, Spain and Italy, matching the GAP of Spain, pirimicarb residues were all < 0.01 (8) and total pirimicarb residues were also all < 0.01 (8) mg/kg (n = 8).

Sugar beet

The Meeting received results of residue trials in France, Italy, Spain and the UK on sugar beet.

In Spain, GAP for sugar beet is 0.05 kg ai/hL (PHI 3 days). In two trials from Italy matching this GAP, pirimicarb residues were: < 0.01 and < 0.01 mg/kg and total pirimicarb residues were also < 0.01 and < 0.01 mg/kg.

In the Czech Republic, GAP for sugar beet is 0.25 kg ai/ha (PHI 7 days). In trials from the UK, matching the GAP in the Czech Republic, pirimicarb residues were: < 0.01 (17), 0.01, 0.01 and 0.02 mg/kg (n = 20) and the total pirimicarb residues were also < 0.01 (17), 0.01, 0.01 and 0.02 mg/kg (n = 20).

The Meeting noted that these two data sets appeared to be from the similar populations, and agreed to combine them. The residues of pirimicarb were: < 0.01 (19), 0.01, 0.01 and 0.02 mg/kg (n = 22) and the total pirimicarb residues were < 0.01 (19), 0.01, 0.01 and 0.02 mg/kg (n = 22).

Potatoes

The Meeting received results of residue trials in France, Germany, Spain and the UK on potatoes.

In Spain, GAP for vegetables is 0.05 kg ai/hL (PHI 3 days except cucurbits). In two potato trials from Spain matching this GAP, pirimicarb residues were: < 0.01 and < 0.01 mg/kg and total pirimicarb residues were also < 0.01 and < 0.01 mg/kg.

In the Czech Republic and in the Netherlands, GAP for potatoes is for up to two applications of 0.25 kg ai/ha (PHI 7 days). In trials from Germany and the UK matching these GAPs, pirimicarb residues were all < 0.01 mg/kg (n = 5) and total pirimicarb residues were also < 0.01 mg/kg (n = 5).

The Meeting noted that these two data sets appeared to be from the same population, and agreed that they could be combined. Residues of pirimicarb were: < 0.01 (7) mg/kg and the total pirimicarb residues were also < 0.01 (7) mg/kg.

The Meeting agreed that the data on carrots, sugar beet and potatoes could be used to support a 'root and tuber vegetables' commodity group maximum residue level and estimated a maximum residue level of 0.05 mg/kg for pirimicarb on root and tuber vegetables and estimated an STMR of 0.01 mg/kg and HR of 0.02 mg/kg based on the sugar beet data.

The Meeting also agreed to withdraw its previous maximum residue levels of 0.05 (*) mg/kg for beetroot; parsnip; potato; radish; sugar beet and 'turnip (garden) because they were being replaced by the maximum residue level for root and tuber vegetables

Artichokes, globe

The Meeting received results of residue trials in France, Italy and Spain on globe artichokes.

In Spain, GAP for vegetables is 0.05 kg ai/hL (PHI 3 days except cucurbits). In six trials from Spain and Italy, matching the GAP of Spain, pirimicarb residues were: 0.33, 0.42, 0.44, 0.73, 1.9 and 2.6 mg/kg and total pirimicarb residues were: 0.44, 0.51, 0.53, 0.85, 2.1 and 2.8 mg/kg.

In six trials from France, matching the French GAP (0.375 kg ai/ha, PHI 7 days), pirimicarb residues were: 0.07, 0.16, 0.18, 0.23, 0.3 and 0.46 mg/kg and total pirimicarb residues were: 0.09, 0.19, 0.22, 0.27, 0.41 and 0.56 mg/kg.

The Meeting noted that the results of the trials matching the GAPs in Spain and in France appeared to be from different populations and agreed to use the results from the trials matching the GAP in Spain.

The Meeting estimated a maximum residue level of 5 mg/kg for pirimicarb in artichoke, globe and estimated an STMR of 0.69 mg/kg and an HR of 2.8 mg/kg for total pirimicarb residues.

Asparagus

The Meeting received results of residue trials in Germany on asparagus.

In France, GAP for asparagus is 0.375 kg ai/ha, applied to the ferns (once harvesting is completed for the season) with a PHI of 200 days (before the new spears are harvested the next season).

In four trials from Germany and Greece, involving application rates higher than in the French GAP, but with similar PHIs, pirimicarb residues were all < 0.01 mg/kg (n = 4) and total pirimicarb residues were also all < 0.01 mg/kg (n = 4).

The Meeting agreed that because residues were all < 0.01 mg/kg in newly emerged spears from treated plants, the results of these trials could be used and the Meeting estimated a maximum residue level of 0.01 (*) mg/kg for pirimicarb in asparagus and estimated an STMR of 0 mg/kg and an HR of 0 mg/kg for total pirimicarb residues.

Barley

The Meeting received results of residue trials in France and the UK on winter barley.

In the Czech Republic, GAP for cereals is for up to two applications (0.15 kg ai/ha), up to the ‘soft dough’ growth stage (BBCH 85). While the label also states a PHI of 14 days, the Meeting agreed to use trials that matched the crop growth stage instruction as being a better indication of GAP.

In trials from France and the UK, matching the Czech Republic GAP (with PHIs ranging from 20 to 29 days), pirimicarb residues were: < 0.01 (6), 0.01 and 0.03 mg/kg and total pirimicarb residues were: < 0.01 (6), 0.01 and 0.05 mg/kg.

Wheat

The Meeting received results of residue trials in France and the UK on winter wheat.

In the Czech Republic, GAP for cereals is for up to two applications (0.15 kg ai/ha), up to the ‘soft dough’ growth stage (BBCH 85). While the label also states a PHI of 14 days, the Meeting agreed to use trials that matched the crop growth stage instruction as being a better indication of GAP.

In trials from France and the UK, matching the Czech Republic GAP (with PHIs ranging from 21 to 46 days), pirimicarb residues were all < 0.01 mg/kg (n = 8) and total pirimicarb residues were also all < 0.01 mg/kg (n = 8).

Maize

The Meeting received results of residue trials in France on maize.

In France, GAP for maize is 0.2 kg ai/ha, up to the end of flowering, with a PHI of 80 days for grain and 60 days for animal forage. In trials from France, Germany and Italy, matching the GAP in France, pirimicarb residues were: < 0.01 (12) and 0.02 mg/kg (n = 13) and total pirimicarb residues were: < 0.01 (12) and 0.04 mg/kg (n = 13).

The Meeting agreed that the data on wheat, barley and maize could be used to support a 'cereal grains (except rice)' commodity group maximum residue level and estimated a maximum residue level of 0.05 mg/kg for pirimicarb on cereal grains (except rice) and estimated an STMR of 0.01 mg/kg (based on the maize data) and HR of 0.05 mg/kg (based on the barley data) for total pirimicarb residues.

The Meeting also agreed to withdraw its previous maximum residue levels of 0.05 mg/kg (*) for barley, oats and wheat because they were being replaced by a maximum residue level for cereal grains (except rice).

Rape seed

The Meeting received results of residue trials in France, Spain and the UK on oil seed rape.

In Spain, GAP is 0.25 kg ai/ha (PHI 21 days) and in two trials from Spain, matching this GAP, pirimicarb residues were < 0.01 and < 0.01 mg/kg total pirimicarb residues were also < 0.01 and < 0.01 mg/kg.

GAP for oil seed rape in the Czech Republic is 0.21 kg ai/ha, with no PHI specified, and in six trials from UK and France, matching this GAP, with PHIs of 14–17 days, pirimicarb residues were: < 0.01 (5) and 0.02 mg/kg and total pirimicarb residues were also ≤ 0.01 (5) and 0.02 mg/kg.

The Meeting agreed to use the results from the trials matching the Czech Republic GAP to estimate a maximum residue level of 0.05 mg/kg for pirimicarb in rape seed (to replace the existing recommendation of 0.2 mg/kg) and estimated an STMR of 0.01 mg/kg and an HR of 0.02 mg/kg for total pirimicarb residues.

Sunflower seed

The Meeting received results of residue trials in France, Italy and Spain on sunflower.

In France, GAP is 0.25 kg ai/ha (PHI 21 days) and in twelve trials from Italy and Spain, matching this GAP, pirimicarb residues were < 0.01, < 0.01, < 0.01, 0.01, 0.01, 0.01, 0.01, 0.03, 0.03, 0.03, 0.03 and 0.05 mg/kg and total pirimicarb residues were: < 0.01, < 0.01, < 0.01, 0.01, 0.01, 0.01, 0.02, 0.03, 0.03, 0.04, 0.04 and 0.07 mg/kg.

The Meeting estimated a maximum residue level of 0.1 mg/kg for pirimicarb in sunflower seed and estimated an STMR of 0.015 mg/kg and an HR of 0.07 mg/kg for total pirimicarb residues.

Residues in animal commodities

Animal feed commodities

The Meeting noted that the two demethyl pirimicarb metabolites can occur in animal feeds at levels averaging about 50% of the total pirimicarb residues, and these metabolites can therefore be a significant component of diet.

Because animal transfer studies have only been conducted with the parent compound, the Meeting considered there was insufficient information to determine the behaviour of the dimethyl carbamate metabolites in animals, and agreed to use the total pirimicarb residue values instead of just

parent pirimicarb residue values to estimate STMRs and highest residues for animal feeds in order to avoid under-estimating the potential for residues of pirimicarb metabolites to transfer into animal commodities.

Bean forage (green)

The Meeting received information on residues in bean forage from trials on fresh beans from Spain, on broad beans from the UK and beans (dry) from France.

In Spain, GAP for vegetables is 0.05 kg ai/hL (PHI of 3 days, except for cucurbits) and in two common bean trials from Spain, matching this GAP, total pirimicarb residues in vines (without pods) were: 3.5 and 7.0 mg/kg.

GAP for legume vegetables in Germany is for up to 3 applications of 0.25 kg ai/ha, at least 10 days apart (PHI 3 days). The Meeting considered that the final applications contributed the majority of the residues and agreed to use data from residue trials with 2 applications (7−12 days apart) from the UK. In these trials, total pirimicarb residues in broad bean forage (without pods) were: 0.21, 0.42, 0.51 and 0.73 mg/kg.

In three trials on beans from France, grown for dry bean production, matching the GAP of Spain, total pirimicarb residues in bean forage (including empty pods) were: 0.34, 0.44 and 0.64 mg/kg.

The total pirimicarb residue results in forage (with or without empty pods) from common beans, broad beans and beans grown for dry bean production were: 0.21, 0.34, 0.42, 0.44, 0.51, 0.64, 0.73, 3.5 and 7 mg/kg.

The Meeting noted that the residues in the common bean forage (3.5 and 7.0 mg/kg) were significantly higher than in the vines (with or without empty pods) from the other bean varieties, and agreed to use these results to estimate STMRs and highest residues for bean forage.

The Meeting estimated an STMR of 5.25 mg/kg and a highest residue of 7 mg/kg for total pirimicarb residues (fresh weight) in bean forage (green) for the purposes of calculating animal dietary burden.

Pea hay or pea fodder (dry)

The Meeting received information on residues in pea foliage from trials on fresh peas in France and on peas grown for dry pea production in France and Spain.

In France, GAP for peas is 0.375 kg ai/ha (PHI 7 days) and in two trials from France, matching this GAP, pirimicarb residues were 0.02 and 0.44 mg/kg and total pirimicarb residues in vines and empty pods were: 0.09 and 0.62 mg/kg.

In Spain, GAP for vegetables is 0.05 kg ai/hL (PHI 3 days except cucurbits). In five trials on peas in France and Spain, matching Spanish GAP, pirimicarb residues in vines and empty pods were 0.34, 0.89, 2.7, 2.9 and 14 mg/kg and total pirimicarb residues were: 0.72, 1.6, 4.1, 4.6 and 18 mg/kg.

The Meeting noted that the residues in the pea forage from the trials matching the GAP of Spain were significantly higher than those matching the GAP in France, and agreed to use the higher results to estimate STMRs and highest residues for pea forage.

Allowing for the standard 25% dry matter for pea vines (*FAO Manual* p 148*)*, the Meeting estimated a maximum residue level of 60 mg/kg (dry weight) for pea hay or pea fodder, dry and estimated an STMR of 16.4 mg/kg and a highest residue of 72 mg/kg (dry weight) for total pirimicarb residues for the purposes of calculating animal dietary burden.

Maize forage

The Meeting received results of residues in maize forage from trials conducted in France, Germany and Italy.

In France, GAP for maize is 0.2 kg ai/ha, up to the end of flowering, with a PHI of 60 days for animal forage. In six trials from France and Italy, matching the GAP in France, pirimicarb residues were all < 0.01 mg/kg (n = 6) and total pirimicarb residues were also all < 0.01 mg/kg (n = 6).

The Meeting estimated an STMR of 0 mg/kg in maize forage and a highest residue of 0 mg/kg for total pirimicarb residues (fresh weight) for the purposes of calculating animal dietary burden.

Barley straw and fodder, dry

The Meeting received results of residues in barley straw from trials on winter barley in France and the UK.

In the Czech Republic, GAP for cereals is for a maximum of two applications, up to the 'soft dough' growth stage (BBCH 85).

In trials from France and the UK, matching the GAP of the Czech Republic (with PHIs ranging from 20 to 29 days), pirimicarb residues in barley straw were: < 0.01, < 0.01, 0.02, 0.02, 0.02, 0.03, 0.08 and 0.13 mg/kg and total pirimicarb residues were: < 0.01, < 0.01, 0.04, 0.04, 0.04, 0.07, 0.11 and 0.22 mg/kg (n = 8).

Wheat straw and fodder, dry

The Meeting received results of residues in wheat straw from trials on winter wheat in France and the UK.

In the Czech Republic, GAP for cereals is for a maximum of two applications, up to the 'soft dough' growth stage (BBCH 85).

In trials from France and the UK, matching the GAP of the Czech Republic (with PHIs ranging from 21 to 46 days), pirimicarb residues were: < 0.01, < 0.01, 0.02, 0.02, < 0.05, 0.07 and 0.16 mg/kg (n = 7) and total pirimicarb residues were: < 0.01, < 0.01, 0.05, 0.08, < 0.09, 0.23 and 0.33 mg/kg (n = 7).

Maize fodder

The Meeting received results of residues in maize fodder from trials conducted in France, Germany and Italy.

In France, GAP for maize is 0.2 kg ai/ha, up to the end of flowering, with a PHI of 80 days for grain. In trials from France, Germany and Italy, matching the GAP of France, pirimicarb residues in maize fodder were: < 0.01 (10) and 0.02 (3) mg/kg (n = 13) for pirimicarb and total pirimicarb residues were: < 0.01 (9), < 0.01, 0.02 (3) mg/kg (n = 13).

The Meeting noted that the results from the wheat straw, barley straw and maize fodder trials appeared to be from the similar populations and agreed to combine the residues to estimate a commodity group maximum residue level, STMR and highest residue. Pirimicarb residues were: < 0.01 (14), 0.02 (8), 0.03, < 0.05, 0.07, 0.08, 0.13 and 0.16 mg/kg (n = 28) and total pirimicarb residues were: < 0.01 (13), <u>< 0.01, 0.02</u> (3) 0.04, 0.04, 0.04, 0.05, 0.07, 0.08, < 0.09, 0.11, 0.22, 0.23 and 0.33 mg/kg (n = 28).

The Meeting estimated a maximum residue level of 0.3 mg/kg for pirimicarb in straw and fodder (dry) of cereal grains except rice and for the purposes of calculating animal dietary burden, estimated an STMR of 0.015 mg/kg and a highest residue of 0.33 mg/kg for total pirimicarb residues.

Sugar beet leaves or tops

The Meeting received information on residues in sugar beet leaves from trials on sugar beet in France, Italy, Spain and the UK.

In Spain, GAP for sugar beet is 0.05 kg ai/hL (PHI 3 days). In two trials from Italy matching this GAP, total pirimicarb residues in sugar beet leaves were: 2.1 and 4.3 mg/kg.

In the Czech Republic, GAP for sugar beet is 0.25 kg ai/ha (PHI 7 days). In trials from the UK, matching the GAP of the Czech Republic, the total pirimicarb residues in sugar beet leaves were: 0.14, 0.45, 0.50, 0.53, 0.59, 0.66, 0.86, 1.2, 1.3 and 3.4 mg/kg (n = 10).

The Meeting noted that these two data sets appeared to be from the same population, and agreed to combine them. The total pirimicarb residues in sugar beet leaves were: 0.14, 0.45, 0.48, 0.53, 0.59, 0.66, 0.86, 1.2, 1.3, 2.1, 3.4 and 4.3 mg/kg (n = 12).

The Meeting estimated an STMR of 0.76 mg/kg (fresh weight) and a highest residue of 4.3 mg/kg (fresh weight) for the total pirimicarb residues in sugar beet leaves or tops for the purposes of calculating animal dietary burden.

FATE OF RESIDUES DURING PROCESSING

Pirimicarb is stable under the standard hydrolysis conditions used to mimic food processing. The only carbamate degradate to be observed was demethyl pirimicarb at < 0.8% of the total radioactivity and this metabolite was also found in plant metabolism studies.

The Meeting received information on the fate of incurred residues of pirimicarb during the processing of apples, plums, tomatoes, Brussels sprouts, head cabbage, kale, potatoes and barley. The processing factors (PF) shown below were calculated from the total residues for the commodities for which MRLs, STMRs and HRs were estimated.

RAC	Processed product	No.	PF	Median PF
Apples	juice	4	0.50, 0.74, 0.75, 1.00	0.745
	sauce	4	0.20, 0.50, 0.50, 1.00	0.5
	wet pomace	1	1.66	1.66
Plums	prunes	4	1.69, 1.92, 2.07, 2.82	2.0
Tomatoes	juice	5	0.50, 0.62, 0.70, 0.86, 1.54	0.70
	puree	5	0.62, 0.64, 1.49, 2.19, 2.33	1.49

Apples were processed into juice, sauce and wet pomace with processing factors of 0.745, 0.5 and 1.66, respectively. Based on the STMR value of 0.18 mg/kg for pome fruit, the STMR-Ps were 0.13 mg/kg, 0.09 mg/kg and 0.3 mg/kg for total pirimicarb residues in apple juice, sauce and wet pomace, respectively.

Plums were processed into dried prunes with a median processing factor of 2. Based on the STMR of 0.23 mg/kg and the HR of 0.43 mg/kg for plums, the STMR-P was 0.46 mg/kg and the HR-P was 0.86 mg/kg for total pirimicarb residues in prunes.

Tomatoes were processed into juice and puree with processing factors of 0.7 and 1.49. Based on the STMR value of 0.105 mg/kg for tomato, the STMR-Ps were 0.07 mg/kg and 0.16 mg/kg for total pirimicarb residues in tomato juice and puree.

Farm animal dietary burden

The Meeting estimated the dietary burden of pirimicarb residues in farm animals from the diets listed in Appendix IX of the *FAO Manual* (FAO, 2002). One feed commodity only from each Codex

Commodity Group is used. Calculation from the highest residue values provides the concentrations in feed suitable for estimating MRLs for animal commodities, while calculation from the STMR values for feed is suitable for estimating STMR values for animal commodities. In the case of processed commodities, the STMR-P value is used for both intake calculations.

Estimated maximum dietary burden of farm animals

Commodity	CC	Residue (mg/kg)	Basis	DM %	Residue ÷ DM	*Diet content (%)*			Residue contribution, mg/kg		
						Beef cattle	*Dairy cows*	*Poultry*	Beef cattle	Dairy cows	Poultry
Apple wet pomace	AB	0.3	STMR-P	40	0.75	***40***	***20***		0.3	0.15	
Pea, field hay	AL	72	Highest residue	100	72.00	***25***	***50***		18	36	
Bean forage (Note)	AL	7	Highest residue	35	20.00	*30*	*30*				
Barley straw	AS	0.33	Highest residue	89	0.37	*10*	***20***			0.074	
Millet straw	AS	0.33	Highest residue	90	0.37	*10*	*10*				
Oats straw	AS	0.33	Highest residue	90	0.37	*10*	*10*				
Rye straw	AS	0.33	Highest residue	90	0.37	*10*	*10*				
Sorghum stover	AS	0.33	Highest residue	88	0.38	*25*	*15*				
Wheat straw	AS	0.33	Highest residue	88	0.38	***10***	*10*		0.038		
Sugar beet tops	AV	4.3	Highest residue	23	18.7	***20***	***10***		3.74	1.87	
Barley grain	GC	0.05	Highest residue	88	0.06	*50*	*40*	*75*			
Corn grain	GC	0.05	Highest residue	88	0.06	***5***	*40*	***80***	0.003		0.045
Corn, pop grain	GC	0.05	Highest residue	88	0.06	*80*	*40*	*80*			
Millet grain	GC	0.05	Highest residue	88	0.06	*50*	*40*	*70*			
Oats grain	GC	0.05	Highest residue	89	0.06	*50*	*40*	*80*			
Rye grain	GC	0.05	Highest residue	88	0.06	*40*	*40*	*50*			
Sorghum grain	GC	0.05	Highest residue	86	0.06	*40*	*40*	*80*			
Wheat grain	GC	0.05	Highest residue	89	0.06	*50*	*40*	*80*			
Pulse seed (Note)	VD	0.15	Highest residue	90	0.17	*20*	*20*	*20*			
Carrot culls	VR	0.02	STMR-P	12	0.17	*25*	*25*				
Potato culls	VR	0.02	STMR-P	20	0.10	*75*	*40*				
TOTAL		.				*100*	*100*	*100*	**22**	**38.1**	**0.08**

Consumption value from soya bean forage

Consumption value from pea seed

Estimated mean dietary burden of farm animals

Commodity	CC	Residue (mg/kg)	Basis	DM %	Residue ÷ DM	*Diet content (%)*			Residue contribution, mg/kg		
						Beef cattle	*Dairy cows*	*Poultry*	Beef cattle	Dairy cows	Poultry
Apple wet pomace	AB	0.3	STMR-P	40	0.75	***40***	***20***		0.3	0.15	
Pea, field hay	AL	16.4	STMR	100	16.40	*25*	***50***			8.2	
Bean forage (Note)	AL	5.25	STMR	35	15.00	***30***	*30*		4.5		
Barley straw	AS	0.015	STMR	89	0.02	*10*	*60*				
Millet straw	AS	0.015	STMR	90	0.02	*10*	*10*				
Oats straw	AS	0.015	STMR	90	0.02	*10*	*10*				
Rye straw	AS	0.015	STMR	88	0.02	*10*	*10*				
Sorghum stover	AS	0.015	STMR	88	0.02	*25*	*15*				
Wheat straw	AS	0.015	STMR	88	0.02	*10*	*10*				
Sugar beet tops	AV	0.76	STMR	23	3.30	***20***	***10***		0.66	0.33	
Barley grain	GC	0.01	STMR	88	0.01	*50*	*40*	*75*			
Corn grain	GC	0.01	STMR	88	0.01	*80*	*40*	*80*			
Corn, pop grain	GC	0.01	STMR	88	0.01	*80*	*40*	*80*			
Millet grain	GC	0.01	STMR	88	0.01	*50*	*40*	*70*			
Oats grain	GC	0.01	STMR	89	0.01	*50*	*40*	*80*			

Commodity	CC	Residue (mg/kg)	Basis	DM %	Residue ÷ DM	*Diet content (%)*			Residue contribution, mg/kg		
						Beef cattle	*Dairy cows*	*Poultry*	Beef cattle	Dairy cows	Poultry
Rye grain	GC	0.01	STMR	88	0.01	*40*	*40*	*50*			
Sorghum grain	GC	0.01	STMR	86	0.01	*40*	*40*	*80*			
Wheat grain	GC	0.01	STMR	89	0.01	*50*	*40*	***80***			0.009
Pulse seeds (Note)	VD	0.075	STMR	90	0.08	***10***	***20***	***20***	0.008	0.017	0.017
Carrot culls	VR	0.01	STMR-P	12	0.08	*25*	*25*				
Potato culls	VR	0.01	STMR-P	20	0.05	*75*	*40*				
TOTAL		.				*100*	*100*	*100*	**5.47**	**8.7**	**0.026**

Consumption value from soya bean forage

Consumption value from pea seed

Farm animal feeding studies

The Meeting received information on feeding studies with lactating cows and laying hens.

A residue transfer study in livestock was conducted with four groups of three Friesian cows that were fed for 28 to 29 days with diets containing pirimicarb. Pirimicarb was applied as a spray to grass 'nuts' tumbling in a drum of a cement mixer. The treated grass nuts were mixed with untreated grass nuts and hay to obtain an actual total feed intake of 18 kg/cow per day. Actual pirimicarb levels in the treated nuts were 423 ppm corresponding to actual feeding levels of 0, 24, 71 and 235 ppm. One cow from each group was slaughtered on day 28 and one cow on day 29, each within 24 hours of the final dose. The remaining cow from each group was maintained on a control diet for a further 7 days before slaughter. Milk was collected at morning and afternoon milking at 2–3 day intervals throughout the study. Liver, kidney muscle, and fat (subcutaneous, peritoneal) were taken for analysis.

No parent was found at any of the feeding levels (< 0.04 mg/kg). R34386 (including R34855) was only found at the highest feeding level (235 ppm) in the range < 0.02–0.088 mg/kg. Residues did not accumulate and declined rapidly when pirimicarb feeding ceased. No parent and no R34836 (including R34855) were found at any of the feeding levels in kidney and liver (< 0.01 mg/kg). Parent and metabolite R34386 (including R34855) were only occasionally found in muscle or fat at levels up to 0.02 mg/kg. Milk samples from control animals were < 0.005 mg/kg for each analyte, except for the day 3 milk sample, where a value of 0.01 mg/kg was found for parent and day 17 and day 26 milk samples, where a value of 0.005 mg/kg was found for R34836.

A residue transfer study in laying hens was conducted with four groups of 40 laying hens + four cockerels. The hens were fed for up to 28 days with basal layers' diet containing pirimicarb at actual feeding levels of 0.083, 1.5, 4.6 and 14.3 ppm parent eq, followed by a recovery period of 14 days on untreated feed. Eggs (10 per treatment group) were collected on days 1, 3, 7, 11, 15, 21, 25 and 27 (treatment period) and days 31, 35, 39 and 42 (post-treatment period). Eggs were separated into whites and yolks. On each day, the white and yolk samples from each group were pooled. Five hens from each group were sacrificed on days 21, 28, 35 and 42 of the trial. No residues were found in pooled egg yolk and pooled egg white samples (< LOQ for each analyte) at any feeding level. No residues were found in pooled composite tissue samples (muscle, skin with fat) at any feeding level (< LOQ for each analyte). Residues in liver were at or below the LOQ: < 0.01 to 0.01 mg/kg for parent and < 0.04 to 0.04 mg/kg for R34836 (including R34855).

Residues in animal commodities

In the feeding study where lactating cows were dosed at 24 and 71 ppm, no pirimicarb residues were detected in tissues and milk. Therefore no residues are to be expected at the maximum calculated dietary burden of 22 mg/kg feed for beef cattle and 38 mg/kg for dairy cattle.

In the feeding study where laying hens were dosed at 1.5, 4.6 and 14.3 ppm, no pirimicarb residues were detected in tissues and eggs. No residues are to be expected at the maximum calculated dietary burden of 0.08 mg/kg feed for poultry.

The Meeting estimated a maximum residue level of 0.01* mg/kg in meat (from mammals except marine mammals), to replace the existing recommendation of 0.05 (*) mg/kg, and estimated HRs and STMRs of 0 mg/kg.

The Meeting also estimated a maximum residue level of 0.01* mg/kg in edible offal (mammalian) and estimated HRs and STMRs of 0 mg/kg.

For milks, the Meeting estimated a maximum residue level of 0.01* mg/kg to replace the existing recommendation of 0.05 (*) mg/kg, and estimated an STMR of 0 mg/kg.

The Meeting estimated a maximum residue level of 0.01* mg/kg in poultry meat, poultry offal and eggs and estimated HRs and STMRs of 0 mg/kg.

RECOMMENDATIONS

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

Definition of the residue (for both plant and animal commodities) for compliance with MRLs: *pirimicarb.*

Definition of the residue for estimation of dietary intake: *Sum of pirimicarb, and its demethyl and demethyl formamido metabolites, expressed as pirimicarb for plant commodities and pirimicarb (parent compound only) for animal commodities.*

The residue is not fat soluble

CCN	Commodity Name	MRL (mg/kg) New	MRL (mg/kg) Previous	STMR or STMR-P (mg/kg)	HR or HR-P (mg/kg)
AL 1020	Alfalfa fodder	W	20 dry wt		
AL 1021	Alfalfa forage (green)	W	50 dry wt		
JF 0226	Apple juice			0.13	
	Apple sauce			0.09	
	Apple wet pomace			0.3	
VS 0620	Artichoke, Globe	5		0.69	2.8
VS 0621	Asparagus	0.01 (*)		0	0
GC 0640	Barley	W note	0.05 (*)	0.01	0.05
AL 1030	Bean forage (green)			5.25	7
VP 0062	Beans, shelled	W note	0.1		
VR 0574	Beetroot	W note	0.05 (*)		
FB 0018	Berries and other small fruits (1)	1		0.36	0.82
FB 0264	Blackberries	W note	0.5		
VB 0040	Brassica (cole or cabbage) vegetables, head cabbages, Flowerhead brassicas	0.5		0.05	0.5
VB 0400	Broccoli	W note	1		
VB 0402	Brussels sprouts	W note	1		
VB 0041	Cabbages, Head	W note	1		
VB 0404	Cauliflower	W note	1		
VS 0624	Celery	W	1		
CG 0080	Cereal grains (2))	0.05		0.01	0.05

CCN	Commodity Name	MRL (mg/kg) New	MRL (mg/kg) Previous	STMR or STMR-P (mg/kg)	HR or HR-P (mg/kg)
	Chilli peppers (dried)	5		1.05	2.5
FC 0001	Citrus fruits	3		0.015	0.08
FC 0001	Citrus fruits (except oranges)	W note	0.05 (*)		
VP 0526	Common bean (pods and/or immature seeds)	W note	1		
SO 0691	Cotton seed	W	0.05 (*)		
VC 0424	Cucumber	W note	1		
FB 0278	Currant, Black	W note	0.5		
DF 0014	Prunes			0.46	0.86
VO 0440	Egg plant	W note	1		
PE 0112	Eggs	0.01 (*)	0.05 (*)		
VL 0476	Endive	W	1		
VC 0045	Fruiting vegetables, cucurbits (3)	1		0.18	0.44
VO 0050	Fruiting vegetables, other than cucurbits (4)	0.5		0.105	0.25
VA 0381	Garlic	0.1	0.5	0.01	0.09
VC 0425	Gherkin	W note	1		
VL 0480	Kale	0.3		0.31	0.6
VB 0405	Kohlrabi	W note	0.5		
VA 0384	Leek	W	0.5		
VP 0060	Legume vegetables (5)	0.7		0.27	0.59
VL 0482	Lettuce, Head	5	1	2	3
VL 0483	Lettuce, leaf	5		2	3
AF 0645	Maize forage			0	0
MO 0105	Edible offal (Mammalian)	0.01 (*)		0	0
MM 0095	Meat (from mammals ex marine mammals)	0.01 (*)	0.05 (*)	0	0
VC 0046	Melons, except Watermelon	0.2		0.025	0.09
ML 0106	Milks	0.01 (*)	0.05 (*)	0	0
GC 0647	Oats	W note	0.05 (*)		
VA 0385	Onion, Bulb	0.1	0.5	0.01	0.09
FC 0004	Oranges, Sweet, Sour	W note	0.5		
HH 0740	Parsley	W	1		
VR 0588	Parsnip	W note	0.05 (*)		
AL 0072	Pea hay or Pea fodder (dry)	60 dry wt		16.4 dry wt	72 dry wt
FS 0247	Peach	W note	0.5		
VP 0063	Peas (pods and succulent=immature seeds)	W note	0.2		
TN 0672	Pecan	W	0.05 (*)		
VO 0444	Peppers, Chili	W note	2		
VO 0445	Peppers, Sweet (incl Pimento or pimiento)	W note	1		
FS 0014	Plums (including prunes)	W note	0.5		
FP 0009	Pome fruits	1	1	0.18	0.91
VR 0589	Potato	W note	0.05 (*)	0	0
PM 0110	Poultry meat	0.01 (*)		0	0
PO 0111	Poultry, Edible offal of	0.01 (*)		0	0
VD 0070	Pulses (6)	0.2		0.075	0.15
VR 0494	Radish	W note	0.05 (*)		
SO 0495	Rape seed	0.05	0.2	0.01	0.02
FB 0272	Raspberries, Red, Black	W note	0.5		

CCN	Commodity Name	MRL (mg/kg) New	MRL (mg/kg) Previous	STMR or STMR-P (mg/kg)	HR or HR-P (mg/kg)
VR 0075	Root and tuber vegetables	0.05		0.01	0.02
VL 0502	Spinach	W	1		
FS 0012	Stone fruits	3		0.99	2.1
AS 0081	Straw and fodder (dry) of cereal grains (7)	0.3 dry wt		0.015	0.33
FB 0275	Strawberry	W	0.5		
VR 0596	Sugar beet	W note	0.05 (*)	0.01	0.02
AV 0596	Sugar beet leaves or tops			0.76	4.3
SO 0702	Sunflower seed	0.1		0.015	0.07
VO 0447	Sweet corn (corn-on-the-cob)	W	0.05 (*)		
VO 1275	Sweet corn (kernels)	0.05		0.01	0.02
VO 0448	Tomato	W note	1	0.12	0.31
JF 0048	Tomato juice			0.07	
	Tomato puree			0.16	
VR 0506	Turnip, Garden	W note	0.05 (*)		
VL 0473	Watercress	W	1		
GC 0654	Wheat	W note	0.05 (*)	0	0

(1) excludes strawberries and grapes

(2) excludes rice

(3) excludes melons and water melons)

(4) excludes edible fungi and sweetcorn (both kernels and corn-on-the-cob)

(5) excludes soya beans

(6) excludes soya bean (dry)

(7) excludes rice straw and fodder, dry

(*) = the MRL is estimated at or about the LOQ

W = Withdrawn

note = Replaced by other MRLs for a wider group of commodities

DIETARY RISK ASSESSMENT

Long term intake

The evaluation of pirimicarb has resulted in recommendations for MRLs and STMRs for raw and processed commodities. Consumption data were available for 53 food commodities and were used in the dietary intake calculation. The results are shown in Annex 3 of the 2006 JMPR Report.

The International Estimated Daily Intakes in the 13 GEMS/Food cluster diets, based on the estimated STMRs were in the range 1–10% of the maximum ADI of 0.02 mg/kg bw (Annex 3). The Meeting concluded that the long-term intake of residues of pirimicarb ((including the demethyl carbamate metabolites) from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intake (IESTI) for pirimicarb was calculated for the food commodities (and their processing fractions) for which maximum residue levels and HRs were estimated and for which consumption data were available. The results are shown in Annex 4 of the 2006 JMPR Report.

The IESTI varied from 0–40% of the ARfD (0.1 mg/kg bw) for the general population. The IESTI varied from 0–70% of the ARfD for children 6 years and below. The Meeting concluded that the short-term intake of residues of pirimicarb (including the demethyl carbamate metabolites) from used considered by the Meeting was unlikely to present a public health concern.

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PROPAMOCARB (148)

First draft prepared by Eloisa Dutra Caldas, University of Brasilia, Brasilia, BRAZIL

EXPLANATION

Propamocarb hydrochloride is a carbamate fungicide with specific activity against Oomycete species that cause seed, seedling, root, foot and stem rots and foliar diseases in a number of edible crops. The compound was evaluated by the JMPR in 1984, 1986, 1987 and 2005, when an ADI of 0–0.4 mg/kg bw and an ARfD of 2 mg/kg bw were established. At the 37th Session of the CCPR it was scheduled for residue evaluation, within the periodic review programme, by the 2006 JMPR. The manufacturer submitted data on metabolism in animal and plants, degradation in soil, residues in succeeding crops; GAP, analytical methods and processing studies. Residue trials submitted were conducted on potato, radish, onion, lettuce, spinach, cabbage, cauliflower, chicory, sweet pepper, tomatoes, summer squash, cantloupe and melon. GAP information and residue trials results on lettuce, cucumber and ginger were provided by the Government of Japan.

IDENTITY

Common name: Propamocarb hydrochloride

Chemical name:

IUPAC: Propyl 3-(dimethylamino) propylcarbamate hydrochloride

CAS: Propyl [3-(dimethylamino)propyl] carbamate hydrochloride

CAS number: 25606-41-1

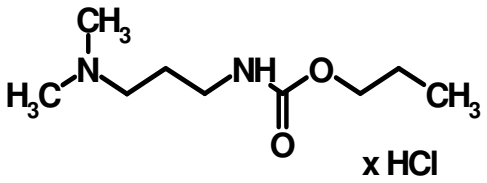
CIPAC number: 399

EEC number: 245-125-9

Molecular formula: $C_9H_{21}ClN_2O_2$

Molecular mass: 224.7 g/mol

Structural formula:



PHYSICAL AND CHEMICAL PROPERTIES

A detailed chemical and physical characterisation of the active ingredient is given below.

Property	Results	Reference Report No.
Colour and odour	Cream coloured sticky crystals with typical carbamate odour/white opaque crystalline soft liquid with weak, sickly sweet odour	(Sixl/Rexer, 1998; C001715/C001717; Walker *et al*., 1995; 722/013)
Melting point	64.2°C	(Lehne, 1990; A89312)
Relative density	1.051 g/cm³ at 20°C/1.15 g/cm³ at 20.5 $\pm$0.5°C	(Bittner/Rexer, C003480. Muehlberger and Lemke, 2004; C044109. Walker *et al*., 1995; 722/013)
Vapour pressure (extrapolated)	3.8×10^{-5} / 1.4×10^{-3} Pa at 20 °C 8.1×10^{-5} / 1.7×10^{-3} Pa at 25 °C 1.6×10^{-4} Pa at 30 °C	(Miklautz, 1990; A85057; Howarth *et al*., 1995; 722/015)

Property	Results	Reference Report No.
Volatility (calculated)	Henry's law constant at 20 °C: 8.50 x 10^{-9} Pa m^3 mol^{-1}	(Renaud, 2005; C046819)
Solubility in water at 20ºC	> 900 g/L at pH 3 > 855 g/L at pH 6.9 > 536 g/L at pH 9.6 between 89.2 and 93.5%w/w at pH 4 between 89.1 and 93.8%w/w at pH 7 between 89.6 and 94.6%w/w at pH 10	(Muehlberger, 2001; C012641/C042353; Renaud, 2004; C045318 ; Walker *et al*., 1995 ; 722/013)
Solubility in organic solvent [g/L] at 20°C s	Hexane: < 0.01 Toluene: 0.14 Methanol: > 656 Dichloromethane: > 626 Ethyl acetate: 4.34 – 4.8 Acetone: 560.3 Xylene: 1.6x10^{-2} Heptane: < 1x10^{-4}	(Müller, 1990; A85046; Walker *et al*., 1995; 722/013; Ryckel, 2002; 20528)
Dissociation constant	pKa=9.3± 0.03 at 20°C pKa=9.63± 0.03 at 20°C	(Miklautz, 1991; A85060; Poerschke, 2001; C014007; Walker *et al*., 1995; 722/013)
Partition coefficient n-octanol/water	Log Pow at 22°C = -2.87 (at pH 2), -1.21 (at pH 7) and 0.67 (at pH 9) Log Pow at 21-22°C = -0.98 (at pH 4), -1.36 (at pH 7) and 0.32 (at pH 9)	(Muehlberger, 2004; C012642; Walker *et al*., 1995; 722/013)
Hydrolysis rate	< 10% hydrolysis after 5 days at 50°C at pH 4, 7 and 9	(Shepler et al., 2001; B003419; Walker *et al*., 1995 ; 722/013)
Photochemical degradation	No photo degradation of propamocarb HCl in aqueous solution by irradiation with artificial sunlight during 22 days	(Klehr, 2003; A85564/A85466; Mullee *et al*. 1995; 722/014)

METABOLISM AND ENVIRONMENTAL FATE

All the metabolism and environmental fate studies submitted to the Meeting were conducted with ^{14}C-propamocarb hydrochloride labelled as shown on Figure 1.

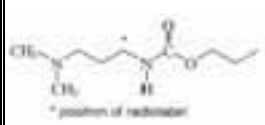
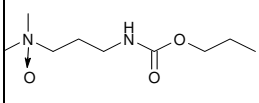
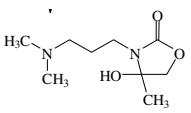
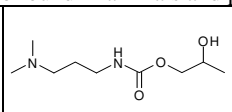
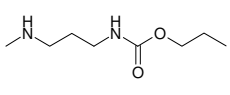
Parent compound	Metabolites of propamocarb found in animals and plants			
				
Labeled propamocarb	Propyl propamocarb N-oxide (Met IV)	Propamocarb oxazolidin-2-one (Met VI)	2-Hydroxy propamocarb	N-desmethyl propamocarb

Figure 1. Position of ^{14}C in propamocarb and the metabolites found in animals and plants.

Animal metabolism

Rat

Four studies conducted in rats with ^{14}C-propamocarb hydrochloride were submitted to the Meeting (Reynolds, 1994, A85144; O'Boyle, 1994, A85146/A91169; Reynolds, 1994, A85148/ A91170;

Morley, 1997, A8386, A84072/C000632). These studies were evaluated by the 2005 JMPR during the toxicological periodic review of propamocarb, and are detailed in the 2005 JMPR Toxicology Evaluation and Report. In summary, propamocarb was rapidly absorbed and extensively metabolised in rat, with no accumulation of parent compound or metabolites in tissues, which are mainly excreted in urine and faeces. Half-life for all tissues ranged from 11–26 hours, with 3 to 20% of the applied dose being excreted as parent compound. The proposed metabolism of propamocarb hydrochloride in the rat involved aliphatic oxidation of the propyl chain, N-oxidation of the tertiary amine and N-dealkyation. Four major metabolites were identified: 2-hydroxy propamocarb, mono-N-desmethyl propamocarb (AEB132677), propylpropamocarb N-oxide (Met IV) and the cyclic propamocarb oxazolidin-2-one (Met VI). There was no evidence of conjugation with glucuronic or sulfuric acid.

Livestock

A lactating cow was orally dosed twice daily for seven consecutive days at a dose level equal to 11.5 mg/kg [^{14}C]-propamocarb HCl equivalents in the diet. Based on dry weight of feed, this corresponds to 2.0 mg propamocarb HCl/kg body weight per day (Daniel and Rupprecht, 2000; B002935). Milk, faeces and urine were collected twice a day during the treatment period. Approximately 15 hours after the last dose, the cow was sacrificed and edible tissues (liver, kidney, muscle, fat and bile) were collected.

Samples of kidney, liver, and muscle were extracted 6 to 8 times with acidified methanol. Radioactivity in the extracts was directly counted by liquid scintillation counting (LSC). Total radioactive residues (TRR) in fat was 0.002 mg/kg propamocarb HCl eq. and no further extraction was performed. Liver and kidney extracts were directly subject to chromatography, but muscle extracts were 'de-fatted' previously with hexane. Milk was extracted with hexane to remove the fat before being dialysed with water. Faeces were extracted with acidic methanol followed by soxhlet extraction with acidic methanol. Identification and quantification of the metabolites in the extracted residue was accomplished by reverse phase and cation exchange HPLC. Samples were analyzed within 2–6 months after collection.

The majority of the administered dose was excreted (81.4%), via the urine (71.9%) and the faeces (9.5%). An overall recovery (including stall wash) of 82.9% of the administered dose was achieved. The residues in the milk were always higher in the afternoon, with a mean of 0.054 ± 0.008 mg/kg propamocarb HCl eq (n=7), and a maximum of 0.057 mg/kg on Day 6 than in the morning (mean: 0.035 ± 0.003 mg/kg propamocarb HCl eq. (n=7) and the maximum of 0.037 mg/kg on Day 5). Cumulative radioactivity recovered in the milk (0.599 mg/kg) accounted for 0.46% of the administered dose. TRR found in tissues and bile accounted for 0.7% of the administered dose. Radioactivity found in tissues, milk and faeces are summarized in Tables 1 and 2. Unextracted residues were not analyzed further.

Table 1. Extractability of residues in tissues, milk and faeces.

Matrix	TRR	Extracted Residue		Unextracted Residue	
	mg/kg[a]	%TRR	mg/kg[a]	%TRR	mg/kg[a]
Kidney	0.107	92.5	0.099	7.2	0.008
Liver	0.415	96.4	0.4	3.6	0.015
Milk[b]	0.057	100	0.057	NA	NA
Muscle	0.019	83.2	0.016	16.8	0.003
Milk fat[b]	< 0.01	NA	NA	NA	NA
Faeces[c]	NA	93.6	-	6.3	-

a. Expressed as propamocarb HCl equivalents; b. sample from day 6 afternoon; c. days 4 and 5; NA= not analysed

The majority of the residue comprised propamocarb, propamocarb N-oxide (Met IV), and the cyclic propamocarb oxazolidin-2-one (Met VI). Minor amounts of 2-hydroxy propamocarb and desmethyl propamocarb (AE B132677) were also identified (Table 2; Figure 1). The majority of the residue was identified in all matrices.

Table 2. Summary of metabolite identification in tissues, milk and excreta.

	Propamocarb		Propyl propamocarb N-oxide (Met IV)		Propamocarb oxazolidin-2-one (Met VI)		2-Hydroxy propamocarb		N-desmethyl propamocarb		Identified	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Kidney	23.5	0.025	40.8	0.044	14.1	0.015	13	0.014	nd	nd	91.4	0.098
Liver	6.2	0.026	49.0	0.203	21.7	0.09	4.8	0.02	nd	nd	81.7	0.339
Milk	6.0	0.003	21.3	0.012	23.4	0.014	37.6	0.022	3.4	0.002	91.7	0.053
Muscle	24.6	0.005	40.5	0.008	2.3	< 0.001	0.9	< 0.001	4.1	0.001	72.4	0.014
Faeces	33.7	NA	24.6	NA	2.0	NA	13.1	NA	7.6	NA	81	NA
Urine	1.2	NA	28.2	NA	59.0	NA	9.9	NA	NA	NA	98.3	NA

Figure 2 shows the proposed metabolic pathway for propamocarb hydrochloride in the cow. The compound is oxidised or N-demethylated at the di-methyl amine group, or is hydroxylated at the propyl side chain, with the subsequent cyclization to form propamocarb oxazolidin-2-one (metabolite VI).

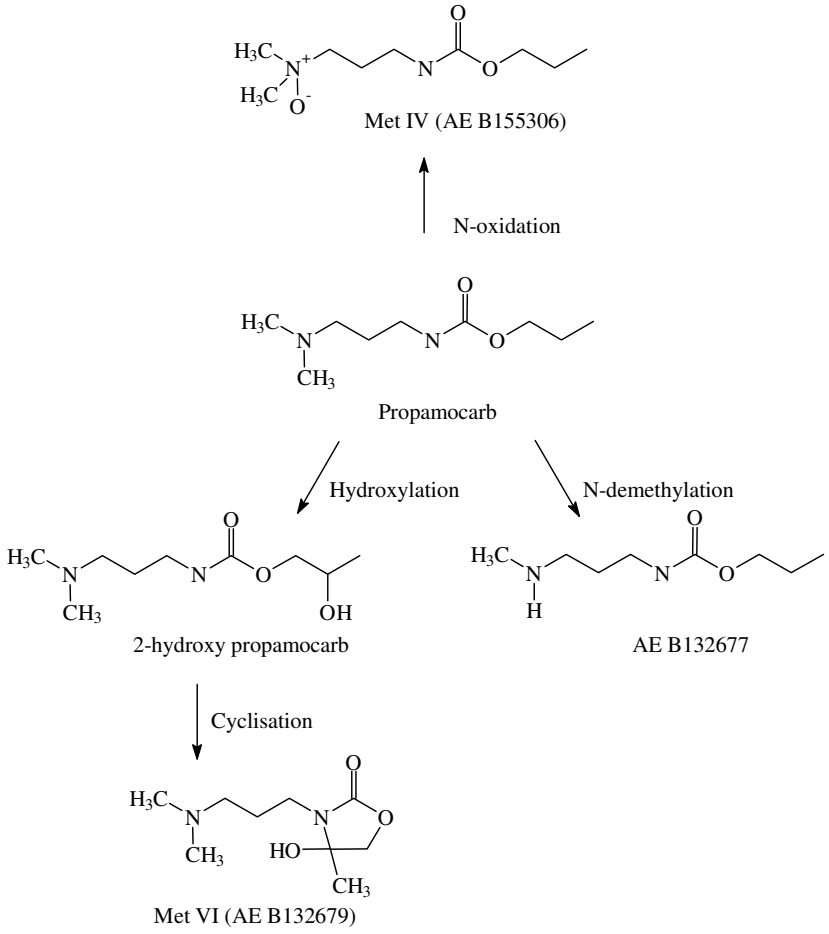


Figure 2. Proposed metabolic pathway of propamocarb hydrochloride in the cow.

Plant metabolism

Spinach

In a study conducted in USA in 2000, [^{14}C]-propamocarb HCl was applied twice to spinach by foliar spray at a rate of 2.53 kg ai/ha (Rupprecht and Daniel, 2000; B002936)**.** Duplicate samples were harvested immediately following the 1st application (day 0), just prior to the second application (Day 20) and 3 days after the second application (Day 23). Samples were extracted three times with methanol/1M hydrochloric acid (99:1), the extract filtered and the ^{14}C content determined by LSC. The filter cake was extracted with acidic methanol in a soxhlet system. Sample extracts were analysed by HPLC and TLC using a radioactive detector. Propamocarb and a selected number of targeted metabolites were used as external standards to identify the residues present. The results are presented in Table 3. Propamocarb was the main residues found in all samples collected.

Table 3. Distribution of metabolites in spinach extracts.

Sample time	TRR, mg/kg[a]	Propamocarb, %TRR	Propyl propamocarb N-oxide (Met IV),%TRR	Propamocarb oxazolidin-2-one (Met VI),%TRR	2-Hydroxy propamocarb, %TRR	N-desmethyl propamocarb, %TRR	Total identified, %TRR
Day 0	203.0	89.2	2.2	1.8	0.0	0.0	93.2
Day 20	207.3	76.0	3.5	2.6	7.1	3.6	92.7
Day 23	236.9	83.1	3.6	2.8	5.4	1.1	96.1

a. Expressed as propamocarb HCl equivalents; mean values from duplicate samples

Lettuce

In one study conducted in UK in 2002, [^{14}C]-Propamocarb HCl (>98% radiochemical purity) was applied (a) to soil on which lettuce was grown three times at 7.22 g ai/m^2, corresponding to 72.2 kg ai/ha immediately after sowing and at intervals of 14 and 28 days thereafter, and (b) three times as a foliar spray in a greenhouse at 1.08 kg ai/ha with 10 day intervals (Goodyear, 2002a; 16669/6-D2149).

Plants were harvested at mature size 38 days after the final soil treatment and 21 days after final foliar treatment. Samples were homogenized in dry ice and stored frozen until analyzed. Portions (approximately 20g) of the homogenates were extracted with methanol, the extracts centrifuged and the radioactivity present determined by LSC. The plant residue remaining was air-dried and the unextracted radioactivity determined by LSC following combustion. The results are shown in Table 4.

Table 4. Radioactive residues in lettuce after soil and foliar treatment.

Treatment, days after the last treatment	TRR (mg/kg)[a]	Methanol (mg/kg)[a]	Methanol (%)	Unextracted (mg/kg)[a]	Unextracted (%)
Soil, 38 days	10.7	3.8	35.5	6.9	64.5
Foliar, 21 days	9.51	8.04	84.3	1.47	15.5
Control	0.35	0.10	29.1	0.24	70.9

a. As propamocarb HCl equivalents.

Larger sub-samples (about 100 g) were extracted sequentially with methanol and water. The plant residue remaining was re-extracted by refluxing with 2M HCl and with 2M NaOH. The liquid extracts in each case were separated by centrifugation and the radioactivity present determined by

LSC. The unextracted radioactivity accounted for 7.1 and 0.2% of the total residue in soil and foliar treated sample respectively (Table 5).

Table 5. Distribution of radioactivity in extracts of lettuce.

Treatment[a]	TRR (mg/kg)	Methanol (mg/kg)	(%)	Water (mg/kg)	(%)	2M HCl (mg/kg)	(%)	2M NaOH (mg/kg)	(%)	Unextracted (mg/kg)	(%)
Soil	8.19	4.37	53.3	0.95	11.7	1.58	19.3	0.71	8.6	0.58	7.1
Foliar	10.7	9.76	91.5	0.69	6.5	0.15	1.4	0.04	0.4	0.02	0.2
Control	0.292	0.20	68.4	NA	NA	0.05	16.9	0.03	11.2	0.01	3.5

a. After 38 days of the last soil treatment and 21 days after foliar application; NA = not analyzed

Methanol and water extracts of plants following soil and the foliar treatments, containing 65% and 98% of TRR, were analysed by HPLC. Table 6 shows that propamocarb formed only in a small proportion of the residue (2.8%) in the lettuce plants grown in treated soil. The residue was composed mainly of an unidentified polar region, Unknown 1. Extracts from the foliar treated samples showed predominantly unchanged propamocarb (90.2%) (Table 7).

Table 6. Profile of radioactive residues in (^{14}C)-propamocarb soil treated lettuce.

Compound	Methanol Extract		Water Extract		Total	
	Residue (mg/kg)	%TRR	Residue (mg/kg)	% TRR	Residue (mg/kg)	%TRR
Propamocarb	0.215	2.6	0.015	0.2	0.230	2.8
Unknown 1	3.650	44.6	0.811	10.0	4.461	54.6
Unknown 4	0.158	1.9	ND	ND	0.158	1.9
Unknown 8	0.274	3.4	0.069	0.8	0.343	4.2
Unknown 10	ND	ND	0.050	0.6	0.050	0.6
Unallocated	0.070	0.8	0.010	0.1	0.080	0.9
Extracted residue	4.367	53.3	0.954	11.7	5.321	65.0
			2M HCl reflux		1.579	19.3
			2M NaOH reflux		0.708	8.6
			Unextracted		0.580	7.1
			TRR = 8.19mg/kg			

Table 7. Profile of radioactive residues in (^{14}C)-propamocarb foliar treated lettuce.

Compound	Methanol Extract		Water Extract		Total	
	Residue (mg/kg)	Percent TRR	Residue (mg/kg)	Percent TRR	Residue (mg/kg)	Percent TRR
Propamocarb	9.016	84.6	0.599	5.6	9.615	90.2
Unknown 1	0.081	0.8	0.053	0.5	0.134	1.3
Unknown 4	0.284	2.7	0.019	0.2	0.303	2.9
Unknown 7	0.318	3.0	0.020	0.2	0.338	3.2
Unallocated	0.057	0.5	0.001	< 0.1	0.058	0.5
Extracted residue	9.756	91.5	0.692	6.5	10.448	98.0
			2M HCl reflux		0.151	1.4
			2M NaOH reflux		0.043	0.4
			Unextracted		0.020	0.2
			TRR = 10.662mg/kg			

A total radioactive residue of 0.346 mg/kg was observed in samples from the untreated control lettuce, 29% of which was extracted with methanol. More exhaustive extraction of a second sub-sample of control lettuce using methanol, water, acid and base reflux, showed that 68% of the total residue was extracted with methanol, 17% with acid and 11% with base.

Potato

In a greenhouse study conducted in 1989 in Germany, [^{14}C]-Propamocarb hydrochloride was applied three times to potato plants, at a rate corresponding to 2.45 kg ai/ha (approximately 20 days between applications) (Förtsch, 1991; A85140).

Potatoes samples were harvested 6 weeks after the final application, extracted using acidified methanol and the radioactivity of the combined extracts measured by LSC. On average, 45.5% TRR was found in the extracts (10 samples), corresponding to a TRR of 0.82 mg/kg propamocarb HCl equivalents. The ^{14}C residue present was equally distributed between peel and flesh (0.96 mg/kg and 0.84 mg/kg, respectively). Control potatoes which were grown in the vicinity of the treated plants contained up to 0.3 mg/kg of propamocarb equivalents, the bulk of which was not extracted with acidified methanol.

Further extraction and partitioning of the extracted residue into chloroform was conducted, following the acidification and alkalisation of the extracts. Between 22 to 31% of the residue partitioned into the aqueous phase (mean = 25.5%) while 14 to 29% of the residue (mean = 23.6%) was present in the organic fraction (n=6). HPLC analysis of the crude methanol extract of sample No. 10 indicated that propamocarb was the main component, representing 58.4% of the total radioactivity extracted (Table 8). The identities of the metabolites shown on Table 8 were not confirmed in the study, but M1 had the same chromatographic behaviour as propyl propamocarb-N-oxide (Met IV).

Table 8. Extracted resides in potato treated with propamocarb.

	Crude methanol extract		After purification	
	% TRR*	mg/kg prop HCl equ.	Chloroform,% TRR*	Aqueous,% TRR*
Propamocarb HCl	27.8	0.23	11.8	1.5
M1	8.6	0.07	2.3	18.8
M2	7.2	0.06	3.5	-
M3	2.0	0.02	1.6	-
M4	-	-	2.5	1.4
Undefined region	2.0	-	0.6	3.6
Total	47.6	0.32	22.3	25.3

* % of the total amount of recovered radiolabel residues of sample No. 10 (47.6% TRR)

In another greenhouse study conducted in German in 1994, potato plants were treated as in the previous study and tubers harvested about 6 weeks after the final treatment (Förtsch, 1994; A85141). Samples were macerated and soxhlet extracted with acidified methanol or acetonitrile followed by alkaline and acid hydrolysis of the remaining material. About 90% of the radiolabeled material was recovered by this method. One sample containing 1.12 mg/kg propamocarb eq, had 31.8% of this residue extracted by acetonitrile and 6.6% unextracted. Table 9 shows the chromatographic profile of this sample using different HPLC elution systems. About 7% of TRR was identified as the parent compound in the two HPLC systems, about 50% of TRR showed the same chromatographic behavior as radiolabeled natural products formed from the exposure of spinach plants with $^{14}CO_2$ gas, and identified as d-glucose.

Table 9. Metabolic patterns of the extracted residues present in plant extracts.

HPLC peak i.d.	Retention time (min)	Macerate extract (%)	Soxhlet extract (%)	HCl hydrolysate (%)	NaOH hydrolysate (%)	Total (%)
Normal phase HPLC analysis of potato tubers, System 1						
Si-1	2- 3	0.17	0.6	1.84	5.86	8.47
Si-2 – d-glucose	3.5-6	5.62	6.18	28.46	13.58	53.84
Si-3	6.5-7	0.14	1.79	1.89	0.5	4.32
Si-4 – propamocarb HCl	8- 10.5	2.75	4.41	n.d.	n.d.	7.16
Si-5	11- 12.5	0.07	0.38	n.d.	n.d.	0.45
Si-6	13- 15	0.35	0.67	n.d.	n.d.	1.02
Si-7	15.5- 18	0.19	n.d.	n.d.	n.d.	0.19

HPLC peak i.d.	Retention time (min)	Macerate extract (%)	Soxhlet extract (%)	HCl hydrolysate (%)	NaOH hydrolysate (%)	Total (%)
Si-8	22- 25	0.06	0.11	n.d.	n.d.	0.17
Total characterized		9.95	14.14	32.19	19.94	75.62
Unassigned ^{14}C		1.6	0.5	6.2	2.9	11.2
% of ^{14}C lost		1.1	5.1	--	--	6.2
% of ^{14}C recovered.		12.1	19.7	38.4	22.8	93
Reverse phase HPLC analysis of potato tubers, System 2						
RP-1 - d-glucose	2 – 4.5	4.83	5.89	27.38	7.4	45.5
RP-2	5.5- 8.5	1.01	0.96	1.61	4016	7.74
RP-3	9 – 11.5	0.72	0.74	1.18	1.16	3.8
RP-4	14 – 16.5	0.32	0.56	1.1	3.53	5.51
RP-5	17 – 19.5	0.29	0.28	n.d.	0.5	1.07
RP-6	20 – 22	0.31	0.64	n.d.	n.d.	0.95
RP-7 propamocarb HCl	23.5 – 25.5	2.5	3.26	0.68	0.47	6.91
RP-8	26 - 28	0.5	1.07	2.03	1.79	5.39
Total characterized		10.47	13.4	33.98	19.01	76.86
Unassigned ^{14}C		0.5	1.2	4.4	3.8	9.9
% of ^{14}C lost		1.1	5.1	--	--	6.2
% of ^{14}C recovered.		12.1	19.7	38.4	22.8	93

In a third study conducted with potato in the UK in 2002 (Goodyear, 2002b; 1669/5-D2149) [^{14}C]-Propamocarb (> 98% radiochemical purity) was applied 6 times as a foliar spray to potatoes grown outdoors in crates at a rate of 2.2 kg ai/ha and at 10.8 kg ai/ha. Initially the treatment solution was applied to the foliage and the drift to soil was small, however by the sixth application the foliage had died back to such an extent that the majority of the treatment solution was sprayed on soil.

Samples were harvested when the tubers reached maturity, about 7 days after the last treatment, or 161 days after sowing. Samples of tubers were washed with water and divided equally into two samples, one of which was peeled. Samples of foliage and roots were also taken.

Fresh sub-samples of the whole tuber, peel, flesh and foliage were extracted sequentially with methanol, water and refluxed in 2M HCl acid and 2M NaOH base. The liquid extracts in each case were separated by centrifugation and the radioactivity present determined by LSC (Table 10).

Table 10. Distribution of residues of [^{14}C]-propamocarb in extracts of parts of treated potato.

Extract	Whole tuber		Peel		Flesh		Foliage	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
2.2 kg ai/ha								
Methanol	48.5	0.054	59.2	0.029	60.2	0.013	38.7	33.260
Water	27.3	0.031	18.6	0.009	15.7	0.003	14.7	12.670
2M HCl	19.4	0.022	11.5	0.006	15.2	0.003	20.4	17.511
2M NaOH	ND	ND	ND	ND	ND	ND	13.9	11.952
Unextracted Residue	4.8	0.005	10.7	0.005	9.0	0.002	12.2	10.520
	TRR =0.112mg/kg		TRR =0.05mg/kg		TRR =0.02mg/kg		TRR =85.9mg/kg	
10.8 kg ai/ha								
Methanol	39.2	0.02	59.9	0.119	52.0	0.164	52.5	249.8
Water	32.3	0.016	16.4	0.033	18.7	0.059	15.5	73.6
2M HCl	22.0	0.011	13.3	0.027	21.2	0.067	14.0	66.62
2M NaOH	ND	ND	ND	ND	ND	ND	11.7	55.93
Unextracted Residue	6.6	0.003	10.3	0.021	8.1	0.025	6.3	30.0
	TRR =0.050 mg/kg		TRR =0.199 mg/kg		TRR =0.316 mg/kg		TRR =476.0 mg/kg	

The methanol, water and acid extract of whole tuber and foliage from the lower rate treatment were analysed by HPLC, TLC and LC-MS (Table 11). Only a small proportion of the residue (< 2% TRR) in whole tuber was identified as propamocarb. The residue was composed predominantly of an

unidentified region (Unknown 1) and five smaller regions containing each ≤ 6% of TRR. In foliage extracts, a greater proportion of the residue was present as unchanged propamocarb (29%). The remainder of the residue showed a similar pattern of metabolites to those seen in the tuber.

HPLC with positive ion mass spectrometry analysis using reference standards tentatively identified the compounds 4, 6 and 7 as being a hydroxypropyl-propamocarb, N-methyl-propamocarb and propamocarb-N-oxide. There was no indication which carbon atom in the propane chain has been hydroxylated. Unknown compounds 2, 3, 5, 8, 9 and 10 were not identified.

Table 11. Total amounts of propamocarb and metabolites in potato tuber and foliage.

Compound	Whole tuber		Foliage	
	Residue (mg/kg)	% TRR	Residue (mg/kg)	% TRR
Propamocarb	0.002	1.9	24.506	28.6
Unknown 1	0.087	77.4	25.843	30.0
Unknown 3	< 0.001	0.4	0.675	0.8
Compound 4: Hydroxypropyl propamocarb	0.001	0.5	1.188	1.4
Unknown 5	0.006	6.0	1.095	1.3
Compound 6: N-methyl propamocarb	ND	ND	4.838	5.7
Compound 7: Propamocarb N-oxide	0.004	3.2	3.543	4.1
Unknown 8	ND	ND	0.359	0.4
Unknown 9	ND	ND	0.567	0.7
Unknown 10	0.001	0.8	ND	ND

The nature of the metabolites was investigated using the water extract of foliage treated at the higher rate. The radioactivity present in the water extract was composed mainly of the polar materials (Unknown 1 and Unknown 5). These materials were isolated and subjected to different treatments: 2M HCl at 60 ºC; β-glycosidase in 0.1M ammonium acetate (pH5) at 37 ºC; cellulase in 0.1M ammonium acetate (pH5) at 37 ºC; hesperidinase in 0.1M ammonium formate (pH3.8) at 37ºC; 0.1M ammonium acetate (pH5) at 37 ºC. Between 91−106% radioactivity was recovered following each treatment. The reaction products analysed by HPLC showed that the treatments had no observable effect on the nature of the radioactivity present and no unchanged propamocarb was released.

Cucumbers

In a greenhouse study, conducted in Germany in 1998, cucumbers were grown in soil treated once with [^{14}C]-propamocarb HCl applied at 2.9 kg ai/ha (11.8 mg ai/plant) and samples harvested at 30 days PHI. Hydroculture-grown cucumbers were treated once at a rate of 53.4 mg ai/plant, applied directly to the hydroponic solution and samples were harvested at 21 days post-application. Analysis of the hydroponic nutrient solution used to feed the cucumber plants showed that propamocarb hydrochloride was the only ^{14}C active compound present. Plant samples were separated into fruit, leaves/stems and roots and were analysed by LSC and by HPLC (Feyerabend and Rupprecht, 1998; A85149).

Cucumber samples were first extracted by maceration with methanol/1 M hydrochloric acid (99:1), centrifuged and the extracted solids re-extracted using the same solvent system in a soxhlet. Sample extracts were analysed using both normal phase and reverse phase HPLC conditions. The majority of the radioactivity was extracted by maceration (about 81% TRR) with unextracted residues representing < 8% of TRR. In the foliar treatment, propamocarb represented < 20% of TRR, and it was the major source of the extracted radioactivity in the hydroponic treatment (Table 12). The polar metabolites were not identified in the study. As part of the same study, sample extracts from spinach grown in a $^{14}CO_2$ enriched atmosphere were analysed in a similar manner and demonstrated that apart

from parent propamocarb the majority of the remaining ^{14}C residues detected were present as a result of the incorporation of ^{14}C into natural products.

Table 12. Extraction profile of cucumber fruit after soil and hydroponic treatments with propamocarb hydrochloride.

	Foliar (TRR = 0.069 mg/kg eq.) mg/kg eq. (% TRR)			Hydroponic (TRR = 3.09 mg/kg eq.) mg/kg eq. (% TRR)		
Acid methanol extract	Total in extract	Propamocarb HCl	Polar metabolites	Total in extract	Propamocarb HCl	Polar metabolites
Maceration	0.056 (81.2)	0.012 (17.4)	0.029 (42)	2.57 (83.3)	1.59 (51.4)	0.885 (28.6)
Soxhlet	0.008 (11.6)	0.0013 (1.9)	0.005 (7.2)	0.341 (11.0)	0.217 (7.0)	0.107 (3.5)
Unextracted	0.005 (7.2)	-	-	0.18 (5.8)	-	-

Tomatoes

In a greenhouse study conducted, in the UK in 2001, on tomato [^{14}C]-propamocarb (> 98% radiochemical purity) was applied 4 times to soil at rates of 0.007 and 0.036 kg ai/ha, and as a single foliar treatment at 2.2 kg ai/ha (Goodyear, 2001; 1669/3-D2149). Immature foliage (BBCH Stage 18, 8 true leaves unfolded) was harvested 7 days after the second soil treatment, i.e., 45 days after sowing. Mature tomatoes from the soil treatment were harvested at intervals of 14, 21, 28 and 35 days following the last application. Mature tomatoes, from foliar treated plants, were harvested at intervals of 7, 14, 21 and 28 days following application. Plant foliage was also sampled at the final harvest interval for both treatments. Samples were homogenised in dry ice and stored until analysis.

Plant material was extracted by maceration with methanol and water, with further extraction in 0.1M HCl and 0.1M NaOH (maceration and reflux) performed as necessary. The resulting extracts were separated by centrifugation and the radioactivity determined by LSC. The residue remaining was air-dried and the unextracted radioactivity was determined by LSC following combustion. Table 13 shows the radioactivities recovered from the foliage from soil and foliar treatments.

Table 13. Radioactivity from foliage extracts.

Treatment, kg ai/ha	PHI	TRR mg/kg eq.	mg/kg eq. (% TRR)				
			Water	methanol	0.1M HCl	0.1M NaOH	Non-extracted
Soil,	7	11.8	6.6 (56.5)	0.792 (8.2)	0.31 (2.6)	1.02 (8.6)	2.8 (23.9)
0.007 (1X)	35	4.9	-	2.1 (43.1)			2.8 (56.9)
Soil,	7	69.4	38.5 (55.5)	5.4 (7.9)	1.76 (2.5)	6.04 (8.7)	17.6 (25.4)
0.036 (5X)	35	19.8	-	8.2 (41.4)	-	-	11.6 (59.6)
Foliar, 2.2	28	5.21	-	367 (70.6)	-	-	1.53 (29.4)

The water and methanol extracts from the homogenised immature foliage (7 days PHI) were partitioned with chloroform and the resulting aqueous fractions contained 43% of TRR for the 1× treated samples and 37% of TRR for the 5× treated samples. HPLC of the extracts showed about 5% of TRR (0.61 and 3.1 mg/kg eq) as propamocarb, and four unidentified regions of radioactivity ranging from 2 to 22% TRR. The largest single region was polar in nature.

Residues in tomato fruit extracts harvested at each interval from the soil and foliar treated plants are shown in Table 14.

Table 14. Residues in mature tomato fruit.

Treatment	Interval (days)	TRR mg/kg eq	Methanol		Unextracted	
			mg/kg eq.	% TRR	mg/kg eq.	% TRR
Soil 1× / 5×	14	1.48 / 8.4	1.0 / 5.35	67.5 / 63.4	0.41 / 3.03	32.5 / 36.1
	21	1.34 / 7.32	0.89 / 4.84	66.3 / 66.1	0.45 / 2.48	33.7 / 33.9
	28	1.39 / 6.17	0.93 / 4.01	67.0 / 65.0	0.46 / 2.16	33.0 / 35.0

Treatment	Interval (days)	TRR mg/kg eq	Methanol		Unextracted	
			mg/kg eq.	% TRR	mg/kg eq.	% TRR
	35	1.23 / 7.17	0.80 / 4.49	65.1 / 62.7	0.43 / 2.68	34.9 / 37.35
Foliar	7	0.09	0.04	46.5	0.01	13.2
	14	0.12	0.10	82.9	0.02	17.1
	21	0.21	0.18	84.4	0.03	15.6
	28	0.27	0.23	85.7	0.04	14.3

HPLC analysis of the methanol and water extracts from the 1× soil treated fruit showed that propamocarb was not present in the sample. The radioactive residue was composed mainly of Unknown 1 and five other unidentified regions, each one with < 0.06 mg/kg eq. (Table 15). Analysis of the corresponding 5× soil treated fruit extracts gave similar results. HPLC analysis of the water and methanol extract from the foliar treated fruit contained mainly propamocarb (0.07mg/kg). The water wash contained mainly propamocarb (0.04 mg/kg) while the methanol extract contained only propamocarb (0.03mg/kg).

Table 15. Distritution of residues in tomatoes extracts.

Sample	Compound	Residue (mg/kg eq.)	% TRR
14 days, 1X soil TRR = 1.48 mg/kg eq.	Propamocarb	Not detected	Not detected
	Unknown 1	1.01	68.4
	Unknown 2 - 6	1.57	10.6
	Unallocated	0.004	0.2
	2M HCL reflux	0.170	11.5
	2M NaOH reflux	0.136	9.2
	Non-extracted	0.002	0.1
7 days, foliar treated fruit TRR = 0.086 mg/kg eq.	Propamocarb	0.065	75.2
	Unknown 1	0.014	16.6
	Unallocated	< 0.001	0.3
	2M HCL reflux	0.003	3.5
	2M NaOH reflux	0.002	2.6
	Non-extracted	0.002	1.8

Total radioactive residues of between 0.32 and 0.39 mg/kg were observed in samples of untreated control fruit, with methanol extraction releasing between 56 and 64% of the residue. When a second sub-sample was extracted 73% appeared in the methanol extract, 15% in the acid extract and the remaining 12% was unextracted. When the methanol extract was analysed by HPLC the radioactivity was present as a single region of polar material. The Unknown 1 observed in treated plants was also observed in control plants when extracts were analysed by HPLC. No explanation was given for the high residue foud in control samples.

The proposed metabolic pathway for propamocarb HCl in crops is presented in Figure 3

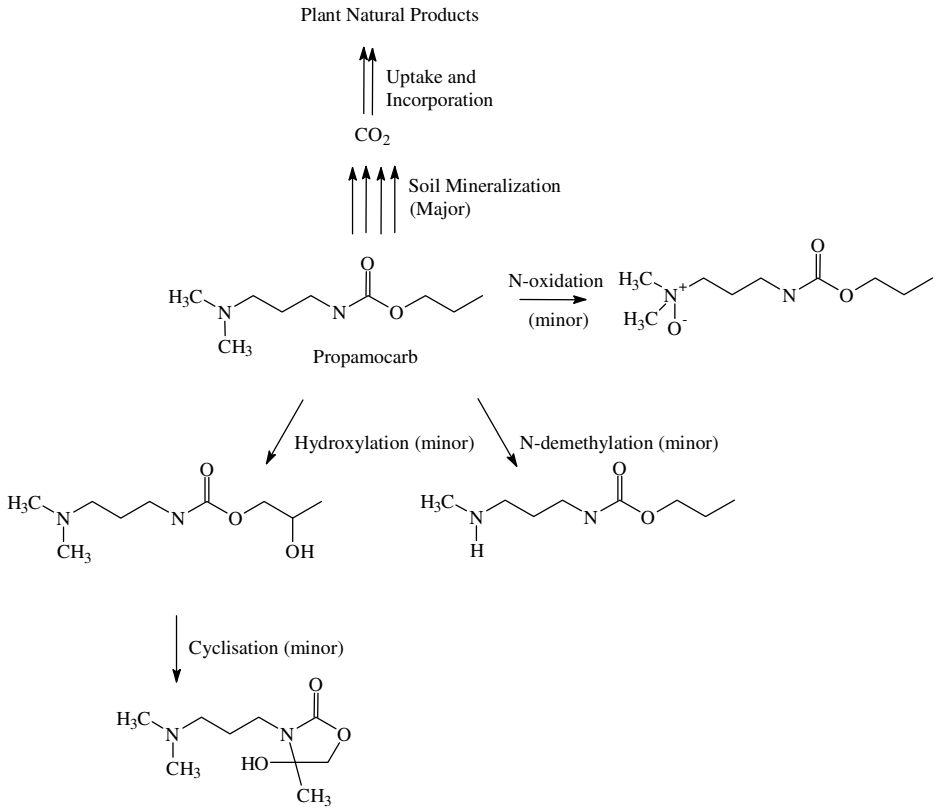


Figure 3. Proposed metabolic pathway for propamocarb hydrochloride in plants.

Rotational crops – confined

In one study conducted in USA under confined conditions, bare soil was treated at 5.96 – 6.16 kg/ha, representing 1.2 times the annual maximum application rate for propamocarb hydrochloride (Meyer, 2000, Report No. B002934). Leafy lettuce, radishes and wheat were planted at 30 days, 120 days and 365 days after treatment. Mature plants and immature wheat were harvested for analysis.

Residues were extracted with acidic methanol at either ambient temperature or by soxhlet and quantified by LSC. Residues remaining in the extracted fibber, as well as total residues in the RACs before extraction, were determined by combustion. Acid and base hydrolysis at elevated temperature was used to release non-extracted residues in the fibber. Methanol extracts and residues released from the fibber were analysed by reverse-phase HPLC and TLC using certified reference standards.

Total residues in the rotational crops, planted into the 30 day aged soil, ranged from 0.36 (radish roots) to 2.33 mg/kg (wheat straw) (Table 16). Total residues were much lower in crops planted in 120 days and 365 days aged soil.

Table 16. Mean Total Radioactive Residue, as mg/kg propamocarb eq.

	Aged treated soil		
Crop	**30 days**	**120 days**	**365 days**
Lettuce	0.79	0.03	0.02
Radish Tops	1.35	0.02	0.02
Radish Roots	0.36	0.03	0.01
Immature Wheat	1.10	0.04	0.04
Wheat Grain	0.66	0.09	0.06
Wheat Straw	2.33	0.08	0.08

Analysis of the extracts at 30 days and also from a wheat sample at 365 days showed a similar profile as in all crops at both time points. Propamocarb was found consistently in all samples and was frequently the major component. The identification of the metabolites is summarized in Table 17. The remaining metabolites identified comprised of 2-hydroxy propamocarb (lettuce and wheat) and the oxazolidine (Met VI) with traces of N-oxide (Met IV) and desmethyl propamocarb (30 days wheat only). The remainding radioactivity was composed of a complex mixture of highly polar components, which eluted at the solvent front on HPLC and at, or close to the origin on TLC.

Residues released after acid and base hydrolysis indicated a similar pattern of metabolites to those in the extracted residues, albeit with generally a higher proportion of the very polar components. Residues which remained unextracted after hydrolysis were less than 10% of the total radioactive residue.

Table 17. Summary of the compounds identified in the extracted residues from crops cultivated on soil treated with propamocarb 30 days befor planting.

	Methanol acidic extract		Propamocarb		Propyl propamocarb N-oxide (Met IV)		Propamocarb oxazolidin-2-one (Met VI)		2-Hydroxy propamocarb		Largest unknown detected	
Crop	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	% TRR	mg/kg eq	%	mg/kg eq	%	mg/kg eq	% TRR
Lettuce	0.551	74.6	0.302	40.9	0.045	6.1	0.049	6.6	0.031	4.1	0.029	4.0
Radish tops	1.103	81.7	0.911	67.4	0.042	3.1	0.06	4.4	nd	nd	0.035	2.6
Radish roots	0.24	72.6	0.104	31.5	0.015	4.6	0.018	5.5	0.01	2.9	0.01	3.0
Wheat forage	0.753	68.8	0.496	45.3	0.035	3.2	0.033	3.0	nd	Nd	0.039	3.6
Wheat grain	0.271	41.2	0.009	1.3	0.008	1.2	0.131	19.9	0.038	5.7	0.004	0.6
Wheat straw	1.06	45.3	0.359	15.4	0.131	5.6	0.231	9.9	0.064	2.6	0.021	0.9
Wheat straw[1]	0.014	16.8	nd	nd	--	--	nd	nd	0.002	2.3	0.069	3.0

1. Planted 365 day after soil treatment

A field study was conducted in the USA to determine residues in soil of propamocarb in rotational crops resulting from four applications to bare ground (Singer, 1999; C003451). The rotational crops selected were those anticipated to be grown after potatoes, in line with the typical agricultural cropping practices for each location. Four applications at a nominal rate of 1.68 kg ai/ha of propamocarb were made to bare soil at five-day intervals. Crops were planted 30, 60 or 365 days after the final soil treatment (Table 18).

Table 18. Summary of rotational crop trials.

Trial	Sate	Soil age	Crops planted		
R01-01	NY	30 and 60	Winter wheat		Soybean
R02-01	NC	30 and 60	Winter wheat		Soybean
R02-02	NJ	30 and 60	Winter wheat		Soybean
R03-01	FL	30 and 60	Winter wheat		Soybean
R05-01	WI	365	Spring wheat		Soybean
R05-02	ND	365	Spring wheat	Sugar beet	Soybean
R06-01	TX	30 and 60	Winter wheat	Sugar beet	
R08-01	CO	30 and 60	Winter wheat	Sugar beet	Dry beans
R10-01	CA	30 and 60	Spring wheat	Table beet	Dry beans
R10-02	CA	30 and 60	Winter wheat		Dry beans
R11-01	ID	365	Spring wheat	Sugar beet	Dry beans

Samples of wheat grain, forage, hay and straw, soybean seed, forage and hay, beets root and tops, and dry bean were harvested at typical harvest times. Winter wheat in some instances yielded a forage crop before the winter dormant period, while in other areas it did not yield forage until spring. All

samples from the 30 days plant back soil and all 60 days wheat forage samples were analysed for propamocarb.

Wheat was the only crop grown on 30 days aged soils which contained residues at or above LOQ. Therefore, only wheat samples were analysed for crops grown in the 60 days aged soil.

Wheat forage grown in soils treated 30 days before seeding contained detectable residues ranging from 0.055 to 0.229 mg/kg. The residues in 60 day wheat forage samples were generally below the LOQ of 0.05 mg/kg. In a few cases where detectable residues were found, they were around the LOQ level, i.e., 0.05–0.07 mg/kg. It was therefore decided that it would be unnecessary to analyse the crops grown in soil treated 365 days before seeding.

The 30 day plant back wheat hay samples, from four trials, contained residues in the range of 0.057 to 0.225 mg/kg, while no residue was detected in the other samples. The samples from the corresponding 60 day sites did not contain residues above LOQ.

All wheat straw samples derived from the 30 day and 60 day plant back sites contained residues below the LOQ of 0.05 mg/kg, with the exception of one replicate (0.05 mg/kg) from a 30 day site and one (0.055 mg/kg) from a 60 day plant back site.

ENVIRONMENTAL FATE

Aerobic soil degradation

The route of degradation for [^{14}C]-propamocarb hydrochloride, under aerobic conditions, has been extensively investigated under a range of temperatures, i.e., 10 to 25 °C. Five studies were conducted from 1978 to 1986 in loamy sand soil treated at 200 mg/kg and incubated at 15 or 25 ^{o}C in the dark (Bruhl and Celorio, 1978; Bruhl, 1979; Bruhl and Celorio, 1980a, b; Bruhl and Celorio, 1986). Propamocarb degraded very rapidly to several unidentified products, each having < 3% TRR, with a half life ranging from 10 to 28 days. After 60 days of incubation, about 80% of the radioactivity had been mineralized.

Fent and Hein (2001a, b, c) conducted three soil degradation studies with [^{14}C[-propamocarb HCl. In one study conducted at 20 ^{o}C (C012748), clay loam (Minnesota), loamy silt (Sarotti), loamy sand (Abington) and silty sand (Borstel) soils were incubated for 120 days with 0.48 mg/kg propamocarb, which corresponded to a field rate of 3.61 kg ai/ha. The amount of parent compound at the end of the study varied from < 2% TRR for loamy and silty sand soils to 27.1% TRR in clay loam soil (higher clay and organic matter content, Table 19). The formation of $^{14}CO_2$ increased steadily in all soils, ranging from 22.8% TRR in Minnesota soil to 66.2% TRR in Sarotti soil after 120 days. Up to eight non-identified metabolites were found in the soil extracts, which represented a total < 10% TRR in the course of the experiment. In another study conducted with loamy silt soil (Sarotti) at the same rate as previously, but at 10 ^{o}C incubation temperature (C012749), 79.1% TRR was assigned to propamocarb on Day 0, which decreased to 2.7% TRR on Day 120, with six unidentified metabolites, each one with < 6% TRR. Non-extracted residues ranged from 14.3% TRR at Day 0 to 21.1% TRR at Day 120, when 59.8% TRR was $^{14}CO_2$.

In the third study (C012750), silty sand soil (Borstel) was incubated at a rate 100 times lower than the previous studies (corresponding to 0.00361 kg ai/ha in the field) for 120 days, at 10 ^{o}C with the residue profile investigated at different soil depth layers. At the end of the study, the radioactivity assigned as propamocarb ranged from 25.7% TRR at 20 cm to 58.1% TRR at 90 cm. Up to nine non-identified metabolites were found in the extracts, none at > 6% TRR. DT_{50} and DT_{90} of the studies conducted by Fent and Hein were reported by Kley (2001a, b, and c) and are shown on Table 19.

Table 19. DT_{50} and DT_{90} of various soils treated with propamocarb HCl under aerobic conditions.

Soil	Clay,% (< 2 µm)	Organic carbon,%	Temperature	Rate, kg ai/ha	DT_{50}, days	DT_{90}, days
Clay loam (Minnesota)	32.2	3.15	20°C	3.61	136	452
Loamy silt (Sarotti)	17.7	1.3	20°C	3.61	11.7	38.9
Loamy sand (Abington	6.4	1.86	20°C	3.61	10.9	23.1
Silty sand (Borstel)	4.0	1.04	20°C	3.61	29.7	98.8
Loamy silt (Sarotti)	17.7	1.3	10°C	3.61	25.3	84.2
Silty sand (Borstel) 20 cm	4.0	1.04	10°C	0.00361	73.7	245
Silty sand (Borstel) 40 cm	4.0	1.04	10°C	0.00361	136	452
Silty sand (Borstel) 60 cm	4.0	1.04	10°C	0.00361	239	794
Silty sand (Borstel) 90 cm	4.0	1.04	10°C	0.00361	267	886

Schnöder (2002a/2003) conducted a study with four sandy loam soils and two clay loam soils incubated with 250 or 10 mg/kg [^{14}C]-propamocarb HCl at 20 or 10 °C for 120 or 365 days (soil A) (Table 20). For all soils, the majority of the radioactivity was assigned to propamocarb, decreasing from > 90% TRR at Day 0 to < 1%TRR in soils A, B, C and E, to 2.3% TRR in soil F and to 22.1% TRR in soil D. Soil D had the lowest organic carbon content and biomass amongst all soils used. The major metabolite was a polar unidentified component, with < 8% TRR after 90 days. Half life ranged from 14.1 to 87.7 days. The amount of $^{14}CO_2$ increased to 42.7% TRR after 365 days (soil A) and to 30.7–48.4% after 120 days (soils B-F). Non-extracted radioactivity increase to 31.1% TRR after 365 days in soil A and from 29.4 to 47.4% TRR after 120 days in soils B to F.

Table 20. DT_{50} and DT_{90} of sandy and clay soils treated with propamocarb HCl under aerobic conditions.

Soil	A	E	F	D	B	C
Type	Sandy loam	Sandy loam	Sandy loam	Sandy loam	Clay loam	Clay loam
PH, in 0.01MCaCL$_2$	6.7	6.7	6.7	4.9	6.2	7.3
Organic carbon (%)	2.5	2.5	2.5	1.3	4.5	2.7
Biomass, ugC/g	451.4	451.4	451.4	198.8	620.6	394.9
Rate, mg/kg	250	250	10	250	250	250
Temperature, °C	20	10	20	20	20	20
DT_{50} (days)	22.4	47.2	14.1	87.7	23.4	17.8
DT_{90} (days)	74.3	156.9	46.8	291.5	77.6	59.0

The metabolism of propamocarb HCl by soil micro-organisms was tested in sterilized and non-sterilized German standard soil 2.2 (Iwan, 1979: A85480; Iwan, 1980: A85481). After 14 days of incubation, recoverable propamocarb contents of sterilized samples remained constant (approximetaely 60% of applied material) the initial decrease being due to adsorption. In microbially active soil, extensive mineralization occurred following a lag-phase of 7 days. Degradation of propamocarb under these conditions was described by zero-order kinetics with a half-life of about 18 days. A mixed culture of bacteria and fungi capable of degrading the pesticide was identified. Intermediate metabolic products did not accumulate in any of the samples investigated.

Anaerobic soil degradation

Two studies were conducted to investigate the degradation of ^{14}C-propamocarb hydrochloride under anaerobic conditions. In one study conducted by Bruhl in 1979 (A85478), a loamy sand soil treated at a rate of 200 mg/kg and kept at 25 °C, propamocarb degraded very slowly, with a half life of 459 days. Three unidentified degradation products were detected at levels < 2.5% TRR. In the second study, a sandy loam soil flooded with water up to 3 cm above the surface was kept in the dark in a chamber at 20°C and purged continuously with nitrogen (Schneider, 2002b). After > 30 days, the system was treated with propamocarb HCl and kept under anaerobic conditions for 365 days (A) or

120 days (B). Total soil and water samples were sampled during the period of the study. Propamocarb rapidly dissipated from the water phase into the soils, leading to increases in radioactivity extracted from the soil. Propamocarb extracted from the soil decreased during the experiment with a consequent increase of the radioactivity in non-extracted residues. The major degradation product detected reached a maximum of 6.6% TRR in the system after 365 days. The half life for propamocarb in the system is shown in Table 21.

Table 21. DT_{50} and DT_{90} of flooded sandy loam treated soil under anaerobic conditions.

	Group A: 250 mg/kg		Group B: 10 mg/kg	
	DT_{50}	DT_{90}	DT_{50}	DT_{90}
Total system	308.2	1024	65.7	218.2
Water phase[a]	72.9/14.7	242.0/318.9	10.7/7.0	35.1/53.1

a. values correspond to 1-phase/2-phase models

Photolysis on soil surfaces

In one study conducted by Tschampel (1990/1994), [^{14}C]-propamocarb HCl was sprayed on to metal plates covered with a loamy sand soil at a concentration corresponding to typical agricultural use. The plates were irradiated with filtered light simulating natural sunlight. The estimated DT_{50} value under irradiated conditions was 35.4 days and over 97% TRR was recovered from the dark controls after 30 days. No degradation products exceed 10% TRR.

Field dissipation

Field dissipation studies (Willard, 2002, AA010716) were conducted in the USA, California (sandy loam) and Georgia (loamy sandy). A SL formulation of propamocarb hydrochloride was applied 4 times to soils, with 7 day intervals, at an application rate of 9.35 kg ai/ha. In each trial there was a treated plot covered in turf grass and a bare soil plot. In the turf grass plots, grass, thatch (0–7.5 cm deep) and turf soil (> 7.5 cm deep) samples were collected. In the bare soil plots, samples were collected up to 56 cm deep. Samples were collected from 1 day before treatment to 4 months after treatment. No residues (< 0.002 mg/kg) were detected at any time in samples > 15 cm deep collected from the bare soil from the Georgia loamy sand and > 30 cm deep from the bare soil from the California sandy loam. DT_{50} and DT_{90} from this study are shown on Table 22.

Table 22. Results from field dissipation studies conducted in USA.

Sample	DT_{50} (days)	DT_{90} (days)
Loamy sand	17.6	58.6
Sandy loam	22.1	73.3
Loamy sand thatch	17.4	57.7
Sandy loam thatch	23.7	78.6
Grass	13.2	43.9
Grass	18.1	60.1

RESIDUE ANALYTICAL METHODS

The residue methods used for plant, animal tissues and soil are based on either the analysis of propamocarb (free base) or propamocarb hydrochloride, depending on the internal standard used and the preparation of the standard solutions (calculated for the molecular weight of propamocarb or propamocarb hydrochloride). The methods involve a solvent extraction step (mostly diluted acetic

acid) followed by different matrix dependant clean up steps. The final determination is carried out by HPLC/MS/MS, GC/N-FID or GC/MSD.

Plant matrices

An enforcement method was validated for the determination of residues of propamocarb hydrochloride in various crops by LC-MS/MS (Diot and Rosati, 2005). For all sample materials, propamocarb hydrochloride was extracted with a mixture of water/acetic acid (99/1). For cauliflower, two extractions were necessary and avocado extracts were de-fatted with n-hexane. After centrifugation and dilution of the final extract, the residues are quantified by HPLC/MS with electrospray ionisation. The quantification was done by an external standardisation in solvent or using matrix matched standards. The LOQ was set at 0.01 mg/kg for propamocarb hydrochloride in all the sample materials. Mean recovery values and relative standard deviations at each fortification level are presented in Table 23.

Table 23. Recovery of propamocarb HCl from plant materials (n=5).

Crop	Level [mg/kg]	Mean [%]	RSD [%]
Lettuce head	0.01	79	13
	0.10	89	4
Chicory Witloof root	0.01	82	7
Chicory Witloof leaf	0.10	84	8
Pepper	0.01	73	4
	0.10	90	3
Potato	0.01	77	2
	0.10	98	3
Spinach	0.01	97	2
	0.10	97	1
Leek*	0.01	92	8
	0.10	77	7
Onion*	0.01	87	7
	0.10	93	13
Cabbage	0.01	75	18
	0.10	87	2
Cauliflower	0.01	73	10
	0.10	73	6
Brussels sprout	0.01	79	4
	0.10	73	4
Broccoli	0.01	88	7
	0.10	79	7
Cucumber	0.01	102	16
	0.10	89	4
Avocado*	0.01	73	4
	0.10	74	9
Wheat grain	0.01	86	8
	0.10	80	5

*recovery rates obtained using matrix matched standards

In another method, propamocarb (free base) was extracted with dilute acetic acid, the extract clean-up by C18 solid-phase-extraction (SPE) and propamocarb eluted with acetonitrile/water/acetic acid (Mende, 2001). Final determination was performed by HPLC-MS/MS, with quantification at m/z 102 and/or 144 (daughter ions). No peaks interfering with propamocarb were detectable (< 0.003 mg/kg) in control samples of all matrices. The limits of quantification for propamocarb were established at 0.01 mg/kg. Mean recovery values and relative standard deviations at each fortification level are presented in Table 24.

Table 24. Recovery of propamocarb (free base) from plants.

Matrix	Fortification level [mg/kg]	Mean recovery [%]	RSD [%]	n
Cabbage head	0.01	82	14	5
	0.1	91	19	5
	1.0	106	13	3
Cabbage plant	0.01	100	4	4
	0.1	97	10	5
	1.0	94	16	3
	100	91	--	1
Cauliflower head	0.01	81	16	9
	0.1	97	10	4
	1.0	106	--	1
Cauliflower plant	0.01	102	8	3
	0.1	91	20	4
	1.0	109	--	1
	100	82	--	1
Cucumber fruit	0.01	98	4	3
	0.1	94	7	7
	1.0	93	1	7
	2.0	108	3	2
	100	82	--	1
Head lettuce	0.01	88	12	3
	0.1	91	5	5
	1.0	87	12	5
	3.0	93	8	2
	10	80	--	1
	30	87	5	2
	50	97	--	1
	100	86	--	1
Melon pulp	0.01	88	8	3
	0.1	92	9	3
	2.0	102	2	3
	10	91	--	1
Melon peel	0.01	105	10	3
	0.1	97	3	4
	10	101	--	1
Melon fruit	0.01	94	4	4
	0.1	94	15	6
	1.0	95	7	4
	10	94	6	3
Sweet pepper fruit	0.01	83	19	13
	0.1	91	16	7
	1.0	95	4	5
Tomato fruit	0.01	82	12	6
	0.1	89	7	6
	1.0	89	2	3

The previous analytical method was validated by independent laboratories for the analysis of propamocarb (free base) in potato tubers and potato products (Class, 2002a and b), lettuce and tomato (Wrede, 2001). The results from both studies are shown in Table 25.

Table 25. Recovery of propamocarb (free base) from plant materials.

Matrix	Fortification Level [mg/kg]	Mean Recovery [%]	RSD [%]	n
Potato	0.01	97	5	3
	0.10	109	0	2
Puree	0.01	78	11	3
	0.10	103	4	3

Matrix	Fortification Level [mg/kg]	Mean Recovery [%]	RSD [%]	n
Fries	0.01	82	32	4
	0.10	91	5	3
Crisp	0.01	99	9	4
	0.10	140	29	3
Flakes	0.01	79	17	3
	0.10	92	12	3
Lettuce	0.01	67	14	5
	0.1	74	11	10
	30	101	4	5
Tomato	0.01	86	3	5
	0.1	77	10	10
Lettuce*	0.01	84	14	5
	0.1	95	10	5
	30	110	4	5
Tomato*	0.01	115	3	5
	0.1	102	10	10

* Matrix matched standards

A GC method was validated for propamocarb hydrochloride in different vegetables matrices after acidified methanol extraction followed by different clean-up steps of the extract (Wrede, 1988). The extract was basified with sodium hydroxide solution, extracted with chloroform and re-extracted with acidic water solution and further with di-isopropryl ether. The free base formed by alkaline hydrolysis was quantitatively determined by GC/N-FID. The method was validated at fortification levels of 0.1–10 mg/kg (Table 26). The same method was validated in another study (Scheuermann, 1983), with the results shown also in Table 26.

Table 26. Recovery of Propamocarb hydrochloride from plant materials (GC/N-FID).

Matrix	Fortification level [mg/kg]	Mean recovery [%]	RSD [%]	n
Cabbage	0.1	71	5	3
Lettuce	0.1	91	1	3
	0.2	105	4	3
	0.5	105	2	3
	1	98	8	3
	5	103	8	6
	10	108	10	3
Potato	0.1	82	3	2
	1.0	117	4	3
Pepper	0.1	82	2	3
	10	107	5	3
Radish	0.1	97	10	2
	1.0	110	3	2
Lettuce	0.1	120	17	7
	0.5	83	--	2
	1	91	5	4
	5	74	--	2
Cucumber	0.2	73	8	3
	1	71	6	6
	5	91	3	5
Spinach	0.5	106	--	2
	2	112	--	2
	20	65	--	2
Tomato	0.2	66	6	5
	1	63	--	2
Radish, Small radish	0.2	83	10	4
	0.5	124	21	4
	10	79	14	4

Matrix	Fortification level [mg/kg]	Mean recovery [%]	RSD [%]	n
Brussels sprouts	0.05	106	18	2
	0.2	63	10	5
	0.5	70	--	2
Cornsalad	0.2	100	--	2
	0.5	93	--	2
	1	99	--	2
	10	77	--	2
	50	91	--	2
	200	80	--	2
Celery	0.5	81	12	4
	1	63	--	2
Red beet roots	0.2	73	--	1
	0.5	76	--	1

This GC method was optimized for the analysis of propamocarb hydrochloride in potato samples (Wrede-Rücker, 1991) and various other crops (Chambers *et al.,* 1997), with the free base quantitatively determined by GC/MSD. For validation of the method, recovery experiments were performed at fortification levels from 0.05 mg/kg (LOQ) to 2.0 mg/kg (Table 27). Calibration curves of 2nd order were applicable over the tested range of 0.1 to 2.5µg/mL. No apparent residue (< 0.3 × LOQ) were detected in the control samples (Chambers *et al.*, 1997).

Table 27. Recovery of Propamocarb hydrochloride from plant samples.

Matrix	Fortification level [mg/kg]	Mean recovery [%]	RSD [%]	n
Potato	0.10	83	19	16
Leek	0.05	91	0	2
	0.10	98	7	3
	0.50	89	18	3
	1.0	83	5	3
Onion	0.05	86	5	3
	0.10	87	10	3
	0.50	87	9	4
Brassicae	0.05	89	6	8
	0.50	91	2	8
Tomato	0.05	78	3	2
	0.10	78	3	4
	0.50	83	3	2
Potato	0.05	83	2	2
	0.50	109	--	1
Melon	0.05	99	6	2
	0.10	91	7	4
	0.20	85	15	5
	0.50	86	4	4
	2.0	72	--	1

Animal matrices

Residues of propamocarb were extracted from animal products with 1.0% HCL in methanol and analyzed by HPLC-MS/MS (Leonard and Oden, 2001). Recovery experiments were performed at fortification levels of 0.01 mg/kg (LOQ) and 0.10 mg/kg. A linear calibration function was applicable over the tested range of 0.02 to 0.10 ng/mL. No peaks interfering with propamocarb were detectable (< 0.003 mg/kg) in the samples. This method was independently validated by another laboratory in milk, meat and eggs (Perez and Perez, 2001). Mean recovery values and relative standard deviations at each fortification level found in both studies are presented in Table 28.

Table 28. Recovery of propamocarb (free base) from animal tissues, milk and egg (n=5).

Matrix	Fortification level [mg/kg]	Mean recovery [%]	RSD [%]
Beef Muscle	0.01	85.5	11.4
	0.10	92.0	3.9
Beef Liver	0.01	86.8	7.4
	0.10	88.3	5.6
Beef Kidney	0.01	87.4	3.8
	0.10	90.0	3.7
Beef Milk	0.01	82.6	3.3
	0.10	91.4	3.0
Chicken Eggs	0.01	100	6.4
	0.10	101	4.1
Milk	0.01	101	8.4
	0.10	88.1	6.2
Meat	0.01	94.3	4.6
	0.10	107	3.5
Egg	0.01	83.7	9.5
	0.10	97.8	15.1

Soil

Residues of propamocarb hydrochloride are extracted from soil using HCL or acidified methanol, followed by different clean-up steps with chloroform and di-isopropyl ether and the free base formed by alkaline hydrolysis is quantitatively determined by GC/N-FID or GC/MSD (Scheuermann, 1983; Moede, 1991; Wrede, 2001). The extraction/clean-up procedure is similar to the one described previoulsly for plants (Wrede, 1988).

For method validation, recovery experiments were performed at fortification levels of 0.026 mg/kg (LOQ) to 50 mg/kg. The results are shown on Table 29.

Table 29. Recovery of Propamocarb hydrochloride from soil.

Reference	Fortification level [mg/kg]	Mean recovery [%]	RSD [%]	n
Scheuermann, 1983	1.0	94	--	2
	50	89	--	2
Moede, 1991	0.026	88	7	3
	0.05	89	8	3
	0.1	76	1	3
	1.0	88	5	3
Wrede, 2001	0.026	88	7	3
	0.05	99	8	11
	0.1	83	14	9
	1.0	79	16	6
Wrede, 2001	0.1	81	7	5
	0.1	50	10	5
	1.0	57	20	16

In another method (Mende, 2002), residues of propamocarb (free base) were extracted from soil using hydrochloric acid, followed by a clean-up of the extract on a C18 column. The final extract is basified with ammonia solution and the free base of propamocarb is quantitatively determined by LC-MS/MS (ratio of m/z = 102 to m/z =144). A linear calibration function was applicable over the tested range of 3 to 800 ng/mL. No peaks interfering with propamocarb were detected (< 0.003 mg/kg) in control samples. The LOQ for propamocarb were established at 0.02 mg/kg in soil (mean recovery of 89%, RSD of 8%; n=5). At 0.20 mg/kg, mean recovery and RSD were 102 and 5%, respectively (n=5). Mean recovery at 0.002 mg/kg was 54%.

Stability of residues in stored analytical samples

The stability of propamocarb HCl under freeze conditions (-18 to -20 °C) was investigated in tomato and lettuce samples stored for up to 26 months. A range of 59 to 106% of the added residues remained after the storage period (Table 30).

Table 30. Stability of propamocarb HCl residues under frozen conditions.

Crop	Fortification level, mg/kg	Storage period, months	% remained	Reference
Tomato	0.5	14.5	82, 83, 98	Moede, 1990; A85300
	5.0	14.5	59, 75, 67	
	0.5*	4	90, 98, 106	Sutton and Charter, 1999 C003740
		8	80, 78, 106	
		17	78, 88, 82	
		26	90, 88, 86	
Lettuce	0.5	14	109, 115	Wrede-Rücker, 1990; A85303
	5.0	14	100, 87.8	

* as free base

USE PATTERN

Formulations containing Propamocarb hydrochloride, alone or co-formulated with other active substances, are registered for use on a wide variety of crops in over 100 countries. Registrations cover foliar treatment of vegetable crops and potatoes, soil drench, application via drip irrigation to vegetables and ornamentals and as seed treatments. Registered uses of propamocarb hydrochloride in crops and countries which were relevant to this evaluation are shown on Table 31. All the labels were provided to the Meeting.

Table 31. Registered uses of propamocarb hydrochloride.

Crop	Country	F/G	Formulation Content ai	Application				PHI days
				Method	ai kg/ha	water L/ha	Max. No.	
Cabbage (brassica)	Belgium	G	SL 722 g/L	Seedbed Drench	36.1	10000	2	n.a.
Cabbage (brassica)	Spain	G	SL 530 g/L	Seedbed drench	15.9	10000-20000	2	14
Cabbage, head	Germany	F	SL 722 g/L	Foliar spray	1.08	400-600	2	21
Cabbage, head	Greece	G	SL 722 g/L	Seedbed drench	36.1	20000-40000	2	n.a.
Cabbage, head	Greece	F/G	SL 722 g/L	Soil drench	21.6	20000-40000	3	21
Cabbage, head	Italy	F/G	SL 722 g/L	Seedbed Incorporation	57.8-86.6	40000-80000	2	20
Cabbage, head	Italy	F/G	SL 722 g/L	Foliar spray	1.08-2.17	1500-2000	3	20
Cabbage, head	Italy	G	SL 530 g/L	Seedbed drench	16	20000-40000	2	20
Cabbage, head	Netherlands	F/G	SL 722 g/L	Seedbed drench	3.61	1000	2	14
Cabbage, head	Netherlands	F/G	SL 722 g/L	Foliar spray	2.2-3.6	500	2	14
Cabbage, head	UK	G	SL 722 g/L	Drench preplanting	72.2	20000-40000	1	n.a.
Cabbage, head	UK	G	SL 722 g/L	Drench postplanting	72.2	20000-40000	1	n.a.
Cauliflower	Belgium	F	SL 722 g/L	Foliar spray	2.17	500-1000	2	n.a.
Cauliflower	Germany	F	SL 722 g/L	Foliar spray	1.08	400-600	2	21
Cauliflower	Greece	G	SL 722 g/L	Seedbed drench	36.1	20000-40000	2	n.a.

Crop	Country	F/G	Formulation Content ai	Application				PHI days
				Method	ai kg/ha	water L/ha	Max. No.	
Cauliflower	Greece	F/G	SL 722 g/L	Soil Drench	21.6	20000-40000	3	21
Cauliflower	Italy	G	SL 530 g/L	Seedbed drench	31.8	20000-40000	1	20
Cauliflower	Italy	F/G	SL 722 g/L	Foliar spray	1.08-2.17	1500-2000	3	20
Cauliflower	Italy	G	SL 530 g/L	Seedbed drench	15.9	20000-40000	2	20
Cauliflower	Luxembourg	F	SL 722 g/L	Foliar spray	2.17	500	2	n.a.
Cauliflower	Netherlands	G	SL 722 g/L	Seedbed drench	3.61	1000	2	14
Cauliflower	Netherlands	F/G	SL 722 g/L	Foliar spray	2.2-3.6	500	2	14
Cauliflower	UK	F/G	SL 722 g/L	Drench preplanting	72.2	20000-40000	1	n.a.
Cauliflower	UK	F/G	SL 722 g/L	Drench postplanting	72.2	20000-40000	1	n.a.
Cauliflower	UK	F/G	SL 722 g/L	Seedbed Foliar spray	3.61	1000	2	14
Cauliflower	UK	F	SL 722 g/L	Foliar spray	1.81	1000	2	21
Chicory	Belgium	*	SL 722 g/L	Hydroponic forcing irrigation	9 g/hL	-	-	n.a.
Chicory Witloof	Cyprus	F	SL 722 g/L	Foliar spray	0.722 kg ai/hL		3	20
Chicory Witloof	France	*	SL 722 g/L	Watering roots after planting	72.2	30000-50000	1	21
Chicory Witloof	France	*	SL 722 g/L	Irrigation via nutrient solution	9 g/hL	-	1	21
Chicory Witloof	Greece	*	SL 722 g/L	Seedbed drenching	0.18 kg ai/hl	20000-40000	2	-
Chicory Witloof	Greece	F/G	SL 722 g/	Soil drench post planting	0.11 kg ai/hl	200-300 ml/plant	3	21
Chicory	Luxembourg	*	SL 722 g/L	Hydroponic forcing irrigation	9 g/hL	-	-	n.a.
Chicory	Luxembourg	F	SL 722 g/L	Foliar spray	1.1	500-1000	3	21
Chicory Witloof	Malta	*	SL 722 g/L	Spray roots after planting	57.8 86.6	40000 60000	1	14
Cucumber	Belgium	G	SL 722 g/L	Seedbed drench	36.1	50000	1	n.a.
Cucumber	Belgium	F/G	SL 722 g/L	Drip appl.	1.1 0.07-0.11 g/plant	-	4	n.a.
Cucumber	Bulgaria	F	SL 722 g/L	Foliar spray	1.81	-	5	14
Cucumber	France	F/G	SL 722 g/L	Foliar spray	2.17	200-1000	1	3
Cucumber	France	F/G	SC 375 g/L	Foliar spray	1.13	200-800	6	3
Cucumber	Germany	G	SL 722 g/L	Seedbed drench	65.0	60000	2	n.a.
Cucumber	Germany	F	SL 722 g/L	Foliar spray	2.2	600	4	4
Cucumber	Greece	*	SL 722 g/L	Seedbed drench	0.18 kg ai/hl	20000-40000	2	n.a.
Cucumber	Greece		SL 722 g/L	Soil drench	0.11 kg ai/hl	200-300 ml/plant	3	21
Cucumber	Greece	F/G	SL 722 g/L	Foliar spray	0.72-5.42	500-2500	3	3
Cucumber	Greece	F/G	SC 248 g/L	Foliar spray	1.44-3.61	500-1000	3	3
Cucumber	Italy	F	SC 375 g/L	Foliar spray	0.94-1.13	1000	3	20
Cucumber	Italy	G	SL 722 g/L	Seed treatment	7.2-28.9 mg/kg seed	-	1	n.a.
Cucumber	Italy	F/G	SL 722 g/L	Soil incorporation before drilling	300 ml/m³	20 l/m³	1	20
Cucumber	Italy	F/G	SL 722 g/L	Seedbed incorporation after drilling	57.8-86.6	40000 - 80000	2	20

Crop	Country	F/G	Formulation Content ai	Application				PHI days
				Method	ai kg/ha	water L/ha	Max. No.	
Cucumber	Italy	F/G	SL 722 g/L	Soil treatment pre- and transplanting via spraying	57.8-86.6	30000-50000	1	20
Cucumber	Italy	F/G	SL 722 g/L	Soil treatment post planting via spraying	0.14 kg ai/hl	0.1-0.2 l/plant	4	20
Cucumber	Italy	F/G	SL 722 g/L	Foliar spray	1.1-2.2	1500-2000	3	20
Cucumber	Italy	G	SL 530 g/L	Seedbed drench	31.8 or 15.9	20000-40000	1 2	20
Cucumber	Italy	F/G	SL 530 g/L	Soil treatment with dripping	1.1-1.6	20000	2	20
Cucumber	Japan	F/G	640 g/kg	Soil drench	4.8	30000	3	21
Cucumber	Luxembourg	G	SL 722 g/L	Seedbed drench	36.1	50000	1	n.a.
Cucumber	Luxembourg	F/G	SL 722 g/L	Plant drench	1.01	1400	2	n.a.
Cucumber	Netherlands	G	SL 722 g/L	Seedbed drenching	36.10	50000	3	n.a.
Cucumber	Netherlands	G	SL 722 g/L	Drench postplanting	1.01	100-150 ml/plant	2	n.a
Cucumber	Netherlands	G	SL 722 g/L	Drip application	0.72-1.44	20000	3	3
Cucumber	Poland	G	SL 530 g/L	Drench	10.6-23.9	20000-30000	2	3
Cucumber	Spain	G	SL 722 g/L	Seedbed treatment	14.4 – 21.7 kg ai./hl	80000-100000	1	-
Cucumber	Spain	F/G	SL 722 g/L	Soil drench (preventive)	0.18 – 0.36 kg ai/hl	20000-30000	1	-
Cucumber	Spain	F/G	SL 722 g/L	Drench to plant root	0.11 kg ai/hl	100ml/plant	1	-
Cucumber	Spain	F/G	SL 722 g/L	Dripping Treatment	1.44-2.17	1500-2000	1	3
Cucumber	Spain	F/G	SL 722 g/L	Foliar spray	0.144 – 0.22 kg ai/hl	300-1500	2	3
Cucumber	Spain	G	SL 530 g/L	Seedbed treatment	15.9	10000-20000	2	3
Cucumber	Spain	F/G	SL 530 g/L	Drip irrigation (preventive)	0.53	3000-15000	2	3
Cucumber	Spain	F/G	SL 530 g/L	Drip irrigation (curative)	1.1-1.6	20000	2	3
Cucumber	Sweden	G	SL 722 g/L	Seedbed drench	1.5-3.03	1400-2800	1	21
Cucumber	UK	F/G	SL 722 g/L	Drench at planting	72.2	20000-40000	4	3
Cucumber	UK	F/G	SL 722 g/L	Compost incorporation	2166-2888	200000-1000000	4	3
Cucumber	UK	F/G	SL 722 g/L	Trickle Irrigation	0.072 kg ai/hl	0.1-0.2 l /plant	4	3
Cucumber	UK	F/G	SL 722 g/L	Rockwood trickle Irrigation	0.009 kg ai/hl	0.1-0.2 l /plant	4	3
Cucumber	UK	F/G	SL 722 g/L	Drench postplanting	72.2	20000-40000	2	3
Cucumber	UK	F/G	SL 722 g/L	Foliar spray	1.81	1000	3	3
Cucumber (cucurbits)	USA	F	SL 722 g/L	Foliar or drip irrigation	1.0 kg ai/ha	140-935	5	2
Ginger	Japan	F	640 g/kg	Soil drench	3.21-4.8	30000	5	30
Lettuce	Belgium	F	SL 722 g/L	Foliar spray	1.08	500-1000	3	21
Lettuce	Germany	F	SL 722 g/L	Foliar spray	1.08	1000	3	21
Lettuce	Greece	G	SL 722 g/L	Seedbed Drench	1.8 g/L	2-4l/m^2	2	n.a.
Lettuce	Greece	F/G	SL 722 g/L	Soil drench postplanting	1.08 g/L	0.2-0.3l/plant	3	21

Crop	Country	F/G	Formulation Content ai	Application				PHI days
				Method	ai kg/ha	water L/ha	Max. No.	
Lettuce	Italy	G	SL530 g/L	Seedbed	16	20000-40000	2	20
Lettuce	Italy	F/G	SL 530 g/L	Foliar spray	1.1-1.6	400-1000	2	14
Lettuce	Japan	F/G	640 g/kg	Foliar spray	0.384	3000	3	14
Lettuce	Netherlands	F/G	SL 722 g/L	Seedbed drench	36.1	500-1000	2	n.a.
Lettuce	Netherlands	F/G	SL 722 g/L	Foliar spray	1.1	1000	2	21
Lettuce	Spain	-	SL 530 g/L	Seedbed treatment	16	10000-20000	2	14
Lettuce	Spain	F	SL 530 g/L	Foliar spray	1.06-1.33	300-800	2	14
Lettuce	UK	F/G	SL 722 g/L	Foliar spray	1.1-1.4	600-1500	3	21
Lettuce	USA	F	SL 722g/L	Foliar spray or Drip irrigation	1.68	GA:140-935 Aerial: 95	4	2
Melon	Belgium	G	SL 722 g/L	Seedbed drench	36.1	50000	1	n.a.
Melon	Belgium	G	SL 722 g/L	Drip appl. (preventive)	1.01	1400	2	n.a.
Melon	Belgium	G	SL 722 g/L	Drip appl. (curative)	1.0-2.0	1400-2800	2	n.a.
Melon	France	F/G	SL 722 g/L	Foliar spray	2.17	200-1000	1	3
Melon	France	F/G	SC 375 g/L	Foliar spray	1.13	200-800	6	3
Melon	Germany	F	SL 722 g/L	Foliar spray	2.17	400-600	4	4
Melon	Greece	G	SL 722 g/L	Seedbed drench	36.1-72.2	20000-40000	2	n.a.
Melon	Greece	F/G	SL 722 g/L	Soil drench	0.18 kg ai/hl	0.2-0.3 ml/plant	3	21
Melon	Italy	F/G	SC 375 g/L	Foliar spray	0.94-1.13	1000	3	20
Melon	Italy	F/G	SL 722 g/L	Seedbed incorporation	57.8-86.6	40000-80000	2	20
Melon	Italy	F/G	SL 722 g/L	Soil spraying Pre and transplanting	57.8-86.6	300000-580000	1	20
Melon	Italy	F/G	SL 722 g/L	Soil post planting	0.14 kg ai/hl	0.1-0.2l/plant	4	20
Melon	Italy	F/G	SL 722 g/L	Foliar	1.1-2.2	1500-2000	2	20
Melon	Italy	G	SL 530 g/L	Seedbed disinfection	31.8 or 15.9	20000-40000	1 2	20
Melon	Italy	G	SL 530 g/L	Drip irrigation	1.06-1.59	20000	2	20
Melon	Netherlands	G	SL 722 g/L	Drench preplanting	36.1	50000	3	3
Melon	Netherlands	G	SL 722 g/L	Drench postplanting	1.01	1400	2	3
Melon	Netherlands	G	SL 722 g/L	Drip Irrigation	0.72-1.44	300-600	3	1
Melon	Spain	G	SL 530 g/L	Seedbed treatment	15.9	20000	2	14
Melon	Spain	F/G	SL 530 g/L	Drip irrigation (preventive)	0.53	3000-15000	2	14
Melon	Spain	F/G	SL 530 g/L	Drip irrigation (curative)	1.06-1.59	20000	2	14
Onion	Poland	F	SC 375 g/L	Foliar spray	0.94	700	5	7
Onion	Sweden	F	SC 248 g/L	Foliar spray	0.5 / 1	150-300	8 / 4	30
Onion	UK	F	SL 722 g/L	Drench preplanting	72.2	20000-40000	1	133
Onion	UK	F	SL 722 g/L	Foliar spray	1.8-3.6	500	2	133
Onion	UK	F	SL 722 g/L	Seed treatment	21.7g/kg seed	-	1	n.a.
Onion	UK	F	SL 722 g/L	Bulb dipping preplanting	2.2 g/L	-	1	n.a.
Peppers	Greece	G	SL 722 g/L	Seedbed drench	1.8 kg ai/hl	20000-40000	2	21

Crop	Country	F/G	Formulation Content ai	Application				PHI days
				Method	ai kg/ha	water L/ha	Max. No.	
Peppers	Greece	F/G	SL 722 g/L	Soil drench	1.1 kg ai/hl	0.2-0.3 l/plant	3	21
Peppers	Italy	G	SL 530 g/L	Seedbed drench	31.8 or 15.9	20000-40000	1 2	20
Peppers	Italy	F/G	SL 530 g/L	Soil treatment with dripping	1.1-1.6	20000	2	20
Peppers	Italy	F/G	SL 722 g/L	Seedbed incorporation	57.8-86.6	40000-80000	2	20
Peppers	Italy	F/G	SL 722 g/L	Soil spraying Pre and transplanting	57.8-86.6	300000-580000	1	20
Peppers	Italy	F/G	SL 722 g/L	Soil treatment post planting	0.14 kg ai/hl	0.1-0.2l/plant	4	20
Peppers	Italy	F/G	SL 722 g/L	Foliar	1.1-2.2	1500-2000	2	
Peppers	USA	F	SL 722 g/L	Foliar Spray or drip irrigation	1.0	GA-average 400 Aerial 47	-	5
Potato	France	F	SC 375 g/L	Foliar spray	1.01	-	-	21
Potato	Germany	F	SC 248 g/L	Foliar spray	0.99	400-600	6	7
Potato	UK	F	SC 375 g/L	Foliar spray	0.56-0.94	200-400	6	7
Potato	USA	F	SL 66.5%	Foliar spray	0.59-1.01	400	8–5	14
Radish	Germany	F/G	SL 722 g/L	Seed treatment	7.22 g/kg seed	-	1	14
Radish	Germany	F/G	SL 722 g/L	Foliar spray	0.72	1000	2	14
Radish	Netherlands	F/G	SL 722 g/L	Foliar spray	1.08	500	2	14
Spinach	Italy	F/G	SL 722 g/L	Seed treatment	0.722-28.9 g/kg seed	-	1	n.a.
Spinach	Italy	G	SL 722 g/L	Seedbed Incorporation	57.8-86.6	40000-80000	2	20
Spinach	Italy	F/G	SL 722 g/L	Foliar spray	1.1-2.2	1500-2000	3	20
Summer squash (cucurbits)	USA	F	SL 66.5%	Foliar spray or drip irrigation	1.0	GA average=400 Aerial 47	5	2
Sweet pepper	Belgium	G	SL 722 g/L	Seedbed drench	36.1	-	1	n.a.
Sweet pepper	Belgium	F/G	SL 722 g/L	Drip appl.	1.1 0.07-0.11 g/plant	-	4	n.a.
Sweet pepper	Netherlands	G	SL 722 g/L	Seedbed drenching	36.1	50000	3	n.a.
Sweet pepper	Netherlands	G	SL 722 g/L	Drench postplanting	1.01	100-150 ml/plant	2	n.a
Sweet pepper	Netherlands	G	SL 722 g/L	Drip application	0.72-1.44	20000	3	3
Sweet pepper	Spain	F/G	SL 722 g/L	Seedbed drench	1200-1800	80000-10000	1	14
Sweet pepper	Spain	F/G	SL 722 g/L	Soil drench (preventive)	36.1-72.2	20000-30000	1	14
Sweet pepper	Spain	F/G	SL 722 g/L	Soil drench to plant root	3.8	3500	1	14
Sweet pepper	Spain	F/G	SL 722 g/L	Dripping treatment	1.44-2.17	1500-2000	1	14
Sweet pepper	Spain	G	SL 530 g/L	Seedbed treatment	15.9	20000	2	3
Sweet pepper	Spain	F/G	SL 530 g/L	Soil drip irrigation (preventive)	0.53	3000-15000	2	3
Sweet pepper	Spain	F/G	SL 530 g/L	Soil drip irrigation (curative)	1.01-1.6	20000	2	3
Sweet pepper	UK	F/G	SL 722 g/L	Foliar spray	2.2-3.6	600-1000	2	14
Tomato	Belgium	G	SL 722 g/L	Seedbed drench	36.1	50000	1	n.a.
Tomato	Belgium	F/G	SL 722 g/L	Drip appl.	1.1 0.07-0.11 g/plant	-	4	n.a.

Crop	Country	F/G	Formulation Content ai	Application				PHI days
				Method	ai kg/ha	water L/ha	Max. No.	
Tomato	Germany	G	SL 722 g/L	Watering before and after planting	65.0	60000	2	n.a.
Tomato	Greece	G	SL 722 g/L	Seedbed drench	1.8 kg ai/hl	20000-40000	2	21
Tomato	Greece	F/G	SL 722 g/L	Soil drench	1.1 kg ai/hl	0.2-0.3 l/plant	3	21
Tomato	Italy	G	SC 375 g/L	Foliar spray	0.94-1.13	1000	5	20
Tomato	Italy	F	SC 375 g/L	Foliar spray	0.94-1.13	1000	3	20
Tomato	Italy	F/G	SL 722 g/L	Seed treatment	7.22-28.88 mg/kg seed	-	1	n.a.
Tomato	Italy	G	SL 530 g/L	Seedbed drench	31.8 or 15.9	20000-40000	1 2	20
Tomato	Italy	F/G	SL 530 g/L	Soil treatment with dripping	1.1-1.6	20000	2	20
Tomato	Italy	F/G	SL 722 g/L	Seedbed incorporation	57.8-86.6	40000-80000	2	20
Tomato	Italy	F/G	SL 722 g/L	Soil spraying Pre and transplanting	57.8-86.6	300000-580000	1	20
Tomato	Italy	F/G	SL 722 g/L	Soil post planting	0.14 kg ai/hl	0.1-0.2l/plant	4	20
Tomato	Italy	F/G	SL 722 g/L	Foliar	1.1-2.2	1500-2000	2	
Tomato	Luxembourg	G	SL 722 g/L	Seedbed drench	36.1	50000	1	n.a.
Tomato	Luxembourg	F/G	SL 722 g/L	Plant drench	1.01	1400	2	n.a.
Tomato	Netherlands	G	SL 722 g/L	Seedbed drenching	36.1	50000	3	n.a.
Tomato	Netherlands	G	SL 722 g/L	Drench postplanting	1.01	100-150 ml/plant	2	n.a
Tomato	Netherlands	G	SL 722 g/L	Drip application	0.72	20000	3	3
Tomato	Spain	F/G	SL722 g/L	Seedbed drench	1200-1800	80000-10000	1	14
Tomato	Spain	F/G	SL 722 g/L	Soil drench (preventive)	36.1-72.2	20000-30000	1	14
Tomato	Spain	F/G	SL 722 g/L	Soil drench to plant root	3.8	3500	1	14
Tomato	Spain	F/G	SL 722 g/L	Dripping treatment	1.44-2.17	1500-2000	1	14
Tomato	Spain	G	SL 530 g/L	Seedbed treatment	15.9	20000	2	3
Tomato	Spain	F/G	SL 530 g/L	Soil drip irrigation (preventive)	0.53	3000-15000	2	3
Tomato	Spain	F/G	SL 530 g/L	Soil drip irrigation (curative)	1.01-1.6	20000	2	3
Tomato	UK	F/G	SL 722 g/L	Drench at planting	72.2	20000-40000	4	14
Tomato	UK			Compost incorporation	2888	20000-1000000		
Tomato	UK	F/G	SL 722 g/L	Trickle Irrigation	2.53-5.05	3500-7000		
Tomato	UK	F/G	SL 722 g/L	Rockwood trickle Irrigation	2.53-3.16	14000-17500		
Tomato	UK	F/G	SL 722 g/L	Foliar spray	1.81-2.17	1000	3	7
Tomato	USA	F	SL 66.5%	Foliar spray	0.59 0.84 1.26	GA average=400 Aerial 47	8 7 5	5 5 5

* protected site = dark forcing room; ** application to roots after transplanting in hydroponic container via drench, spray or irrigation via nutrient solution; GA = ground application

RESIDUE FROM SUPERVISED TRIALS

A number of residue trials have been performed with propamocarb hydrochloride on several vegetable crops. Table 32 provides a summary of the data provided to the Meeting. All studies were conducted according to GLP and the reports included detailed information on trial conditions and analytical method validation with the exception of radish trials conducted in the 80´s. The trials were conducted either in glasshouses (GH) or in the field (F). The Japanese trial results were submitted to the Meeting by the Japanese Government in a summary table.

When residues were reported in the studies as propamocarb hydrolcholoride, the values were multiplied by 0.84 (MW propamocarb/MWpropamocarb HCl) and expressed as propamocarb. Residues within 30% GAP are underlined and were considered for recommendation of STMR, HR and MRL. Residues on the crop pulp were double underlined and considered only for recommendation of STMR and HR.

Table 32. Summary of supervised trials conducted with propamocarb hydrolcoride.

Crop	Number of trials	Countries	Table
Onion	7	France, Germany, Netherlands, Spain, UK	33
Cabbage	18	France, Germany, Italy, Spain	34
Cauliflower	23	France Germany, Greece, Italy, Spain, UK	35
Cucumber	41	Belgium, France, Germany, Greece, Italy, Netherlands, Spain, USA, Japan	36
Melons	55	France, Germany, Greece, Italy, Netherlands, Portugal, Spain, USA	37
Summer Squash	6	USA	38
Sweet peper	35	Belgium, Germany, Greece, Italy, Netherlands, Spain, USA	39
Tomato	45	Belgium, France, Germany, Greece, Italy, Netherlands, Spain, USA	40
Lettuce	26	France, Germany, Greece, Italy, Netherlands, Spain, USA, Japan	41
Spinach	7	Belgium, Germany, Italy, Spain	42
Potato	32	France, Germany, UK, USA	43
Radish	13	Germany and Netherlands	44
Chicory witloof	20	France Germany, Netherlands	45
Ginger	4	Japan	46

Onion

Seven field trials were conducted with propamocarb hydrochloride on onion in Europe from 1988 to 2003. The results are shown in Table 33.

Table 33. Residue field trials with propamocarb hydrochloride conducted with foliar treatment on onion.

Country, Year *Variety*	Application				PHI (Days)	Residues, as Propamocarb mg/kg	Residues, as propamocarb HCl, mg/kg	Report
	Form.	kg ai/ha	Water L/ha	No.				
France, 2003 *Barito*	450 SC 375 g ai/L	0.75	300	4	0 14	0.25 0.04	0.30* 0.05*	C047478
Germany 2003 *Stuttgarter Riesen*	450 SC 375 g ai/L	0.75	300	4	0 14	0.075 < 0.008	0.09* < 0.01*	C047478
Germany 2003 *Stuttgarter Riesen*	450 SC 375 g ai/L	0.75	300	4	0 14	0.08 < 0.008	0.10* < 0.01*	C047478
Netherlands 2003 *Hyskin*	450 SC 375 g ai/L	0.75	300	4	0 14	0.29 0.03	0.35* 0.04*	C047478

Country, Year *Variety*	Application				PHI (Days)	Residues, as Propamocarb mg/kg	Residues, as propamocarb HCl, mg/kg	Report
	Form.	kg ai/ha	Water L/ha	No.				
Spain, 1988 *Babosa*	722 SL 722 g ai/L	1.8		4	14	< 0.08	< 0.1*	A85277
Spain, 1988 *Babosa*	722 SL 722 g ai/L	2.9		4	14	0.17	0.2*	A85277
UK, 2003	450 SC 375 g ai/L	0.75	300	4	0 14	0.23 0.03	0.27* 0.04*	C047478

* value reported

Cabbage

Eighteen field trials were conducted with propamocarb hydrochloride on cabbage in Europe using a seedbed drench followed by foliar spray applications. The results are shown in Table 34.

Table 34. Residue field trials with propamocarb hydrochloride conducted on cabbage.

Country, Year of trial *Variety*	Application					PHI (Days)	Sample analysed	Residues as Propamocarb mg/kg	Residues Propamocarb HCl, mg/kg	Report
	Form.	Method	kg ai/ha application	Water L/ha	No.					
France 2001 *Tex 600 F1*	722 SL 722 g ai/L	Seedbed drench Spray	72.5-36.3 2.1-3.1	20000 486-714	2 2	0 42 54	Head Head Head	8.2 0.02 0.02	9.8* 0.03* 0.02*	C022939
France 2001 *Delus*	722 SL 722 g ai/L	Seedbed drench Spray	72.8-36.4 2.2	20156 500	2 2	0 42 54	Head Head Head	3.9 0.008 0.02	4.6* 0.01* 0.02*	C022939
France, 2001 *Milan*	722 SL 722 g ai/L	Seedbed drench Spray	72.2-36.1 2.2	20000 500	2 2	0 45 56	Whole Plant Head Head	23 0.20 0.43	28* 0.24* 0.51*	C022953
France 2000 *Destiny F1*	722 SL 722 g ai/L	Seedbed drench Spray	72.2-36.1 3.7	20000 616-829	2 2	0 14 34 47 55	Whole Plant Whole Plant Head Head Head	36 1.1 < 0.008 < 0.008 < 0.008	43* 1.3* < 0.01* < 0.01* < 0.01*	C015430
France 2000 *Delus F1*	722 SL 722 g ai/L	Seedbed drench Spray	72.2-36.1 3.8-3.5	20000 630-770	2 2	0 14 55 62 69	Whole Plant Whole Plant Head Head Head	56.0 7.7 < 0.008 < 0.008 < 0.008	66* 9.2* < 0.01* < 0.01* < 0.01*	C015430
Germany 2000 *Bartolo*	722 SL 722 g ai/L	Seedbed drench Spray	71-36.1 3.6	20000 500	2 2	0 12 47 68 96	Whole Plant Whole Plant Head Head Head	39 0.9 < 0.008 < 0.008 < 0.008	46* 1.1* < 0.01* < 0.01* < 0.01*	C015430
Germany 2000 *Lennox*	722 SL 722 g ai/L	Seedbed drench Spray	71.5-37.7 3.5	20000 500	2 2	0 6 42 63 93	Whole Plant Whole Plant Head Head Head	42 8.0 < 0.008 < 0.008 < 0.008	51* 9.5* < 0.01* < 0.01* < 0.01*	C015430
Germany 2000 *Perfecta F1*	722 SL 722 g ai/L	Seedbed drench Spray	72.2-36.1 3.65	20000 603-827	2 2	0 12 36 50 55	Whole Plant Whole Plant Head Head Head	37 2.8 < 0.008 < 0.008 < 0.008	44* 3.4* < 0.01* < 0.01* < 0.01*	C015430
Germany 2001 *Tex 600 F1*	722 SL 722 g ai/L	Seedbed drench Spray	72.5-36.3 2..2	20000 500	2 2	0 42 56	Head Head Head	9.2 0.02 0.01	11* 0.02* 0.01*	C022939
Germany 2001 *Bartolo*	722 SL 722 g ai/L	Seedbed drench Spray	73.6-36.1 2.2	20000 500	2 2	0 45 59	Head Head Head	11 < 0.008 < 0.008	13* < 0.01* < 0.01*	C022939

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha application	Water L/ha	No.	PHI (Days)	Sample analysed	Residues as Propamocarb mg/kg	Residues Propamocarb HCl, mg/kg	Report
Italy	722 SL	Seedbed	72.2-36.1	20000	2	0	Whole Plant	58	69*	C022953
2001	722 g	drench				44	Head	< 0.008	< 0.01*	
Marcanta	ai/L	Spray	2.1	490	2	58	Head	< 0.008	< 0.01*	
Italy, 2000	722 SL	Seedbed	69.5-36.9	20000	2	0	Whole Plant	40	48*	C015431
Mercato di	722 g	drench				23	Whole Plant	0.04	0.5*	
Copenhage	ai/L					30	Head	< 0.008	< 0.01*	
n		Spray	3.6	500	2	36	Head	< 0.008	< 0.01*	
						41	Head	< 0.008	< 0.01*	
Italy, 2000	722 SL	Seedbed	75.2-35.8	20000	2	0	Whole Plant	109	130*	C015431
Mercato di	722 g	drench				26	Whole Plant	1.0	1.1*	
Copenhage	ai/L					34	Head	0.02	0.02*	
n		Spray	3.6	600	2	41	Head	< 0.008	< 0.01*	
						46	Head	< 0.008	< 0.01*	
Italy, 2000	722 SL	Seedbed	72.3-36.9	20000	2	0	Whole Plant	43	52*	C015431
Mercato di	722 g	drench				15	Whole Plant	3.7	4.4*	
Copenhage	ai/L					22	Head	0.03	0.04*	
n		Spray	3.81-3.57	600	2	29	Head	0.02	0.02*	
						36	Head	0.008	0.01*	
Spain, 2001	722 SL	Seedbed	71.9-36.1	20000	2	0	Whole Plant	98	117*	C022953
Sentinel	722 g	drench				48	Head	< 0.008	< 0.01*	
	ai/L	Spray	2.2	500	2	64	Head	< 0.008	< 0.01*	
Spain, 2001	722 SL	Seedbed	72.1-36.7	20000	2	0	Whole Plant	30	36*	C022953
Sentinel	722 g	drench				49	Head	< 0.008	< 0.01*	
	ai/L	Spray	2.2	500	2	61	Head	< 0.008	< 0.01*	
Spain, 2000	722 SL	Seedbed	72.2-36.1	20000	2	0	Whole Plant	24	29*	C015431
Sentinel	722 g	drench				82	Whole Plant	0.04	0.05*	
	ai/L					113	Head	< 0.008	< 0.01*	
		Spray	3.7-3.5	550	2	123	Head	< 0.008	< 0.01*	
						138	Head	< 0.008	< 0.01*	
Spain, 2000	722 SL	Seedbed	72.2-36.1	20000	2	0	Whole Plant	96	115*	C015431
Sentinel	722 g	drench				71	Whole Plant	0.1	0.1*	
	ai/L					89	Head	< 0.008	< 0.01*	
		Spray	3.5-3.8	481-637	2	110	Head	< 0.008	< 0.01*	
						125	Head	< 0.008	< 0.01*	

*actual value reported

Cauliflower

Twenty three field trials were conducted on cauliflower with propamocarb hydrochloride in Europe using a seedbed drench followed by a foliar application. The results are shown in Table 35.

Table 35. Residue field trials with propamocarb hydrochloride conducted on cauliflower.

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha	Water L/ha	No.	PHI (Days)	Sample analysed	Residues Propamocarb mg/kg	Residues as Propamocarb HCl, mg/kg	Report
France	722 SL	Seedbed	72.2-36.0	20000	2	0	Head	1.4	1.7*	C033812
2002	722 g	drench				7	Head	0.05	0.06*	
Cortes	ai/L					14	Head	0.05	0.06*	
		Spray	3.6	500	2	20	Head	0.03	0.04*	
						28	Head	0.02	0.03*	
France	722 SL	Seedbed	72.2-36.0	20000	2	0	Head	0.24	0.29*	C033812
2002	722 g	drench				7	Head	0.03	0.04*	
Amerigo	ai/L					15	Head	0.03	0.03*	
		Spray	3.6	500	2	21	Head	0.09	0.11*	
						28	Head	0.11	0.13*	

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha	Water L/ha	No.	PHI (Days)	Sample analysed	Residues Propamocarb mg/kg	Residues as Propamocarb HCl, mg/kg	Report
Germany	722 SL	Seedbed	72.2-	20000	2	0	Shoots	42	50*	C033812
2002	722 g	drench	36.0			7	Shoots	13	16*	
Spacestar	ai/L					14	Shoots	7.9	9.4*	
		Spray	3.6	600	2	21	Shoots	0.69	0.82*	
						41	Head	0.02	0.03*	
Germany	722 SL	Seedbed	72.2-	20000	2	0	Shoots	47	56*	C033812
2002	722 g	drench	36.0			7	Shoots	10	12*	
Fremont	ai/L					14	Shoots	0.51	0.61*	
		Spray	3.6	600	2	21	Head	0.02	0.03*	
						28	Head	0.08	0.10*	
France	722 SL	Seedbed	72.8-	20156	2	0	Whole	71	84*	C023895
2001	722 g	drench	36.4			44	Plant	0.008	0.01*	
CLX 33903	ai/L	Spray		482-	2	58	Head	< 0.008	< 0.01*	
			2.1-2.3	528			Head			
France	722 SL	Seedbed	72.5-	20156	2	0	Whole	79	94*	C023895
2001	722 g	drench	36.23			44	Plant	0.03	0.03*	
Aviso F1	ai/L	Spray		482-	2	58	Head	< 0.008	< 0.01*	
			2.15	528			Head			
France	722 SL	Seedbed	72.3-	20000	2	0	Whole	60	72*	C024417
2001	722 g	drench	33.2			49	Plant	< 0.008	< 0.01*	
Fremont	ai/L	Spray		498	2	56	Head	< 0.008	< 0.01*	
			2.2				Head			
France,	722 SL	Seedbed	72.2-	20000	2	0	Whole	95	113*	C015428
2000	722 g	drench	36.0			34	Plant	0.03	0.04*	
Fremont F1	ai/L					55	Whole	< 0.008	< 0.01*	
RS		Spray	3.68-	612-	2	62	Plant	< 0.008	< 0.01*	
			3.48	772		64	Head	< 0.008	< 0.01*	
							Head			
							Head			
France	722 SL	Seedbed	72.2-	20000	2	0	Whole	100	119*	C015428
2000	722 g	drench	36.0			26	Plant	0.03	0.04*	
Fremont	ai/L					39	Whole	< 0.008	< 0.01*	
		Spray	3.8	625-	2	43	Plant	< 0.008	< 0.01*	
				852		47	Head	< 0.008	< 0.01*	
							Head			
							Head			
Germany	722 SL	Seedbed	69.0-	19200	2	0	Whole	70	83*	C015428
2000	722 g	drench	35.8	19834		12	Plant	2.7	3.2*	
Aviso	ai/L					47	Whole	< 0.008	< 0.01*	
		Spray	3.7	513-	2	54	Plant	< 0.008	< 0.01*	
				510		57	Head	0.008	0.01*	
							Head			
							Head			
Germany	722 SL	Seedbed	72.4-	20000	2	0	Whole	95	113*	C015428
2000	722 g	drench	35.9			6	Plant	13.4	16.0*	
BK Fargo	ai/L					42	Whole	< 0.008	< 0.01*	
		Spray	3.6	500	2	48	Plant	< 0.008	< 0.01*	
						51	Head	< 0.008	< 0.01*	
							Head			
							Head			
Germany	722 SL	Seedbed	72.2-	20000	2	0	Whole	85	101*	C015428
2000	722 g	drench	36.0			28	Plant	1.3	1.5*	
Fremont F1	ai/L					43	Whole	< 0.008	< 0.01*	
RS		Spray	3.7-3.6	620-	2	50	Plant	< 0.008	< 0.01*	
				806		55	Head	< 0.008	< 0.01*	
							Head			
							Head			
Germany	722 SL	Seedbed	72.2-	20156	2	0	Whole	49	58*	C023895
2001	722 g	drench	36.0			49	Plant	< 0.008	< 0.01*	
Aviso	ai/L			482-	2	58	Head	< 0.008	< 0.01*	
		Spray	2.4-2.3	528			Head			

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha	Water L/ha	No.	PHI (Days)	Sample analysed	Residues Propamocarb mg/kg	Residues as Propamocarb HCl, mg/kg	Report
Germany 2001 *Aviso F1*	722 SL 722 g ai/L	Seedbed drench Spray	72.5- 36.3 2.2	20156 482- 528	2 2	0 45 60	Whole Plant Head Head	52 < 0.008 < 0.008	62* < 0.01* < 0.01*	C023895
Greece 2001 *Siria*	722 SL 722 g ai/L	Seedbed drench Spray	72.2- 36.0 2.0	20000 487- 453	2 2	0 49 65	Whole Plant Head Head	52 0.09 0.12	62* 0.11* 0.14*	C024417
Italy, 2000 *Aviso*	722 SL 722 g ai/L	Seedbed drench Spray	70.236. 5 3.5-3.62	19450 20236 588- 602	2 2	0 47 64 70 76	Whole Plant Whole Plant Head Head Head	57 0.02 < 0.008 < 0.008 < 0.008	68* 0.02* < 0.01* < 0.01* < 0.01*	C015429
Italy, 2000 *Aviso*	722 SL 722 g ai/L	Seedbed drench Spray	70.9- 35.8 3.60	19646 19842 600	2 2	0 55 73 80 84	Whole Plant Whole Plant Head Head Head	106 0.02 < 0.008 < 0.008 < 0.008	127* 0.02* < 0.01* < 0.01* < 0.01*	C015429
Italy, 2000 *Aviso*	722 SL 722 g ai/L	Seedbed drench Spray	71.6- 36.89 3.5-3.6	19842 20432 587- 599	2 2	0 40 56 63 68	Whole Plant Whole Plant Head Head Head	59 0.03 < 0.008 < 0.008 < 0.008	70* 0.04* < 0.01* < 0.01* < 0.01*	C015429
Spain, 2001 *Arizona*	722 SL 722 g ai/L	Seedbed drench Spray	72.6- 36.0 2.3	20000 540	2 2	0 49 63	Whole Plant Head Head	52 0.008 < 0.008	62* 0.01* < 0.01*	C024417
Spain, 2001 *Arizona*	722 SL 722 g ai/L	Seedbed drench Spray	71.8- 35.9 2.3-2.2	19880 532- 512	2 2	0 44 56	Whole Plant Head Head	39 0.03 0.008	46* 0.03* 0.01*	C024417
UK 2002 *Freedom*	722 SL 722 g ai/L	Seedbed drench Spray	72.2- 36.0 3.6	20000 600	2 2	0 7 14 22 28	Head Head Head Head Head	1.3 0.10 0.008 <u>0.008</u> 0.05	1.6* 0.12* 0.01* 0.01* 0.06*	C033812
Spain, 2000 *Dunkel*	722 SL 722 g ai/L	Seedbed drench Spray	72.3- 36.0 3.8-3.61	20000 526- 603	2 2	0 116 125 138 141	Whole Plant Whole Plant Head Head Head	54 < 0.008 < 0.008 < 0.008 < 0.008	64* < 0.01* < 0.01* < 0.01* < 0.01*	C015429
Spain, 2000 *Dunkel*	722 SL 722 g ai/L	Seedbed drench Spray	72.2- 36.1 3.4-3.65	20000 472- 607	2 2	0 104 110 113 125	Whole Plant Whole Plant Head Head Head	117 < 0.008 < 0.008 0.05 < 0.008	140* < 0.01* < 0.01* 0.06* < 0.01*	C015429

*actual value reported

Cucumber

Thirty seven trials with propamocarb hydrochloride on cucumber were reported from Europe and the USA. The propamocarb hydrochloride was applied using either drench irrigation, drip irrigation and or spray application. The results are shown in Table 36.

Table 36. Results of residue trials with propamocarb hydrochloride conducted on cucumber receiving drench, drip and/or foliar treatments.

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha or kg ai/hL[a]	Water L/ha	No.	PHI (Days)	Residues as Propamocarb mg/kg	Residues Propamocarb HCl, mg/kg	Report F/GH
Belgium 2004 *Grendel*	840 SL 530 g ai/L	Drench irrig. Drip Irrigation	15.9 1.59		2 4	0 1 3 7 14	1.3* 2.1* 1.4* 0.98* 0.51*		RA 2552/04 GH
France, 2000 *Kansas*	722 SL 722 g ai/L	Seedbed drench Drip irrig. Spray Drip irrig	72.2-36.0 0.144[a] 0.36/0.18[a] 0.144[a]	20000 1215 470-498 1215	2 1 2 1	0 1 4 7 14	1.0* 0.9* 0.8* 1.1* 0.8*	1.2* 1.1* 1.0* 1.3* 0.9*	C016108 GH
Germany 1992, *Orthello*	722 SL 722 g ai/L	Spray	2.2	600	4	0 2 4 7 9	2.9 1.0 0.9 0.67 0.66	3.5* 1.2* 1.1* 0.82* 0.79*	A85339 F
Germany 1992 *Orthello*	722 SL 722 g ai/L	Spray	2.2	600	4	0 2 4 7 9	2.6 1.2 0.9 0.68 0.68	3.1* 1.4* 1.1* 0.81* 0.81*	A85339 F
Germany 1992 *Passavia*	722 SL 722 g ai/L	Spray	2.2	600	4	0 2 4 6 8	1.2 0.9 0.55 0.68 0.55	1.4* 1.1* 0.66* 0.81* 0.65*	A85339 F
Germany 1992 *Profi*	722 SL 722 g ai/L	Spray	2.2	600	4	0 2 4 6 8	0.9 0.62 0.54 0.7 0.62	1.1* 0.74* 0.64* 0.83* 0.74*	A85339 F
Germany 1992 *Profi*	722 SL 722 g ai/L	Spray	2.2	600	4	0 2 4 6 8	4.9 2.5 1.3 1.3 1.1	5.9* 3.0* 1.5* 1.5* 1.3*	A85339 F
Germany 1992 Nienhagen *Alexis*	722 SL 722 g ai/L	Spray	2.5	700	4	0 2 4 7 8	2.1 1.6 1.0 0.9 1.0	2.5* 1.9* 1.2* 1.1* 1.2*	A85339 F
Germany 1991 *Paramount F*	722 SL 722 g ai/L	Spray	2.2	600	4	0 2 4 6 8	2.0 0.69 0.6 0.4 0.54	2.4* 0.82* 0.71* 0.5* 0.64*	A85341 F
Germany 1991 *Orestes*	722 SL 722 g ai/L	Spray	2.2	600	4	0 2 4 6 8	3.5 0.6 0.9 0.54 0.62	4.2* 0.73* 1.1* 0.65* 0.74*	A85341 F

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha or kg ai/hL[a]	Water L/ha	No.	PHI (Days)	Residues as Propamocarb mg/kg	Residues Propamocarb HCl, mg/kg	Report F/GH
Germany 2000 *Korinda*	722 SL 722 g ai/L	Seedbed drench Soil drench Spray Soil drench	29.0 2.6 2.8 2.7	10000 1823 430-630 1860	1 2 2 2	0 3 5 7	1.1* <u>1.3</u>* 0.8* 0.7*	1.3* 1.5* 0.9* 0.8*	C015432 GH
Germany 2000 *Europa*	722 SL 722 g ai/L	Seedbed drench Soil drench Spray Soil drench	28.44 2.7 2.7 2.65	98490 1850 416-604 1832	1 2 2 2	0 3 5 7	1.3* 1.4* <u>1.7</u>* 0.9*	1.5* 1.6* 2.0* 1.1*	C015432 GH
Germany 2000 *Europa*	722 SL 722 g ai/L	Seedbed drench Soil drench Spray Soil drench	28.50 2.2 2.2-2.1 2.2	98680 1714 410-590 1720	1 2 2 2	0 3 5 7	1.6* 1.1* <u>1.4</u>* 0.8*	1.9* 1.3* 1.7* 1.0*	C015432 GH
Germany 2000 *Paramos F1*	722 SL 722 g ai/L	Seedbed drench Soil drench Spray Soil drench	29.0 2.90 3.0 2.90	416667 2000 316-530 2000	1 2 2 2	0 3 5 7	1.2* <u>1.0</u>* 0.8* 0.6*	1.5* 1.2* 1.0* 0.7*	C015432 GH
Germany 2000 *Sudica*	722 SL 722 g ai/L	Seedbed drench Soil drench Spray Soil drench	29.0 2.87 2.8-2.98 2.9	390625 1990 290-515 2000	1 2 2 2	0 3 5 7	0.5* <u>0.7</u>* 0.6* 0.6*	0.6* 0.8* 0.7* 0.7*	C015432 GH
Germany 2000 *Paramos F1*	722 SL 722 g ai/L	Seedbed drench Soil drench Spray Soil drench	29.0 1.9 2.0-1.71 1.9	416667 1300 314-455 1300	1 2 2 2	0 3 5 7	0.7* <u>1.0</u>* 0.9* 0.5*	0.8* 1.2* 1.1* 0.6*	C015432 GH
Germany 2000 *Dominica*	722 SL 722 g ai/L	Seedbed drench Soil drench Spray Soil drench	29.0 1.9 1.9-1.8 1.9	390625 1294 310-481 1315	1 2 2 2	0 3 5 7	0.8* <u>0.8</u>* 0.5* 0.6*	1.0* 1.0* 0.6* 0.7*	C015432 GH
Germany 2001 *Indira RZ F1*	840 SL 530 g ai/L	Seedbed drench Drip irrigation[1]	16 1.5-2.0	20000 1500-1910	2 4	0 3	1.4 <u>0.59</u>	1.7* 0.70*	C021346 GH
Germany 2004 *Pinto F1*	840 SL 530 g ai/L	Drench irrig. Drip Irrigation	15.9 1.59		2 4	0 3	0.04* 0.06*		RA 2552/04 GH
Germany 2004 *Ladner*	840 SL 530 g ai/L	Drench irrig. Drip Irrigation	15.9 1.59		2 4	0 3	0.37* 0.42*		RA 2552/04 GH
Greece 2000 *Palmera*	722 SL 722 g ai/L	Seedbed drench Drip irrig. Spray Drip irrig.	72.2-36.0 2.40 2.0 2.41	20000 1665 570-548 1665	2 1 2 1	0 1 4 7 14	4.0* 3.4* <u>4.8</u>* 4.1* 2.1*	4.7* 4.0* 5.7* 4.8* 2.5*	C016108 GH
Italy, 2000 40026 Imola Emilia-Romag *Kansas*	722 SL 722 g ai/L	Seedbed drench Drip irrig. Spray Drip irrig.	72.5-35.23 3.0 3.0-3.1 3.0	20000 2023 592-618 2083	2 1 2 1	0 1 4 7 14	0.8* 0.9* <u>0.6</u>* 0.5* 0.4*	1.0* 1.0* 0.7* 0.6* 0.5*	C016108 GH
Japan, 1980	SL 640g/kg	sowing	0.16 kg ai/L	300 ml/plant	3	21 35 49	0.39 0.19 0.09	0.46* 0.23* 0.11*	(1) GH

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha or kg ai/hL[a]	Water L/ha	No.	PHI (Days)	Residues as Propamocarb mg/kg	Residues Propamocarb HCl, mg/kg	Report F/GH
Japan, 1980	SL 640g/kg	sowing	0.16 kg ai/L	300 ml/plant	3	21 35 49	0.37 0.33 0.31	0.44* 0.39* 0.37*	(1) GH
Japan, 1980	SL 640g/kg	sowing	0.16 kg ai/L	300 ml/plant	3	21 35 49	0.34 0.16 0.07	0.41* 0.19* 0.09*	(1) GH
Japan, 1980	SL 640g/kg	sowing	0.16 kg ai/L	300 ml/plant	3	21 35 49	0.42 0.22 0.13	0.50* 0.26* 0.16*	(1) GH
Netherlands 2001, *Toledo*	840 SL 530 g ai/L	Seedbed drench Drip irrigation[1]	16 2.66	20000 2500	2 4	0 3	0.78 0.83	0.93* 0.99*	C021346 GH
Netherlands 2001, *Enduro*	840 SL 530 g ai/L	Seedbed drench Drip irrigation[1]	16 2.66	20000 2500	2 4	0 3	0.78 1.0	0.93* 1.2*	C021346 GH
Netherlands 2004 *Grendel*	840 SL 530 g ai/L	Drench irrig Drip Irrigation	15.9 1.59		2 4	0 1 3 7 14	0.39* 1.3* 1.5* 0.75* 0.19*		RA 2552/04 GH
Spain 2000 *Serena*	722 SL 722 g ai/L	Seedbed drench Drip irrig. Spray Drip irrig	72.2-36.0 3.0 2.2-2.1 3.01	20000 2083 399-590 2083	2 1 2 1	0 1 4 7 14	0.5* 0.4* 0.4* 0.4* 0.4*	0.6* 0.5* 0.5* 0.4* 0.4*	C016108 GH
Spain 2000 46800 Xativa Valencia *Cornichon*	722 SL 722 g ai/L	Seedbed drench Drip irrig. Spray Drip irrig	72.2-36.0 2.1 2.2 2.1	20000 1464 397-519 1464	2 1 2 1	0 1 4 7 14	1.4* 1.1* 1.0* 0.6* 0.3*	1.7* 1.3* 1.2* 0.7* 0.4*	C016108 GH
Spain 2000 *Serena*	722 SL 722 g ai/L	Seedbed drench Soil drench Spray Soil drench	29.0 3.2 3.2 3.2	20000 2201 585-734 3669-4403	1 2 2 2	0 3 5 7	1.5* 1.2* 1.4* 1.2*	1.8* 1.4* 1.7* 1.4*	C015432 GH
Spain 2000 *Serena*	722 SL 722 g ai/L	Seedbeddrench Soil drench Spray Soil drench	29.0 3.3 3.5-3.2 3.30	20000 2286 491-589 3810-4571	1 2 2 2	0 3 5 7	0.9* 1.1* 1.7* 1.8*	1.1* 1.3* 2.0* 2.2*	C015432 GH
Spain, 2001 *Serena*	840 SL 530 g ai/L	Seedbed drench Drip irrigation[1]	16 1.99	20000 1875-2343	2 4	0 3	0.56 0.54	0.67* 0.64*	C021346 GH
USA, 1997 *Poinsett 76*	750 SC 375 g ai/L	Spray	1.0	195-200	5	2	0.29*		B002741 F
USA, 1997 *Poinsett 76*	750 SC 375 g ai/L	Spray	1.0	183-187	5	2	0.32*		B002741 F
USA, 1997 *Poinsett 76*	750 SC 375 g ai/L	Spray	1.0	187	5	2	0.26*		B002741 F
USA, 1997 *Dasher 2*	750 SC 375 g ai/L	Spray	1.0	180-199	5	2	0.62*		B002741 F
USA, 1997 *SMR 58*	750 SC 375 g ai/L	Spray	1.0	184-196	5	2	0.69*		B002741 F

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha or kg ai/hL[a]	Water L/ha	No.	PHI (Days)	Residues as Propamocarb mg/kg	Residues Propamocarb HCl, mg/kg	Report F/GH
USA, 1997 *Straight Eight*	750 SC 375 g ai/L	Spray	1.0	183-201	5	2	0.75*		B002741 F
USA, 1997 *Pointsett 76*	750 SC 375 g ai/L	Spray	1.0	186-196	5	1 2 4 6 8	0.55* 0.61* 0.26* 0.19* 0.16*		B002741 F

*actual value reported; 1. only a summary report of the trial was provided;

Melons

Fourty eight field and glasshouse trials in melons were reported from Europe, spanning 1993 to 2004. Seven trials were also reported in field grown cantaloupe, conducted in 1997, from the USA. Methods of application included the use of drench, drip and/or foliar spray (Table 37).

Table 37. Residue trials with propamocarb hydrochloride conducted in melon in the field and glass house.

Country, Year of trial *Variety*	Aplication Form.	Method	kg ai/ha	Water L/ha	No.	PHI (Days)	Sample analysed	Residues as Propamocarb mg/kg	Residue Propamocab HCl, mg/kg	Report F/GH
France 1993 *Delta*	750 SC 375 g ai/L	Spray	2.2	333	5	3 5 7	Fruit Fruit Fruit	0.44 0.81 0.17	0.53* 0.97* 0.2*	A89361 F
France 1993 *Bastion*	750 SC 375 g ai/L	Spray	2.2	333	4	3 5 7	Fruit Fruit Fruit	0.49 0.13 0.3	0.58* 0.15* 0.3*	A89361 F
France 1994 *Manta*	750 SC 375 g ai/L	Spray	1.1	333	3	0 3 0 3	Fruit Fruit Pulp Pulp	0.60 0.38 < 0.08 ≤ 0.08	0.72* 0.45* < 0.1* < 0.1*	A89363 F
France 1994 *Bastion*	750 SC 375 g ai/L	Spray	1.1	333	3	0 3 0 3	Fruit Fruit Pulp Pulp	0.32 0.11 < 0.08 ≤ 0.08	0.38* 0.13* < 0.1* < 0.1*	A89363 F
France 1994 *Delta*	750 SC 375 g ai/L	Spray	1.1	333	3	0 3 0 3	Fruit Fruit Pulp Pulp	0.18 0.23 < 0.08 ≤ 0.08	0.21* 0.28* < 0.1* < 0.1*	A89363 F
France 1994 *Delta*	750 SC 375 g ai/L	Spray	1.1	333	3	0 3 0 3	Fruit Fruit Pulp Pulp	0.18 0.24 0.13 0.21	0.22* 0.29* 0.15* 0.25*	A89363 F
France 1994 *Manta*	750 SC 375 g ai/L	Spray	2.2	333	3	0 3 0 3	Fruit Fruit Pulp Pulp	0.49 0.44 < 0.08 ≤ 0.08	0.58* 0.52* < 0.1* < 0.1*	A89363 F
France 1994 *Bastion*	750 SC 375 g ai/L	Spray	2.2	333	3	0 3 0 3	Fruit Fruit Pulp Pulp	0.32 0.28 < 0.08 ≤ 0.08	0.38* 0.34* < 0.1* < 0.1*	A89363 F
France *Delta*	750 SC 375 g ai/L	Spray	2.2	333	3	0 3 0 3	Fruit Fruit Pulp Pulp	0.44 0.40 0.11 ≤ 0.08	0.52* 0.48* 0.13* < 0.1*	A89363 F

Country, Year of trial *Variety*	Aplication						Sample analysed	Residues as Propamocarb mg/kg	Residue Propamocab HCl, mg/kg	Report F/GH
	Form.	Method	kg ai/ha	Water L/ha	No.	PHI (Days)				
France	750 SC	Spray	2.2	333	3	0	Fruit	0.70	0.83*	A89363
1994	375 g ai/L					3	Fruit	1.1	1.3*	F
Delta						0	Pulp	0.13	0.16*	
						3	Pulp	0.13	0.15*	
France	722 SL	Spray	2.1	300	3	0	Fruit	0.54	0.64*	A83662
1995	722 g ai/L					3	Fruit	0.65	0.78*	F
Alpha						0	Pulp	0.08	0.1*	
						3	Pulp	0.07	0.08*	
France	722 SL	Spray	2.1	300	3	0	Fruit	1.8	2.2*	A83662
1995	722 g ai/L					3	Fruit	0.92	1.1*	F
Sierra						0	Pulp	0.09	0.11*	
						3	Pulp	0.04	0.05*	
France	722 SL	Spray	2.1	300	3	0	Fruit	1.17	1.4*	A83662
1995	722 g ai/L					3	Fruit	0.57	0.68*	F
Averell						0	Pulp	0.1	0.12*	
						3	Pulp	≤ 0.04	< 0.05*	
France	722 SL	Spray	2.1	300	3	0	Fruit	0.84	1.0*	A83662
1995	722 g ai/L					3	Fruit	0.38	0.45*	F
Dalton						0	Pulp	< 0.04	< 0.05*	
						3	Pulp	≤ 0.04	< 0.05*	
France	722 SL	Seedbed	22	20000	2	0	Fruit	0.84*	1.0*	C017451
2000	722 g ai/L	drench				3	Fruit	0.50*	0.6*	F
Galonbet		Spray	2.2	1046	2	7	Fruit	0.25*	0.3*	
						14	Fruit	0.25*	0.3*	
						21	Fruit	0.25*	0.3*	
						0	Peel	3.2*	3.9*	
						3	Peel	1.0*	1.2*	
						7	Peel	1.5*	1.8*	
						14	Peel	0.59*	0.7*	
						21	Peel	0.7*	0.9*	
						0	Pulp	< 0.01*	< 0.01*	
						3	Pulp	< 0.01*	< 0.01*	
						7	Pulp	< 0.01*	< 0.01*	
						14	Pulp	< 0.01*	< 0.01*	
						21	Pulp	0.02*	0.02*	
France	722 SL	Seedbed	22	20000	2	0	Fruit	0.42*	0.5*	C017451
2000	722 g ai/L	drench				3	Fruit	0.34*	0.4*	F
Innenheim		Spray	2.2	1025	2	7	Fruit	0.17*	0.2*	
Bastion						14	Fruit	0.09*	0.1*	
						21	Fruit	0.17*	0.2*	
						0	Peel	1.0*	1.2*	
						3	Peel	1.1*	1.3*	
						7	Peel	0.43*	0.5*	
						14	Peel	0.25*	0.3*	
						21	Peel	0.25*	0.3*	
						0	Pulp	< 0.01*	< 0.01*	
						3	Pulp	< 0.01*	< 0.01*	
						7	Pulp	< 0.01*	< 0.01*	
						14	Pulp	< 0.01*	< 0.01*	
						21	Pulp	≤ 0.008*	< 0.01*	

Country, Year of trial *Variety*	Aplication					PHI (Days)	Sample analysed	Residues as Propamocarb mg/kg	Residue Propamocab HCl, mg/kg	Report F/GH
	Form.	Method	kg ai/ha	Water L/ha	No.					
France	722 SL	Seedbed	22	20000	2	0	Fruit	1.5*	1.7*	C017451
2000	722 g ai/L	drench				3	Fruit	0.67*	0.8*	
Ardor F1		Spray	2.2	1007	2	7	Fruit	0.76*	0.9*	F
						14	Fruit	0.50*	0.6*	
						21	Fruit	0.67*	0.8*	
						0	Peel	3.4*	4.1*	
						3	Peel	1.9*	2.2*	
						7	Peel	3.2*	3.8*	
						14	Peel	1.3*	1.6*	
						21	Peel	1.0*	1.2*	
						0	Pulp	< 0.01*	< 0.01*	
						3	Pulp	< 0.01*	< 0.01*	
						7	Pulp	0.02*	0.02*	
						14	Pulp	0.02*	0.02*	
						21	Pulp	0.02*	0.02*	
France	722 SL	Seedbed	24	22292	2	0	Fruit	2.5*	3.0*	C017451
2000	722 g ai/L	drench				3	Fruit	0.59*	0.7*	F
Heliobel		Spray	2.2	1039	2	7	Fruit	0.50*	0.6*	
						14	Fruit	0.67*	0.8*	
						21	Fruit	0.17*	0.2*	
						0	Peel	1.1*	1.3*	
						3	Peel	1.0*	1.1*	
						7	Peel	2.2*	2.6*	
						14	Peel	1.4*	1.6*	
						21	Peel	0.84*	1.0*	
						0	Pulp	0.59*	0.7*	
						3	Pulp	0.09*	0.1*	
						7	Pulp	0.07*	0.08*	
						14	Pulp	0.06*	0.07*	
						21	Pulp	0.03*	0.04*	
France	722 SL	Seedbed	22	11146	2	0	Fruit	1.8*	2.2*	C017451
2000	722 g ai/L	drench				3	Fruit	2.5*	2.9*	F
Buffalo		Spray	2.2	995	2	7	Fruit	1.5*	1.8*	
						14	Fruit	1.5*	1.8*	
						21	Fruit	0.3*	0.4*	
						0	Peel	5.4*	6.4*	
						3	Peel	9.7*	12.0*	
						7	Peel	5.5*	6.5*	
						14	Peel	4.8*	5.8*	
						21	Peel	1.1*	1.3*	
						0	Pulp	0.34*	0.4*	
						3	Pulp	0.08*	0.1*	
						7	Pulp	0.04*	0.05*	
						14	Pulp	0.07*	0.08*	
						21	Pulp	0.02*	0.02*	
France	722 SL	Seedbed	21.6	19966	2	0	Fruit	0.52*	0.62*	C020966
2001	722 g ai/L	drench				13	Fruit	0.51*	0.61*	F
Marlene		Spray	2.33	538	2	0	Peel	3.3*	3.9*	
						13	Peel	1.4*	1.7*	
						0	Pulp	0.39*	0.47*	
						13	Pulp	0.02*	0.02*	
France	722 SL	Seedbed	21.5	19886	2	0	Fruit	0.80*	0.95*	C020966
2001	722 g ai/L	drench				14	Fruit	0.34*	0.41*	F
Sierra		Spray	2.2	511	2	0	Peel	3.8*	4.5*	
						14	Peel	0.62*	0.74*	
						0	Pulp	0.07*	0.08*	
						14	Pulp	< 0.01*	0.01*	

Country, Year of trial *Variety*	Aplication							Residues as Propamocarb mg/kg	Residue Propamocab HCl, mg/kg	Report F/GH
	Form.	Method	kg ai/ha	Water L/ha	No.	PHI (Days)	Sample analysed			
France	722 SL	Seedbed	22	20000	2	0	Fruit	0.15*	0.17*	C020952
2001	722 g ai/L	drench				14	Fruit	0.18*	0.21*	GH
Fiesta		Spray	2.1	492	2	0	Peel	1.4*	1.7*	
						14	Peel	1.3*	1.5*	
						0	Pulp	0.05*	0.06*	
						14	Pulp	0.02*	0.02*	
Germany	840 SL	Drench	16.0	20030	2	0	Fruit	0.44	0.53*	C024908
2001	530 g ai/L			-		13	Fruit	1.42	1.7*	GH
Melina F1		Drip	2.0	21380	2	0	Peel	0.60	0.71*	
		Irrigation		1860-		13	Peel	0.32	0.38*	
				1900		0	Pulp	0.14	0.17*	
						13	Pulp	0.08	0.09*	
Greece	722 SL	Seedbed	21.4	19770	2	0	Fruit	2.4*	2.9*	C017451
2000	722 g ai/L	drench				3	Fruit	1.2*	1.5*	F
Ananas		Spray	2.2	1045	2	7	Fruit	1.7*	2.0*	
						14	Fruit	0.76*	0.9*	
						21	Fruit	0.08*	0.1*	
						0	Peel	6.3*	7.6*	
						3	Peel	5.2*	6.2*	
						7	Peel	2.6*	3.1*	
						14	Peel	1.4*	1.7*	
						21	Peel	0.25*	0.3*	
						0	Pulp	0.50*	0.6*	
						3	Pulp	0.17*	0.2*	
						7	Pulp	0.08*	0.1*	
						14	Pulp	0.04*	0.05*	
						21	Pulp	0.02*	0.02*	
Greece	722 SL	Seedbed	22	20000	2	0	Fruit	1.2*	1.5*	C016109
2000	722 g ai/L	drench				3	Fruit	2.6*	3.1*	
Gallia		Spray	2.1	1013	2	7	Fruit	1.9*	2.3*	GH
						14	Fruit	1.5*	1.8*	
						21	Fruit	2.2*	2.6*	
						0	Peel	2.0*	2.4*	
						3	Peel	5.3*	6.3*	
						7	Peel	3.1*	3.7*	
						14	Peel	4.1*	4.9*	
						21	Peel	5.6*	6.6*	
						0	Pulp	0.08*	0.1*	
						3	Pulp	0.17*	0.2*	
						7	Pulp	0.07*	0.08*	
						14	Pulp	0.08*	0.1*	
						21	Pulp	0.08*	0.1*	
Italy, 2000	722 SL	Seedbed	22	20722	2	0	Fruit	2.7*	2.6*	C016109
Scudo	722 g ai/L	drench				3	Fruit	1.0*	1.2*	
		Spray	2.2	1029	2	7	Fruit	0.84*	1.0*	GH
						14	Fruit	0.76*	0.9*	
						21	Fruit	0.67*	0.8*	
						0	Peel	1.8*	2.2*	
						3	Peel	1.3*	1.6*	
						7	Peel	1.9*	2.2*	
						14	Peel	1.5*	1.7*	
						21	Peel	0.76*	0.9*	
						0	Pulp	0.08*	0.1*	
						3	Pulp	0.02*	0.02*	
						7	Pulp	0.02*	0.02*	
						14	Pulp	0.02*	0.02*	
						21	Pulp	0.02*	0.02*	

Country, Year of trial *Variety*	Aplication					PHI (Days)	Sample analysed	Residues as Propamocarb mg/kg	Residue Propamocab HCl, mg/kg	Report F/GH
	Form.	Method	kg ai/ha	Water L/ha	No.					
Italy	722 SL	Seedbed	21	19760	2	0	Fruit	1.5*	1.8*	C017451
2000	722 g ai/L	drench				3	Fruit	0.67*	0.8*	
Bingo		Spray	2.1	992	2	7	Fruit	1.0*	1.2*	F
						14	Fruit	0.42*	0.5*	
						21	Fruit	0.59*	0.7*	
						0	Peel	2.2*	2.6*	
						3	Peel	1.7*	2.0*	
						7	Peel	1.4*	1.6*	
						14	Peel	1.0*	1.2*	
						21	Peel	0.17*	0.2*	
						0	Pulp	0.02*	0.02*	
						3	Pulp	0.02*	0.02*	
						7	Pulp	< 0.01*	< 0.01*	
						14	Pulp	0.02*	0.02*	
						21	Pulp	0.03*	0.04*	
Italy	722 SL	Seedbed	22	19966	2	0	Fruit	0.53*	0.63*	C020952
2001	722 g ai/L	drench				14	Fruit	0.19*	0.22*	GH
Bingo		Spray	2-2.2	484-516	2	0	Peel	1.1*	1.3*	
						14	Peel	0.34*	0.4*	
						0	Pulp	0.02*	0.02*	
						14	Pulp	< 0.01*	0.01*	
Italy	722 SL	Drench	20-22.0	18604 -	2	0	Fruit	0.59*	0.7*	C020966
2001	722 g ai/L					14	Fruit	0.11*	0.13*	F
Bingo		Spray	2.2	20000	2	0	Peel	1.6*	1.8*	
				513		14	Peel	0.19*	0.23*	
						0	Pulp	0.02*	0.03*	
						14	Pulp	< 0.01*	< 0.01*	
Italy, 2004	840 SL	Drench	15.9	20000	2	0	Fruit	< 0.008*	< 0.01	RA 2554/04
Proteo	530 g ai/L	Drip Irrigation	1.59	100	2	14	Fruit	< 0.008*	< 0.01	GH
Italy, 2004	840 SL	Drench	15.9	20000	2	0	Fruit	< 0.008*	< 0.01	RA 2554/04
H. Best Jumbo	530 g ai/L	Drip Irrigation	1.59	100	2	14	Fruit	< 0.008*	< 0.01	GH
Netherlands	722 SL	Spray	2.1	1543	3	0	Fruit	0.80	0.96*	C004255
1997	722 g ai/L					3	Fruit	0.12	0.14*	
Lunastar						0	Peel	1.1	1.3*	GH
						3	Peel	0.31	0.37*	
						0	Pulp	< 0.04	< 0.05*	
						3	Pulp	< 0.04	< 0.05*	
Netherlands	722 SL	Spray	2.1	1493	3	0	Fruit	0.61	0.73*	C004255
1997	722 g ai/L					3	Fruit	0.1	0.15*	
Lunastar						0	Peel	1.3	1.5*	GH
						3	Peel	0.55	0.66*	
						0	Pulp	0.04	0.05*	
						3	Pulp	< 0.04	< 0.05*	
Netherlands	722 SL	Spray	2.2	1547	3	0	Fruit	0.52	0.62*	C004255
1997	722 g ai/L					3	Fruit	0.14	0.17*	
Lunastar						0	Peel	1.3	1.5*	GH
						3	Peel	0.9	1.1*	
						0	Pulp	0.04	0.05*	
						3	Pulp	< 0.04	< 0.05*	
Portugal	840 SL	Drench	15.9	20000	2	0	Fruit	< 0.008*	< 0.01	RA 2554/04
2004, *Galas*	530 g ai/L	Drip Irrigation	1.59	100	2	14	Fruit	0.12*	0.14	GH
Portugal	840 SL	Drench	15.9	20000	2	0	Fruit	< 0.008*	< 0.01	RA 2554/04
2004	530 g ai/L	Drip	1.59	100	2	1	Fruit	0.02*	0.02	GH
Ananas de America		Irrigation				3	Fruit	0.173*	0.2	
						7	Fruit	0.02*	0.02	
						14	Fruit	0.04*	0.05	

Country, Year of trial *Variety*	Aplication						Sample analysed	Residues as Propamocarb mg/kg	Residue Propamocab HCl, mg/kg	Report F/GH
	Form.	Method	kg ai/ha	Water L/ha	No.	PHI (Days)				
Spain, 2004	840 SL	Drench	15.9	20000	2	0	Fruit	< 0.008*	< 0.01	RA 2554/04
Vulcano	530 g ai/L	Drip	1.59	100	2	1	Fruit	< 0.008*	< 0.01	
		Irrigation				3	Fruit	< 0.008*	< 0.01	GH
						7	Fruit	< 0.008*	< 0.01	
						14	Fruit	< 0.008*	< 0.01	
Spain, 2001	840 SL	Drench	16.0	20300	2	0	Fruit	0.63	0.75*	C024908
Deltex F1	530 g ai/L			-		15	Fruit	1.0	1.2*	GH
(Galia)		Drip	2.0	19930	2	0	Peel	1.9	2.3*	
		Irrigation		1600		15	Peel	2.0	2.4*	
						0	Pulp	0.62	0.74*	
						15	Pulp	0.53	0.63*	
Spain, 2001	840 SL	Drench	16.0	20000	2	0	Fruit	0.18	0.22*	C024908
Deltex F1	530 g ai/L	Drip	2.0	2340	2	14	Fruit	0.21	0.25*	GH
(Galia)		Irrigation				0	Peel	0.35	0.42*	
						14	Peel	0.4	0.48*	
						0	Pulp	0.04	0.05*	
						14	Pulp	0.06	0.07*	
Spain, 2001	840 SL	Drench	16.0	20000	2	0	Fruit	0.36	0.43*	C024908
Deltex F	530 g ai/L	Drip	2.0	2610	2	13	Fruit	0.45	0.54*	GH
(Galia)		Irrigation				0	Peel	1.1	1.3*	
						13	Peel	1.0	1.2*	
						0	Pulp	0.29	0.35*	
						13	Pulp	0.17	0.2*	
Spain	722 SL	Drench	22.0	20000	2	0	Fruit	0.17*	0.20*	C020966
2001	722 g ai/L	Spray	2.1-2.3	492-	2	13	Fruit	0.10*	0.12*	F
Pinonet				538		0	Peel	0.07*	0.09*	
						13	Peel	0.30*	0.36*	
						0	Pulp	0.03*	0.04*	
						13	Pulp	0.01*	0.02*	
Spain	722 SL	Drench	22.0	20000	2	0	Fruit	1.4*	1.7*	C020966
2001	722 g ai/L	Spray	2.1	500	2	14	Fruit	0.93*	1.1*	F
Pinonet						0	Peel	1.7*	2.0*	
						14	Peel	1.3*	1.6*	
						0	Pulp	0.29*	0.35*	
						14	Pulp	0.03*	0.03*	
Spain	722 SL	Drench	22.0	20000	2	0	Fruit	0.35*	0.41*	C020952
2001	722 g ai/L	Spray	2.2	514	2	14	Fruit	0.12*	0.15*	GH
Pinonet						0	Peel	0.73*	0.87*	
						14	Peel	0.39*	0.46*	
						0	Pulp	0.08*	0.1*	
						14	Pulp	0.02*	0.02*	
Spain	722 SL	Drench	22.0	20000	2	0	Fruit	0.81*	0.97*	C020952
2001	722 g ai/L	Spray	2.2	521	2	14	Fruit	0.56*	0.67*	GH
Pinonet						0	Peel	1.4*	1.7*	
						14	Peel	0.92*	1.1*	
						0	Pulp	0.19*	0.23*	
						14	Pulp	0.03*	0.04*	
Spain	722 SL	Drench	22	20000	2	0	Fruit	0.84*	1.0*	C017451
2000	722 g ai/L	Spray	2.1	999	2	3	Fruit	0.34*	0.4*	F
Sancho						7	Fruit	0.92*	1.1*	
						14	Fruit	0.25*	0.3*	
						21	Fruit	0.17*	0.2*	
						0	Peel	1.0*	1.2*	
						3	Peel	1.0*	1.2*	
						7	Peel	0.50*	0.6*	
						14	Peel	0.67*	0.8*	
						21	Peel	0.42*	0.5*	
						0	Pulp	0.008*	0.01*	
						3	Pulp	0.008*	0.01*	
						7	Pulp	0.008*	0.01*	
						14	Pulp	0.008*	0.01*	
						21	Pulp	0.008*	0.01*	

Country, Year of trial *Variety*	Aplication					PHI (Days)	Sample analysed	Residues as Propamocarb mg/kg	Residue Propamocab HCl, mg/kg	Report F/GH
	Form.	Method	kg ai/ha	Water L/ha	No.					
France	722 SL	Drench	24	22230	2	0	Fruit	0.84*	1.0*	C016109
2000	722 g ai/L	Spray	2.1	1004	2	4	Fruit	0.42*	0.5*	
Lunastar						7	Fruit	0.42*	0.5*	GH
						14	Fruit	0.17*	0.2*	
						21	Fruit	0.08*	0.1*	
						0	Peel	1.8*	2.2*	
						4	Peel	1.1*	1.3*	
						7	Peel	1.8*	2.2*	
						14	Peel	0.7*	0.8*	
						21	Peel	0.25*	0.3*	
						0	Pulp	0.08*	0.1*	
						4	Pulp	0.08*	0.1*	
						7	Pulp	0.04*	0.05*	
						14	Pulp	0.02*	0.02*	
						21	Pulp	0.08*	0.01*	
Spain	722 SL	Drench	22	20000	2	0	Fruit	0.4*	0.5*	C016109
2000	722 g ai/L	Spray	2.2	1031	2	3	Fruit	0.34*	0.4*	
Sancho						7	Fruit	0.34*	0.4*	GH
						14	Fruit	0.25*	0.3*	
						21	Fruit	0.07*	0.08*	
						0	Peel	1.0*	1.2*	
						3	Peel	0.34*	0.4*	
						7	Peel	0.34*	0.4*	
						14	Peel	0.42*	0.5*	
						21	Peel	0.25*	0.3*	
						0	Pulp	0.25*	0.21*	
						3	Pulp	0.01*	0.01*	
						7	Pulp	0.02*	0.02*	
						14	Pulp	< 0.01*	< 0.01*	
						21	Pulp	0.01*	0.01*	
Spain	722 SL	Drench	22	20000	2	0	Fruit	0.17*	0.2*	C016109
2000	722 g ai/L	Spray	2.1	992	2	3	Fruit	0.18*	0.2*	
Sancho						7	Fruit	0.18*	0.2*	GH
						14	Fruit	0.08*	0.1*	
						21	Fruit	0.04*	0.05*	
						0	Peel	0.50*	0.6*	
						3	Peel	0.60*	0.8*	
						7	Peel	0.3*	0.3*	
						14	Peel	0.20*	0.3*	
						21	Peel	0.17*	0.2*	
						0	Pulp	< 0.01*	< 0.01*	
						3	Pulp	< 0.01*	< 0.01*	
						7	Pulp	< 0.01*	< 0.01*	
						14	Pulp	< 0.01*	< 0.01*	
						21	Pulp	0.17*	0.2*	
USA, 1997	750 SC	Spray	1.0	191-195	5	2	Fruit	0.29*		B002741 F
Hale´s best										
USA, 1997	750 SC	Spray	1.0	187	5	2	Fruit	1.4*		B002741 F
USA, 1997	750 SC	Spray	1.0	179-199	5	1	Fruit	0.34*		B002741 F
Perlita										
USA, 1997	750 SC	Spray	1.0	182-195	5	2	Fruit	0.77*		B002741 F
Tam uvalde										
USA, 1997	750 SC	Spray	1.0	185-198	5	2	Fruit	0.44*		B002741 F

Country, Year of trial *Variety*	Aplication					PHI (Days)	Sample analysed	Residues as Propamocarb mg/kg	Residue Propamocab HCl, mg/kg	Report F/GH
	Form.	Method	kg ai/ha	Water L/ha	No.					
USA, 1997	750 SC	Spray	1.0	185-198	5	2	Fruit	0.29		B002741 F
USA, 1997 *Top mark*	750 SC	Spray	1.0	187-188	5	1 2 4 6 8	Fruit	0.90* 0.66* 0.60* 0.50* 0.26*		B002741 F

*actual value reported

Summer squash

Six field trials were reported with propamocarb in summer squash, from the USA, conducted in 1997 (Table 38).

Table 38: Residue field trials with propamocarb hydrochloride conducted in summer squash received foliar treatment (Report B002741).

Country, Year of trial *Variety*	Application					PHI (Days	Residues as Propamocarb mg/kg
	Form.	Method	kg ai/ha	Water L/ha	No.		
USA, 1997 *Supreme*	750 SC	Spray	1.0	191-195	5	2	0.99*
USA, 1997 *E Yellow Straightneck*	750 SC	Spray	1.0	187	5	2	0.49*
USA, 1997 *Dark Green Zucchini*	750 SC	Spray	1.0	179-199	5	2	0.37*
USA, 1997 *Early Polific Strain*	750 SC	Spray	1.0	182-195	5	2	1.1*
USA, 1997 Samma Yellow	750 SC	Spray	1.0	185-198	5	2	0.43*
USA, 1997 *Straightneck Early*	750 SC	Spray	1.0	187-188	5	1 2 4 6 8	0.51* 0.64* 0.63* 0.58* 0.48*

Peppers, sweet

Thirty five trials were conducted with propamocarb hydrochloride on greenhouse (GH) grown sweet peppers in Europe from 1999 to 2004 and 10 trials in field (F) grown sweet peppers from the USA in 1997 using drench, drip or foliar treatment (Table 39).

Table 39. Residue trials with propamocarb hydrochloride conducted on sweet pepper in the greenhouse (Europe) and in the field (USA).

Country, Year of trial *Variety*	Application					PHI (Days	Residues as Propamocarb mg/kg	Residues Propamocarb HCl, mg/kg	Report no.
	Form.	Method	kg ai/ha	Water L/ha	No.				
Belgium 2004, *Rapido*	840 SL 530 g ai/L	Drench irrig. Drip irrig.	31.8-15.9 1.59	20000 250	2 1 4	0 1 3	0.20* 0.12* 0.16*	0.24 0.14 0.19	RA 2559/04
Germany 2001 *Bell Boy F1*	840 SL 530 g ai/L	Seedbed drench Drench/drip.	16-32 1.9	20000 1800	2 4	0 3	0.08 0.10	0.10* 0.13*	C024482

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha	Water L/ha	No	PHI (Days	Residues as Propamocarb mg/kg	Residues Propamocarb HCl, mg/kg	Report no.
Germany,2001	840 SL	Seedbed	15-32	20000	2	0	0.09	0.11*	C024482
Mazurka RZ	530 g ai/L	drench Drench/drip.	1.9-2.5	1800	4	3	0.11	0.13*	
Greece	722 SL	Seedbed	72.2-36	20000	2	0	< 0.03*	< 0.04*	C016110
2000	722 g	drench				1	0.05*	0.06*	
Balo	ai/L	Drench/drip.	2.4	1667	3	3	< 0.008*	< 0.01*	
						7	< 0.008*	< 0.01*	
						14	< 0.008*	< 0.01*	
Greece	840 SL	Seedbed	16-32	20000	2	0	0.16	0.19*	C024482
2001 *Florina*	530 g ai/L	drench Drench/drip.	3.31	3125	4	3	1.0	1.2*	
Greece, 2003	840 SL	Seedbed	15.9	5000-	1	0	0.07	0.08*	C048490
Raiko RZ	530 g ai/L	drench Drench irrig..	1.59	10000 1267	1 4	3	0.14	0.17*	
Italy	722 SL	Seedbed	74.4-	20000	2	0	< 0.008*	< 0.01*	C016110
2000	722 g	drench	35.7			1	< 0.008*	< 0.01*	
Linares	ai/L	Drench/drip.	1.93	1333	3	3	< 0.008*	< 0.01*	
						7	< 0.008*	< 0.01*	
						14	< 0.008*	< 0.01*	
Italy, 2001	840 SL	Seedbed	16-32	20000	2	0	0.61	0.73*	C024482
Magnigold	530 g ai/L	drench Drench/drip	3.22	3030	4	3	0.22	0.26*	
Italy	840 SL	Drench irrig.	31.8-	20000	2	0	0.01*	0.01	RA 2559/04
2004	530 g		15.9			1	0.02*	0.02	
Adina	ai/L	Drip irrig.	1.59	100	4	3	0.02*	0.02	
Italy, 2003	840 SL	Seedbed	15.9	5000-	2	0	0.008	< 0.01*	C048490
Valdor	530 g ai/L	drench Drench irrig..	1.59	10000 1267	4	3	0.008	< 0.01*	
Netherlands	722 SL	Seedbed	29.0	50000	1	0	< 0.008	< 0.01*	C016842
1999	722 g	drench	0.72-	1500-	4	3	< 0.008	< 0.01*	
Mazurka	ai/L	Drench	2.17	3000		5	< 0.008	< 0.01*	
						7	< 0.008	< 0.01*	
Netherlands	722 SL	Seedbed	29.0	50000	1	0	< 0.008	< 0.01*	C016842
1999	722 g	drench	0.72-	1500-	4	3	< 0.008	< 0.01*	
Fiesta	ai/L	Drench	2.17	3000		5	< 0.008	< 0.01*	
						7	< 0.008	< 0.01*	
Netherlands	722 SL	Seedbed	29.0	50000	1	0	< 0.008	< 0.01*	C016842
1999	722 g	drench				3	< 0.008	< 0.01*	
Mazurka	ai/L	Drench	0.72-	1500-	4	5	< 0.008	< 0.01*	
			2.17	3000		7	< 0.008	< 0.01*	
Netherlands	722 SL	Seedbed	29.0	50000	1	0	< 0.008	< 0.01*	C016842
1999	722 g	drench				3	< 0.008	< 0.01*	
Fiesta	ai/L	Drench	0.72-	1500-	4	5	< 0.008	< 0.01*	
			2.17	3000		7	< 0.008	< 0.01*	
Netherlands	722 SL	Seedbed	29.0	50000	1	0	< 0.008	< 0.01*	C016842
1999	722 g	drench				3	0.06	0.07*	
Spirit	ai/L	Drench	0.72-	1500-	4	5	< 0.008	< 0.01*	
			2.17	3000		7	< 0.008	< 0.01*	
Netherlands	722 SL	Seedbed	29.0	50000	1	0	< 0.008	< 0.01*	C016842
1999	722 g	drench				3	< 0.008	< 0.01*	
Basanova	ai/L	Drench	0.72-	1500-	4	5	< 0.008	< 0.01*	
			2.17	3000		7	< 0.008	< 0.01*	

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha	Water L/ha	No	PHI (Days	Residues as Propamocarb mg/kg	Residues Propamocarb HCl, mg/kg	Report no.
Netherlands 1999 *Spirit*	722 SL 722 g ai/L	Seedbed drench Drench	29.0 0.72- 2.17	50000 1500- 3000	1 4	0 3 5 7	0.10 <u>< 0.008</u> < 0.008 0.008	0.12* < 0.01* < 0.01* 0.01*	C016842
Netherlands 1999 *Basanova*	722 SL 722 g ai/L	Seedbed drench Drench	29.0 0.72- 2.17	50000 1500- 3000	1 4	0 3 5 7	< 0.008 <u>< 0.008</u> < 0.008 < 0.008	< 0.01* < 0.01* < 0.01* < 0.01*	C016842
Netherlands 2003, *Zerto*	840 SL 530 g ai/L	Seedbed drench Drench irrig..	15.9 1.59	5000- 10000 1267	2 4	0 3	0.02 <u>0.03</u>	0.02* 0.03*	C048490
Netherlands 2004, *Festivo*	840 SL 530 g ai/L	Drench irrig. Drip irrig..	31.8- 15.9 1.59	20000 250	2 4	0 1 3	0.15* 0.18* <u>0.15*</u>	0.18 0.21 0.18	RA 2559/04
Spain, 2003 *Flamenco*	840 SL 530 g ai/L	Seedbed drench Drench irrig..	15.9 1.59	5000- 10000 1267	2 4	0 3	0.02 <u>0.008</u>	0.02* < 0.01*	C048490
Spain 2004 *Olmo*	840 SL 530 g ai/L	Drench irrig. Drip irrig.	31.8- 15.9 0.60 1.59	20000 - 100	2 1 4	0 1 3	0.12* 0.11* <u>0.08*</u>	0.14 0.13 0.10	RA 2559/04
Spain 2000 *Turia*	722 SL 722 g ai/L	Seedbed drench Drench/drip.	72.2-36 5.55	20000 3846	2 3	0 1 3 7 14	0.04* 0.02* 0.05* 0.03* 0.03*	0.05* 0.02* 0.06* 0.04* 0.04*	C016110
Spain 2000 *Taliano*	722 SL 722 g ai/L	Seedbed drench Drench/drip.	72.2-36 2.7	20000 1860- 9354	2 3	0 1 3 14	< 0.008* < 0.008* < 0.008* 0.02*	< 0.01* < 0.01* < 0.01* 0.02*	C016110
Spain 2000 *Cipari*	722 SL 722 g ai/L	Seedbed drench Drench/drip.	72.2-36 1.5	20000 1082	2 3	0 1 3 7 14	< 0.008* 0.008* 0.008* 0.008* 0.008*	< 0.01* 0.01* 0.01* 0.01* 0.01*	C016110
USA, 1997 *Wonder*	750 SC 375 g ai/L	Spray	1.0	186- 191	5	5	<u>0.27*</u>	0.32	B003364
USA, 1997 *Enterprise*	750 SC 375 g ai/L	Spray	1.0	165- 182	5	5	<u>0.62*</u>	0.74	B003364
USA, 1997 *Camelot X3R*	750 SC 375 g ai/L	Spray	1.0	182- 192	5	4	<u>0.32*</u>	0.38	B003364
US, 1997 *Jupiter*	750 SC 375 g ai/L	Spray	1.0	183- 189	5	5	<u>0.07*</u>	0.08	B003364
USA, 1997 *Bell*	750 SC 375 g ai/L	Spray	1.0	174- 197	5	5	<u>1.8*</u>	1.4	B003364
USA, 1997 *Jalepeno*	750 SC 375 g ai/L	Spray	1.0	187- 190	5	5	<u>0.16*</u>	0.19	B003364
USA, 1997 *Big Jim*	750 SC 375 g ai/L	Spray	1.0	176- 195	5	5	<u>0.23*</u>	0.27	B003364

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha	Water L/ha	No	PHI (Days	Residues as Propamocarb mg/kg	Residues Propamocarb HCl, mg/kg	Report no.
USA, 1997 *Jupiter*	750 SC 375 g ai/L	Spray	1.0	188-190	5	5	0.26*	0.31	B003364
USA, 1997 *TMR23*	750 SC 375 g ai/L	Spray	1.0	187-190	5	5	0.98*	1.2	B003364
USA, 1997 *Yolo Wonder B*	750 SC 375 g ai/L	Spray	1.0	187-190	5	1 3 5 7 9	0.18* 0.32* 0.20* 0.16* 0.18*	0.21 0.38 0.24 0.19 0.21	B003364

*actual value reported

Tomato

Fourty four tomato trials from Europe and 18 trials from the USA were reported. The European results were reported from either greenhouses (GH) or field (F) trials while the US data was all from field trials. The results are shown in Table 40.

Table 40. Field and glass house residue trials with propamocarb hydrochloride conducted on tomato.

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha	Water L/ha	No.	PHI (Days)	Residues as Propamocarb mg/kg	Residues Propamocarb HCl, mg/kg	Report no. F/GH
Belgium 2004 *Clotilde*	840 SL 530 g ai/L	Drench irrig. Drip irrig.	15.9 1.59	20000 250[2]	2 4	0 1 3 7 14	0.06* 0.05* 0.07* 0.04* 0.02*	0.07 0.06 0.08 0.05 0.02	RA 2506/04 GH
Germany 2004 *Culina*	840 SL 530 g ai/L	Drench irrig. Drip irrig.	15.9 1.59	20000 100[2]	2 4	0 1 3 7 14	< 0.008* < 0.008* < 0.008* < 0.008* < 0.008*	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA 2506/04 GH
Italy, 2004 *Conchita*	840 SL 530 g ai/L	Drench irrig. Drip irrig.	15.9 1.59	20000 100[2]	2 4	1 3	< 0.008* < 0.008*	< 0.01 < 0.01	RA 2506/04 GH
Spain, 2004 *Pitenza*	840 SL 530 g ai/L	Drench irrig. Drip irrig.	15.9 0.60-1.59	20000 100	2 5	1 3	0.02* 0.02*	0.02 0.02	RA 2506/04 GH
Germany, 2001, *Rougella RZ F1*	840 SL 530 g ai/L	Seedbed drench Drench/Drip	15.92 1.89-2.02	20026 1763-1914	2 4	1 3	0.02 < 0.008	0.02* < 0.01*	C021852 GH
Netherlands 2001 *Fergie*	840 SL 530 g ai/L	Seedbed drench Drench/Drip	15.78 2.65	19860 2500	2 4	1 3	0.05 0.04	0.06* 0.05*	C021852 GH
Netherlands 2001, *Rapsodie*	840 SL 530 g ai/L	Seedbed drench Drench/Drip	15.90 2.66	20000 2500	2 4	1 3	0.03 0.03	0.03* 0.04*	C021852 GH
Spain, 2001 *Salvador*	840 SL 530 g ai/L	Seedbed drench Drench/Drip	15.96 1.99	20074 2300	2 4	1 3	0.04 0.08	0.05* 0.10*	C021852
France, 2001 *Cobra*	722 SL 722 g ai/L	Seedbed drench Drench	72.2-36.0 3.77-3.62	20000 2607-2328	2 2	0 3 5 7	< 0.008 < 0.008 < 0.008 < 0.008	< 0.01* < 0.01* < 0.01* < 0.01*	C023899 F

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha	Water L/ha	No.	PHI (Days)	Residues as Propamocarb mg/kg	Residues Propamocarb HCl, mg/kg	Report no. F/GH
Greece, 2001 *ACE*	722 SL 722 g ai/L	Seedbed drench Drench	72.2-36.0 3.6	20000 2500	2 2	0 3 5 7	< 0.008 < 0.008 < 0.008 < 0.008	< 0.01* < 0.01* < 0.01* < 0.01*	C023899 F
Italy, 2001 *Italdor*	722 SL 722 g ai/L	Seedbed drench Drench	72.2-36.0 2.41	20000 1667	2 2	0 3 5 7	< 0.008 < 0.008 < 0.008 < 0.008	< 0.01* < 0.01* < 0.01* < 0.01*	C023899 F
Spain, 2001 *Robin*	722 SL 722 g ai/L	Seedbed drench Drench	72.2-36.8 2.9	20000 25000	2 2	0 3 5 7	< 0.008 0.02 0.008 < 0.008	< 0.01* 0.02* 0.01* < 0.01*	C023899 F
Spain 2001 *Robin*	722 SL 722 g ai/L	Seedbed drench Drench	72.2-36.8 2.87	20000 29800	2 2	0 3 5 7	< 0.008 < 0.008 < 0.008 < 0.008	< 0.01* < 0.01* < 0.01* < 0.01*	C023899 F
Germany 2000 *Jamaica*	722 SL 722 g ai/L	Seedbed drench Drench	29.0 2.74-2.77	10000 0 1897-1917	1 4	0 3 5 7	< 0.01* < 0.01* < 0.01* 0.01*	< 0.01* < 0.01* < 0.01* 0.01*	C015427 GH
Germany 2000 *Rougella*	722 SL 722 g ai/L	Seedbed drench Drench	28.5 2.6-2.77	98530 1621-1905	1 4	0 3 5 7	0.05* 0.05* 0.09* 0.04*	0.06* 0.06* 0.1* 0.05*	C015427 GH
Germany 2000 *Rougella*	722 SL 722 g ai/L	Seedbed drench Drench	28.4 2.2	98426 1841	1 4	0 3 5 7	< 0.01* < 0.01* < 0.01* 0.02*	< 0.01* < 0.01* < 0.01* 0.02*	C015427 GH
Germany 2000 *Rabor*	722 SL 722 g ai/L	Seedbed drench Drench	29.0 3.6	39062 5 2500	1 4	0 3 5 7	< 0.01* < 0.01* < 0.01* < 0.01*	< 0.01* < 0.01* < 0.01* < 0.01*	C015427 GH
Germany 2000 *Transfero*	722 SL 722 g ai/L	Seedbed drench Drench	29.2 3.6	39523 2 2500	1 4	0 3 5 7	< 0.01* < 0.01* < 0.01* < 0.01*	< 0.01* < 0.01* < 0.01* < 0.01*	C015427 GH
Germany 2000 *Rougella*	722 SL 722 g ai/L	Seedbed drench Drench	29.0 4.6	15703 1 3185	1 4	0 3 5 7	< 0.01* < 0.01* < 0.01* < 0.01*	< 0.01* < 0.01* < 0.01* < 0.01*	C015427 GH
Germany 2000 *Halifax*	722 SL 722 g ai/L	Seedbed drench Drench	29.0 4.6	15429 7 3185	1 4	0 3 5 7	< 0.01* < 0.01* < 0.01* < 0.01*	< 0.01* < 0.01* < 0.01* < 0.01*	C015427 GH
Spain, 2000 *Daniela*	722 SL 722 g ai/L	Seedbed drench Drench	29.0 3.3	20000 2301-4603	1 4	0 3 5 7	< 0.01* < 0.01* < 0.01* < 0.01*	< 0.01* < 0.01* < 0.01* < 0.01*	C015427 GH
Spain, 2000 *Daniela*	722 SL 722 g ai/L	Seedbed drench Drench	29.0 3.3	20000 2286-4571	1 4	0 3 5 7	< 0.01* < 0.01* < 0.01* < 0.01*	< 0.01* < 0.01* < 0.01* < 0.01*	C015427 GH

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha	Water L/ha	No.	PHI (Days)	Residues as Propamocarb mg/kg	Residues Propamocarb HCl, mg/kg	Report no. F/GH
France, 2000 *Frya*	722 SL 722 g ai/L	Drench Drench/Drip Irrigation	72.2-36.0 2.0	20000 1877	2 2	0 1 3 7 14	< 0.01* < 0.01* < 0.01* < 0.01* < 0.01*	< 0.01* < 0.01* < 0.01* < 0.01* < 0.01*	C015573 GH
Greece, 2000 *Garnell 534 Emben*	722 SL 722 g ai/L	Drench Drench/Drip Irrigation	72.2-36.0 1.8	20000 1664	1 2	0 1 3 7 14	< 0.01* < 0.01* < 0.01* < 0.01* < 0.01*	< 0.01* < 0.01* < 0.01* < 0.01* < 0.01*	C015573 GH
Italy, 2000 *Vivaldi HY*	722 SL 722 g ai/L	Drench Drench/Drip Irrigation	72.2-36.0 4.3	20118 4000	1 2	0 1 3 7 14	< 0.01* < 0.01* < 0.01* < 0.01* < 0.01*	< 0.01* < 0.01* < 0.01* < 0.01* < 0.01*	C015573 GH
Spain, 2000 *James Bond*	722 SL 722 g ai/L	Drench Drench/ Drip Irrigation	72.2-36.0 2.6-2.7	20000 2389-2535	1 2	0 1 3 7 14	< 0.01* < 0.01* < 0.01* < 0.01* < 0.01*	< 0.01* < 0.01* < 0.01* < 0.01* < 0.01*	C015573 GH
Spain, 2000 *Raff*	722 SL 722 g ai/L	Drench Drench/ Drip Irrigation	73.6-36.0 2.0-2.1	20384 1844-1984	1 2	0 1 3 7 14	< 0.01* < 0.01* < 0.01* < 0.01* < 0.01*	< 0.01* < 0.01* < 0.01* < 0.01* < 0.01*	C015573 GH
USA, 1996 *Celebrity*	750 SC 375 g ai/L	Spray	1.32	210	5	5	0.16*	0.19	C002417 F
USA, 1996 *8892*	750 SC 375 g ai/L	Spray	1.32	195	5	5	0.25*	0.30	C002417 F
USA, 1996 *UC-82B*	750 SC 375 g ai/L	Spray	1.32	190	5	5	0.86*	1.0	C002417 F
USA, 1996 *Jackpot*	750 SC 375 g ai/L	Spray	1.32	192	5	5	0.94*	1.1	C002417 F
USA, 1996 *3155*	750 SC 375 g ai/L	Spray	1.32	188	5	5	0.65*	0.78	C002417 F
USA, 1996 *512*	750 SC 375 g ai/L	Spray	1.32	187	5	5	0.68*	0.81	C002417 F
USA, 1996 *Rio Grande*	750 SC 375 g ai/L	Spray	1.32	193	5	5	0.60*	0.72	C002417 F
USA, 1996 *6229*	750 SC 375 g ai/L	Spray	1.32	187	5	5	0.61*	0.73	C002417 F
USA, 1996 *Rio Grande*	750 SC 375 g ai/L	Spray	1.32	187	5	5	0.23*	0.27	C002417 F
USA, 1996 *Rio Grande*	750 SC 375 g ai/L	Spray	1.32	195	5	5	0.51*	0.61	C002417 F
USA, 1996 *Celebrity*	750 SC 375 g ai/L	Spray	1.32	224	5	5	0.14*	0.17	C002417 F
USA, 1996 *AgriSet*	750 SC 375 g ai/L	Spray	1.32	192	5	5	0.40*	0.48	C002417 F

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha	Water L/ha	No.	PHI (Days)	Residues as Propamocarb mg/kg	Residues Propamocarb HCl, mg/kg	Report no. F/GH
USA, 1996 *AgriSet*	750 SC 375 g ai/L	Spray	1.32	184	5	5	0.61*	0.73	C002417 F
USA, 1996 *Heinz 9035*	750 SC 375 g ai/L	Spray	1.32	190	5	5	0.34*	0.41	C002417 F
USA, 1996 *CAL-ACE*	750 SC 375 g ai/L	Spray	1.32	195	5	5	0.37*	0.44	C002417 F
USA, 1996 *Apex 1000*	750 SC 375 g ai/L	Spray	1.32	190	5	5	1.4*	1.6	C002417 F
USA, 1996 *Better Boy*	750 SC 375 g ai/L	Spray	1.32	190	5	1 3 5 7 9	0.52* 0.47* 0.38* 1.1* 0.46*	0.62 0.56 0.45 1.3 0.55	C002417 F
USA, 1996 *Shady Lady*	750 SC 375 g ai/L	Spray	1.32	193	5	1 3 5 7 9	0.97* 0.62* 0.52* 0.35* 0.35*	1.2 0.74 0.44 0.42 0.42	C002417 F

*actual value reported

Lettuce

Sixty eight greenhouse (GH) and field (F) trials were with propamocarb hydrochloride were reported for lettuce. The propamocarb was applied either as a drench and/or foliar spray in Europe and USA between 1997 and 2003. The results are shown in Table 41.

Table 41. Results of residue trials with propamocarb hydrochloride conducted in lettuce.

Country, Year, *Variety*	Application Form.	Method	kg ai/ha	Water L/ha	No.	PHI (Days)	Sample analysed	Residues as Propamocarb mg/kg	Residues as Propamocarb HCL, mg/kg	Report F/GH
France, 1993 *Batavia*	722 SL 722g ai/L	Spray	1.08	1000	4	21 40	Head	20 10	24* 12*	A85676 GH
France, 1993 *Batavia*	722 SL 722g ai/L	Spray	1.44	1000	4	21 40	Head	39 11	47* 13*	A85676 GH
France. 1993 *Batavia*	722 SL 722g ai/L	Spray	1.44	1000	4	21 28	Head	40 23	48* 28*	A85676 GH
France, 1993 *Ramona*	722 SL 722g ai/L	Spray	1.08	1000	4	21 28	Head	14 9.2	17* 11*	A85676 GH
France, 1993 *Ramona*	722 SL 722g ai/L	Spray	1.44	1000	4	21 28	Head	15 18	18* 14*	A85676 GH
France, 1993 *Ramona*	722 SL 722g ai/L	Spray	1.44	1000	4	20	Head	24	29*	A85676 GH
France,. 1994 *Samourai*	722 SL 722g ai/L	Spray	1.1	500	3	0 21	Head	24 4.9	29* 5.9*	A83358 GH
France, 1994 *Canasta*	722 SL 722g ai/L	Spray	1.1	1000	3	0 20	Head	22 1.2	26* 1.4*	A85675 GH
France, 1994 *Canasta*	722 SL 722g ai/L	Spray	1.3	1000	3	0 20	Head	24. 1.7	29* 2.0*	A85675 GH
France, 1994 *Rosalba*	722 SL 722g ai/L	Spray	1.1	-	3	0 21	Head	18 6.5	21* 7.8*	A85679 GH

Country, Year, *Variety*	Application					PHI (Days)	Sample analysed	Residues as Propamocarb mg/kg	Residues as Propamocarb HCL, mg/kg	Report F/GH
	Form.	Method	kg ai/ha	Water L/ha	No.					
France	840 SL	Seedbed	15.9	20000	2	0	Head	17	20*	C01573
2000	530 g ai/L	drench				14	Head	7.1	8.5*	4
Macarena		Spray	1.25-1.43	470-540	2	21	Head	0.56	0.90*	GH
						28	Head	0.08	0.1*	
France	SL	Seedbed	72.2-36.1	20000	2	0	Head	54	65*	C02295
2001	722g ai/L	drench				7	Head	12*	15*	1
Nadine						14	Head	3.2*	3.8*	F
		Spray		400-1000	2	21	Head	0.3*	0.4*	
			1.66			14	Head [1]	1.7*	2.0*	
						14	Outer Leaves	11*	13*	
France	SL	Seedbed	72.2-36.1	20000	2	0	Head	103*	123*	C02295
2001	722g ai/L	drench				7	Head	7.9*	9.5*	1
Macarena						14	Head	1.0*	1.2*	
		Spray		400-1000	2	21	Head	0.1*	0.2*	F
			1.66			14	Head [1]	0.1*	0.2*	
						14	Outer Leaves	3.2*	3.8*	
France	SL	Seedbed	72.2-36.1	20000	2	0	Head	11	13*	C02415
2001	722g ai/L	drench				13	Head	8.1	9.7*	7
Sensai		Spray		400-1000	2	21	Head	2.9	3.5*	
			1.66							
France, 2003	840 SL	Seedbed	15.90	20000	2	0	Head	31*	43*	RA
Sensai	530g ai/L	drench				3	Head	16*	19*	2712/03
		Spray	1.33	400	2	7	Head	16*	19*	GH
						14	Head	13*	15*	
						21	Head	8.2*	9.8*	
France, 2002	722 SL	Seedbed	72.2-36.1	20000	2	0	Head	69*	83*	C03377
Laitue Batavia	722g ai/L	drench				21	Head	0.17*	0.2*	F
Eole		Spray		400	2					
			1.66							
France	722 SL	Seedbed	72.2-36.1	20000	2	0	Head	81*	97*	C03371
2002	722g ai/L	drench				21	Head	2.2*	2.7*	7
Autan		Spray		400	2					F
			1.66							
France	722 SL	Seedbed	72.2-36.1	20000	2	0	Head	25	30*	C01557
2000	722g ai/L	drench				14	Head	14	17*	2
Mistral						21	Head	11	13*	F
		Spray		1000	2	28	Head	10	12*	
			1.66							
France	722 SL	Seedbed	72.2-36.1	20000	2	0	Head	39	46*	C01557
2000	722g ai/L	drench				14	Head	31	37*	2
Mistral						20	Head	31	37*	F
		Spray		1000	2	27	Head	23	28*	
			1.66							
France	722 SL	Seedbed	72.2-36.1	20000	2	0	Head	71	85*	C01542
2000	722g ai/L	drench				14	Head	40	48*	3
Flandra RZ						21	Head	32	31*	GH
		Spray		472-830	2	28	Head	14	17*	
			1.66			35	Head	13	16*	
France	722 SL	Seedbed	72.2-36.1	20000	2	0	Head	25	30*	C01542
2000	722g ai/L	drench				14	Head	7.9	9.4*	3
RZ 42-77						21	Head	1.5	1.8*	GH
		Spray		475-838	2	28	Head	0.08	0.1*	
			1.66			35	Head	< 0.08	< 0.1*	

Country, Year, *Variety*	Application					PHI (Days)	Sample analysed	Residues as Propamocarb mg/kg	Residues as Propamocarb HCL, mg/kg	Report F/GH
	Form.	Method	kg ai/ha	Water L/ha	No.					
France	722 SL	Seedbed	72.2-	20000	2	0	Head	22	26*	C01542
2001	722g ai/L	drench	36.1			14	Head	9.2	11*	6
Macarena		Spray		480-520	2	21	Head	0.7	0.8*	GH
			1.66			28	Head	0.1	0.14*	
France	722 SL	Seedbed	72.2-	20000	2	0	Head	5.1	6.1*	C01542
2002	722g ai/L	drench	36.1			14	Head	2.0	2.4*	6
Macarena		Spray		470-520	2	21	Head	1.7	2.0*	GH
			1.66			28	Head	< 0.008	< 0.01*	
Germany	840 SL	Seedbed	15.9	20000	2	0	Head	26	31*	C01573
2000	530 g ai/L	drench				14	Head	4.5	5.4*	4
Macarena		Spray	1.3-1.4	500-820	2	21	Head	1.7	2.0*	GH
						28	Head	0.03	0.04*	
Germany	840 SL	Seedbed	15.9	20000	2	0	Head	25	30*	C01573
2000	530 g ai/L	drench				14	Head	8.1	9.6*	4
Flandria		Spray	1.3-1.4	400-630	2	21	Head	1.1	1.3*	GH
						28	Head	0.05	0.06*	
Germany	SL	Seedbed	72.2-	20000	2	0	Head	44*	53*	C02295
2001	722g ai/L	drench	36.1			7	Head	6.9*	8.3*	1
Comina						14	Head	1.9*	2.3*	F
		Spray		400-	2	21	Head	0.3*	0.4*	
			1.66	1000		14	Head [1]	0.6*	0.7*	
						14	Outer Leaves	14*	16*	
Germany	SL	Seedbed	72.2-	20000	2	0	Head	60*	72*	C02295
2001	722g ai/L	drench	36.1			7	Head	9.3*	11*	1
Einstein						14	Head	4.2*	5.0*	
		Spray		400-	2	21	Head	2.5*	3.0*	F
			1.66	1000		14	Head [1]	0.3*	0.3*	
						14	Outer Leaves	7.5*	9.0*	
Germany	SL	Seedbed	72.2-	20000	2	0	Head	62*	74*	C02295
2001	722g ai/L	drench	36.1			7	Head	4.6*	5.5*	1
Nadine						14	Head	0.7*	0.8*	
		Spray		400-	2	21	Head	0.04*	0.05*	F
			1.66	1000		14	Head [1]	0.1*	0.2*	
						14	Outer Leaves	4.5*	5.3*	
Germany	722 SL	Seedbed	72.2-	20000	2	0	Head	37	44*	C01542
2000	722g ai/L	drench	36.1			14	Head	16	19*	3
Trobadur RZ						21	Head	10	12*	GH
Greenhouse		Spray		380-630	2	28	Head	9.2	11*	
			1.66			34	Head	5.5	6.6*	
Germany	722 SL	Seedbed	72.2-	20000	2	0	Head	50	60*	C01542
2000	722g ai/L	drench	36.1			14	Head	20	24*	3
Tzigone RZ						21	Head	15	18*	GH
Greenhouse		Spray		400-640	2	28	Head	21	25*	
			1.66			34	Head	13	16*	
Germany	722 SL	Seedbed	72.2-	20000	2	0	Head	49	59*	
2000	722g ai/L	drench	36.1			14	Head	29	34*	C01542
RZ 42-77						21	Head	12	14*	3
Greenhouse		Spray		476-838	2	28	Head	18	21*	GH
			1.66			35	Head	6.6	7.9*	
Germany	722 SL	Seedbed	72.2-	20000	2	0	Head	11	13*	C01542
2000	722g ai/L	drench	36.1			14	Head	17	20*	3
RZ 42-77						21	Head	9.2	11*	GH
Greenhouse		Spray		495-819	2	28	Head	4.3	5.1*	
			1.66			35	Head	7.7	9.2*	

Country, Year, *Variety*	Application					PHI (Days)	Sample analysed	Residues as Propamocarb mg/kg	Residues as Propamocarb HCL, mg/kg	Report F/GH
	Form.	Method	kg ai/ha	Water L/ha	No.					
Germany	722 SL	Seedbed	72.2-	20000	2	0	Head	82	98*	C03371
2002	722g ai/L	drench	36.1			21	Head	0.64	0.76*	7
Nadine		Spray		600	2					F
			1.66							
Germany	722 SL	Seedbed	72.2-	20000	2	0	Head	105	125*	C03371
2002	722g ai/L	drench	36.1			21	Head	0.57	0.68*	7
Nadine		Spray		400	2					F
			1.66							
Germany	840 SL	Seedbed	15.9	18750	2	0	Head	37*	44*	RA
2003	530g ai/L	drench				3	Head	14*	17*	2712/03
Alexandria		Spray	1.33	400	2	7	Head	11*	13*	GH
						14	Head	3.9*	4.7*	
						21	Head	0.64*	0.76*	
Germany	840 SL	Seedbed	15.9	18750	2	0	Head	31*	37*	RA
2003	530g ai/L	drench				3	Head	16*	19*	2712/03
Alexandria		Spray	1.33	400	2	7	Head	7.4*	8.8*	GH
						14	Head	4.0*	4.8*	
						21	Head	0.71*	0.85*	
Germany	722 SL	Seedbed	72.2-	20000	2	0	Head	20	24*	C01542
2000	722g ai/L	drench	36.1			14	Head	13	15*	6
Elton		Spray		399-	2	21	Head	1.8	2.2*	GH
			1.66	1019		28	Head	< 0.008	< 0.01*	
Germany	722 SL	Seedbed	72.2-	20000	2	0	Head	12	14*	C01542
2000	722g ai/L	drench	36.1			14	Head	7.4	8.8*	6
Flandria		Spray		562-638	2	21	Head	0.4	0.5*	GH
			1.66			28	Head	0.008	0.01*	
Germany	722 SL	Seedbed	72.2-	20000	2	0	Head	31	37*	C01542
2000	722g ai/L	drench	36.1			14	Head	6.5	7.7*	6
Macarena		Spray		480-840	2	21	Head	1.8	2.2*	GH
			1.66			28	Head	1.8	0.05*	
Greece	SL	Seedbed	72.2-	20000	2	0	Head	76	91*	C02415
2001	722g ai/L	drench	36.1			14	Head	13	15*	7
Estivena		Spray		400-	2	21	Head	12	14*	
			1.66	1000						F
Greece	722 SL	Seedbed	72.2-	20000	2	0	Head	28	33*	C01557
2000	722g ai/L	drench	36.1			7	Head	9.2	11*	2
Romana		Spray		581-	2	14	Head	6.0	7.2*	F
			1.66	1018		21	Head	3.3	3.9*	
Greece	840 SL	Seedbed	14.1	18000	2	0	Head	19*	23	C01557
2000	530 g ai/L	drench				14	Head	11*	13	7
Romana		Spray	1.3	600-970	2	21	Head	2.2*	2.6	F
						28	Head	0.84*	1.0	
Italy	840 SL	Seedbed	16.7-	20000	2	0	Head	43*	51	C01557
2000	530 g ai/L	drench	17.2			14	Head	1.8*	2.2	7
Titan		Spray		500-700	2	21	Head	0.07*	0.08	
			1.3			28	Head	< 0.008*	< 0.01	F
Italy, 2000	722 SL	Seedbed	72.2	20000	2	0	Head	35	42*	C01557
Titan	722g ai/L	drench	36.1			14	Head	1.0	1.2*	2
		Spray	1.66	493-727	2	21	Head	0.04	0.05*	F
						28	Head	< 0.008	< 0.01*	

Country, Year, *Variety*	Application: Form.	Method	kg ai/ha	Water L/ha	No.	PHI (Days)	Sample analysed	Residues as Propamocarb mg/kg	Residues as Propamocarb HCL, mg/kg	Report F/GH
Italy	840 SL	Seedbed	15.9	20000	2	0	Head	65*	78*	RA
2003	530g ai/L	drench				3	Head	27*	32*	2712/03
Settelune						7	Head	14*	17*	GH
		Spray	1.33	400	2	14	Head	0.92*	1.1*	
						21	Head	0.08*	0.1*	
Japan, 1991	SL	?	1.28	1000	3	7	leaf	22	26*	(2)
Gokuwase	640 g/kg					14		1.8	2.1*	F
shisuko						21		0.10	0.12*	
						28		0.15	0.18*	
Japan, 1991	SL	?	1.28	1000	3	7	leaf	14	17*	(2)
Shinanogreen	640 g/kg					14		0.28	0.33*	F
						21		0.08	0.10*	
						28		0.04	0.05*	
Japan, 1991	SL	?	1.28	1000	3	7	leaf	20	24*	(2)
Gokuwase	640 g/kg					14		1.6	1.9*	F
shisuko						21		0.10	0.12*	
						28		0.09	0.11*	
Japan, 1991	SL	?	1.28	1000	3	7	leaf	16	19*	(2)
Shinanogreen	640 g/kg					14		0.60	0.68*	F
						21		0.11	0.13*	
						28		0.06	0.07*	
Netherland	840 SL	Seedbed	15.9	20000	2	0	Head	58*	69*	RA
2003	530g ai/L	drench				3	Head	32*	38*	2712/03
Alexandria		Spray	1.33	400	2	7	Head	25*	30*	GH
						14	Head	9.8*	12*	
						21	Head	4.2*	5.0*	
Netherland	840 SL	Seedbed	15.9	20000	2	0	Head	53*	63*	RA
2003	530g ai/L	drench				3	Head	30*	36*	2712/03
Alexandria		Spray	1.33	400	2	7	Head	21*	25*	GH
						14	Head	9.4*	11*	
						21	Head	4.0*	4.8*	
Spain, 2000	722 SL	Seedbed	72.2	20000	2	0	Head	27	32*	C01557
Inverna	722g ai/L	drench	36.1			14	Head	4.7	5.6*	2
		Spray	1.66	670-988	2	21	Head	0.08	0.1*	F
						28	Head	< 0.008	< 0.01*	
Spain, 2000	722 SL	Seedbed	72.2	20000	1	0	Head	10	12*	C01542
Cabezo	722g ai/L	drench	36.1	20000	1	14	Head	3.4	4.1*	3
Greenhouse		Spray	1.66	401-941	1	21	Head	3.3	4.0*	GH
					1	28	Head	1.0	1.2*	
						35	Head	4.4	5.3*	
Spain, 2000	722 SL	Seedbed	72.2-	20000	2	0	Head	29	35*	C01542
Cabezo	722g ai/L	drench	36.1			14	Head	15	18*	3
Greenhouse		Spray		404-	2	21	Head	15	18*	GH
			1.66	1004		28	Head	6.9	8.2*	
						35	Head	2.2	2.6*	
Spain, 2000	722 SL	Seedbed	72.2-	20000	2	0	Head	28	33*	C01542
Cabezo	722g ai/L	drench	36.1			14	Head	14	17*	3
Greenhouse		Spray		384-980	2	21	Head	16	19*	GH
			1.66			28	Head	10	12*	
						35	Head	5.7	6.8*	
Spain	SL	Seedbed	72.2-	20000	2	0	Head	87	104*	C02415
2001	722g ai/L	drench	36.1			13	Head	3.3	4.0*	7
Estivena		Spray		400-	2	21	Head	0.34	0.41*	
			1.66	1000						F
Spain, 2001	SL	Seedbed	72.2-	20000	2	0	Head	86	103*	C02415
Estivena	722g ai/L	drench	36.1			13	Head	2.8	3.4*	7
		Spray		400-	2	20	Head	0.56	0.67*	
			1.66	1000						F

Country, Year, *Variety*	Application					PHI (Days)	Sample analysed	Residues as Propamocarb mg/kg	Residues as Propamocarb HCL, mg/kg	Report F/GH
	Form.	Method	kg ai/ha	Water L/ha	No.					
Spain, 2000 *Inverna*	840 SL 530 g ai/L	Seedbed drench Spray	15.8 1.3-1.76	20000 700-1000	2 2	0 14 21 28	Head Head Head Head	24* <u>10</u>* 0.50* 0.03*	29 12 0.60 0.04	C01557 7 F
USA, 1997 *Gene Corp* *Green*	724.5 SL 724.5 g ai/L	Spray	1.6	154-189	4	2	Leaves	<u>41</u>*	49	B002740 F
USA, 1997 *Darkland* *Romaine*	724.5 SL 724.5 g ai/L	Spray	1.6	185-189	4	2	Leaves	<u>31</u>*	36	B002740 F
USA, 1997 *Presidio*	724.5 SL 724.5 g ai/L	Spray	1.6	176-195	4	2	Leaves	<u>10</u>*	12	B002740 F
USA, 1997 *Rapids Waldman*	724.5 SL 724.5 g ai/L	Spray	1.6	187-193	4	1 2 4 6 8	Leaves Leaves Leaves Leaves Leaves	88* <u>60</u>* 60* 45* 50*	105 71.3 72.1 53.2 59.3	B002740 F
USA, 1997 *Black Seeded* *Simpson*	724.5 SL 724.5 g ai/L	Spray	1.6	182-187	4	2	Leaves	<u>51</u>*	61	B002740 F
USA, 1997 *Romaine*	724.5 SL 724.5 g ai/L	Spray	16	171-190	4	2	Leaves	<u>17</u>*	20	B002740 F
USA, 1997 *Blacks Simpson*	724.5 SL 724.5 g ai/L	Spray	1.6	187	4	2	Leaves	<u>86</u>*	102	B002740 F
USA, 1997 *Crispino*	SL 724.5 g ai/L	Spray	1.6	184-191	4	2	Head Head[1]	<u>48</u>* 8.0*	58 9.6	B002740 F
USA, 1997 *Iceberg*	SL 724.5 g ai/L	Spray	1.6	171-190	4	2	Head Head[1]	<u>8.2</u>* 0.21*	9.8 0.3	B002740 F
USA, 1997 *Ithaca*	SL 724.5 g ai/L	Spray	1.6	191	4	1	Head Head[1]	<u>11</u>* 0.31*	13 0.4	B002740 F
USA, 1997 *Magnum*	SL 724.5 g ai/L	Spray	1.6	154-189	4	2	Head Head[1]	<u>11</u>* 0.23*	13 0.3	B002740 F
USA, 1997 *Lagacy*	SL 724.5 g ai/L	Spray	1.6	183-194	4	2	Head Head[1]	<u>19</u>* 0.34*	22 0.4	B002740 F
USA, 1997 *Top Gun*	SL 724.5 g ai/L	Spray	1.6	184-191	4	2	Head Head[1]	<u>9.7</u>* 1.5*	12 1.8	B002740 F
USA 1997	SL 724.5 g ai/L	Spray	1.6	187-193	4	1 2 4 6 8 1 2 4 6 8	Head Head[1]	38* <u>41</u>* 34* 27* 20* 2.1* 2.1* 0.99* 0.64* 0.52*	46 49 41 32 24 2.5 2.5 1.2 0.8 0.6	B002740 F

1 Head without wrapper leaves;

2. only a summary of the trial was provided;

*actual value reported

Spinach

Seven field trials were conducted with propamocarb hydrochloride on spinach were reported from Belgium, Germany, Italy and Spain using foliar application. The results are shown in Table 42.

Table 42. Residues from field trials with Propamocarb hydrochloride conducted in spinach.

Country, Year of trial *Variety*	Application					PHI (Days)	Residues as Propamocarb mg/kg	Report
	Form.	Method	kg ai/ha	Water L/ha	No.			
Belgium 2004, *Mig*	840 SL	Spray	1.325	300	3	0 14	83* 1.6*	RA 2558/04
Germany 2004, *Matador*	840 SL	Spray	1.325	300	3	0 14	73* 18*	RA 2558/04
Germany 2003 *Fentos*	840 SL 530 g ai/L	Spray	1.325	300	3	0 3 7 14 21	79* 22* 13* 2.9* 0.41*	RA-2619/03
Germany 2003 *Matador*	840 SL 530 g ai/L	Spray	1.325	300	3	0 3 7 14	49* 27* 18* 10*	RA-2619/03
Italy, 2004 *Riccio D'america*	840 SL 530 g ai/L	Spray	1.59	500	2	0 3 7 14 21	100* 53* 42* 16* 14*	RA 2557/04
Italy, 2004 *Riccio D'america*	840 SL 530 g ai/L	Spray	1.59	450	2	0 3 7 14 21	54* 52* 37* 8.3* 8.4*	RA 2557/04
Spain 2004 *Dolfin*	840 SL 530 g ai/L	Spray	1.59	400	2	0 3 7 14 21	99* 58* 46* 45* 29*	RA 2557/04

*actual value reported

Potato

Thirty two field trials on potatoes were reported with propamocarb HCl, conducted between 1990 and 2003, using foliar application in Europe (13) and USA (19). The results are shown in Table 43.

Table 43. Residues from field trials with propamocarb hydrochloride conducted in potato, foliar spray.

Country, Year of trial *Variety*	Form.	Application			PHI (Days)	Sample analysed	Residues, as Propamocarb mg/kg	Residues, as propamocarb HCl, mg/kg	Report
		kg ai/ha	Water L/ha	No.					
France 2003, *Spunta*	450 SC 386 g ai/L	0.75	400	6	0 7	Tuber Tuber	< 0.01* < 0.01*	< 0.012 < 0.012	C042791
Germany 1990 *Akula*	549.6 SC 248 g ai/L	0.99	400	6	0 2 5 7	Tuber Tuber Tuber Tuber	< 0.08 < 0.08 < 0.08 < 0.08	< 0.1* < 0.1* < 0.1* < 0.1*	A85312
Germany 1990 *Bintje*	549.6 SC 248 g ai/L	0.99	400	6	0 3 5 7	Tuber Tuber Tuber Tuber	0.25 < 0.08 < 0.08 < 0.08	0.3* < 0.1* < 0.1* < 0.1*	A85312

Country, Year of trial *Variety*	Form.	Application kg ai/ha	Application Water L/ha	Application No.	PHI (Days)	Sample analysed	Residues, as Propamocarb mg/kg	Residues, as propamocarb HCl, mg/kg	Report
Germany 1990 *Hansa*	549.6 SC 248 g ai/L	0.99	400	6	0	Tuber	0.25	0.3*	A85312
					3	Tuber	0.08	0.1*	
					5	Tuber	0.17	0.2*	
					7	Tuber	0.17	0.2*	
Germany 1990 *Hansa*	549.6 SC 248 g ai/L	0.99	400	6	0	Tuber	0.17	0.2*	A85312
					3	Tuber	0.08	0.1*	
					5	Tuber	0.17	0.2*	
					7	Tuber	0.17	0.2*	
Germany 1991 *Celena*	549.6 SC 248 g ai/L	0.99	400	6	0	Tuber	< 0.08	< 0.1*	A85332
					3	Tuber	0.08	0.1*	
					5	Tuber	< 0.08	< 0.1*	
					7	Tuber	< 0.08	< 0.1*	
					0	Tuber, washed	< 0.08	< 0.1*	
					7	Tuber, washed	< 0.08	< 0.1*	
					0	Tuber, peeled	< 0.08	< 0.1*	
					7	Tuber, peeled	≤ 0.08	< 0.1*	
					0	Peel, washed	< 0.08	< 0.1*	
					7	Peel, washed	< 0.08	< 0.1*	
Germany 1991 *Roxy*	549.6 SC 248 g ai/L	0.99	400	6	0	Tuber	< 0.08	< 0.1*	A85332
					3	Tuber	0.08	0.1*	
					5	Tuber	< 0.08	< 0.1*	
					7	Tuber	< 0.08	< 0.1*	
					0	Tuber, washed	< 0.08	< 0.1*	
					7	Tuber, washed	< 0.08	< 0.1*	
					0	Tuber, peeled	< 0.08	< 0.1*	
					7	Tuber, peeled	≤ 0.08	< 0.1*	
					0	Peel, washed	0.08	0.1*	
					7	Peel, washed	< 0.08	< 0.1*	
Germany 1991 *Grandifolia*	549.6 SC 248 g ai/L	0.99	400	6	0	Tuber	0.08	0.1*	A85332
					3	Tuber	< 0.08	< 0.1*	
					5	Tuber	< 0.08	< 0.1*	
					7	Tuber	< 0.08	< 0.1*	
Germany 1992 *Grandifolia*	549.6 SC 248 g ai/L	1.2	400	6	0	Tuber	< 0.08	< 0.1*	A85349
					3	Tuber	< 0.08	< 0.1*	
					5	Tuber	< 0.08	< 0.1*	
					7	Tuber	< 0.08	< 0.1*	
Germany 1992 *Sommergold*	549.6 SC 248 g ai/L	0.995	400	6	0	Tuber	< 0.08	< 0.1*	A85349
					3	Tuber	< 0.08	< 0.1*	
					5	Tuber	< 0.08	< 0.1*	
					7	Tuber	< 0.08	< 0.1*	
Germany 1992 *Anosta*	549.6 SC 248 g ai/L	1.2 – 1.33	400	6	0	Tuber	< 0.08	< 0.1*	A85349
					3	Tuber	< 0.08	< 0.1*	
					5	Tuber	< 0.08	< 0.1*	
					7	Tuber	< 0.08	< 0.1*	
Germany 2003, *Cilena*	450 SC 375 g ai/L	0.75	600	6	0	Tuber	< 0.01*	< 0.012	C042791
					7	Tuber	< 0.01*	< 0.012	
UK, 2003 *Spey*	450 SC 375 g ai/L	0.75	400	6	0	Tuber	< 0.01*	< 0.012	C042791
					7	Tuber	< 0.01*	< 0.012	
USA, 1996 *Superior*	750 SC 375 g ai/L	1.9	187	5	15	Tuber	< 0.05*	< 0.06	A91233
USA, 1996 *Chippewa*	750 SC 375 g ai/L	1.0	187	5	14	Tuber	< 0.05*	< 0.06	A91233
USA, 1996 *Superior*	750 SC 375 g ai/L	1.0	187	5	14	Tuber	< 0.05*	< 0.06	A91233
USA, 1996 *WF31-4*	750 SC 375 g ai/L	1.0	187	5	14	Tuber	0.05*	0.06	
USA, 1996 *Red Pontiac*	750 SC 375 g ai/L	1.0	187	5	14	Tuber	0.05*	0.05	A91233
USA, 1996 *Superior*	750 SC 375 g ai/L	1.0	187	5	14	Tuber	< 0.05*	< 0.06	A91233

Country, Year of trial *Variety*	Form.	Application kg ai/ha	Application Water L/ha	Application No.	PHI (Days)	Sample analysed	Residues, as Propamocarb mg/kg	Residues, as propamocarb HCl, mg/kg	Report
USA, 1996 *N. Dark Red*	750 SC 375 g ai/L	1.0	187	5	14	Tuber	< 0.05*	< 0.06	A91233
USA, 1996 *Atlantic*	750 SC 375 g ai/L	1.0	187	5	10 12 14 16 18	Tuber Tuber Tuber Tuber Tuber	< 0.05* < 0.05* < 0.05* < 0.05* < 0.05*	< 0.06 < 0.06 < 0.06 < 0.06 < 0.06	A91233
USA, 1996 *Atlantic*	750 SC 375 g ai/L	1.0	187	5	14	Tuber	< 0.05*	< 0.06	A91233
USA, 1996 *Atlantic*	750 SC 375 g ai/L	1.0	187	5	14	Tuber	< 0.05*	< 0.06	A91233
USA, 1996 *Norkotah*	750 SC 375 g ai/L	1.0	187	5	14	Tuber	< 0.05*	< 0.06	A91233
USA, 1996 *Chieftan*	750 SC 375 g ai/L	1.0	187	5	14	Tuber	< 0.05*	< 0.06	A91233
USA, 1996 *R. Burbank*	750 SC 375 g ai/L	1.0	187	5	14	Tuber	< 0.05*	< 0.06	A91233
USA, 1996 *R. Burbank*	750 SC 375 g ai/L	1.0	187	5	14	Tuber	< 0.05*	< 0.06	A91233
USA, 1996 *R.Burbank*	750 SC 375 g ai/L	1.0	187	5	14	Tuber	< 0.05*	< 0.06	A91233
USA, 1996	750 SC 375 g ai/L	1.0	187	5	14	Tuber	< 0.05*	< 0.06	A91233
USA, 1996 *Mac*	750 SC 375 g ai/L	1.0	187	5	14	Tuber	< 0.05*	< 0.06	A91233
USA, 1996 *R. Burbank*	750 SC 375 g ai/L	1.0	187	5	14	Tuber	< 0.05*	< 0.06	A91233
USA, 1996 *Russet Burbank*	750 SC 375 g ai/L	1.0	187	5	10 12 14 16 18	Tuber	< 0.05* < 0.05* < 0.05* < 0.05* < 0.05*	< 0.06 < 0.06 < 0.06 < 0.06 < 0.06	A91233

* value reported

Radish

Eleven glasshouse trials with radish, conducted between 1984 and 2002, were reported from Germany and the Netherlands using seed and/or foliar treatment. The results are shown in Table 44.

Table 44. Residue trials with propamocarb hydrochloride conducted in radish in the glass house.

Country, Year *Variety*	Application Form.	Application Method	Application kg ai/ha	Application Water L/ha	Application No.	PHI (Days)	Sample analysed	Residues, Propamocarb mg/kg	Residues, Propamocarb HCl, mg/kg	Report
Germany, 1984 *Hilds Karissima*	722 SL 722g ai/L	Seed Treatment	7.22 g/kg seed	n.a	1	24 31 34 38 45	Leaves Root Root Root Root	29.53 11.05 2.35 2.20 1.05	35.24* 13.19* 2.80* 2.63* 1.25*	A85238
		Spray	0.72	1000	1					
Germany 1984 *Cherry Belle*	722 SL 722g ai/L	Seed Treatment	7.22 g/kg seed	n.a	1	27 34 38 41 45 48	Leaves Leaves Root Root Root Root	0.75 0.14 0.11 0.09 < 0.08 < 0.08	0.89* 0.17* 0.13* 0.11* < 0.1* < 0.1*	A85238
		Spray	0.72	1000	1					

Country, Year *Variety*	Application					PHI (Days)	Sample analysed	Residues, Propamocarb mg/kg	Residues, Propamocarb HCl, mg/kg	Report
	Form.	Method	kg ai/ha	Water L/ha	No.					
Germany	722 SL	Seed	7.22	n.a	1	17	Leaves	1.84	2.2*	A85238
1984	722g ai/L	Treatment	g/kg			24	Root	0.33	0.39*	
Juwasprint			seed			26	Root	< 0.08	< 0.1*	
		Spray	0.72	1000	1	32	Root	< 0.08	< 0.1*	
						34	Root	< 0.08	< 0.1*	
Germany	722 SL	Seed	7.22	n.a	1	21	Leaves	3.75	4.47*	A85238
1984	722g ai/L	Treatment	g/kg			29	Root	0.36	0.43*	
Saxa			seed			31	Root	0.45	0.54*	
		Spray	0.72	1000	1	35	Root	0.39	0.46*	
						37	Root	0.15	0.18*	
						42	Root	0.18	0.22*	
Germany	722 SL	Seed	7.22	n.a	1	7	Leaves	5.6	6.7*	A85238
1984	722g ai/L	Treatment	g/kg			14	Leaves	1.76	2.1*	
Hild's Topsi			seed			17	Root	0.33	0.39*	
GS		Spray	0.72	1000	1	22	Root	0.23	0.28*	
						27	Root	0.13	0.16*	
Germany	722 SL	Seed	7.22	n.a	1	18	Leaves	0.59	0.71*	A85238
1984	722g ai/L	Treatment	g/kg			26	Root	0.09	0.11*	
Eterna			seed			28	Root	< 0.08	< 0.1*	
		Spray	0.72	1000	1	32	Root	< 0.08	< 0.1*	
						39	Root	< 0.08	< 0.1*	
Germany	722 SL	Seed	7.22	n.a	1	14	Leaves	3.85	4.59*	A85238
1984	722g ai/L	Treatment	g/kg			21	Root	0.16	0.19*	
Rota			seed			24	Root	0.15	0.18*	
		Spray	0.72	1000	1	28	Root	0.20	0.24*	
						33	Root	0.22	0.26*	
Netherlands	722 SL	Spray	1.1	500	2	13	Root	0.42	0.5*	A85223
1983	722g ai/L									
Heemskerk										
Netherlands	722 SL	Spray	1.1	500	1	15	Root	0.34	0.4*	A85223
1983	722g ai/L									
Heemskerk										
Netherlands	840 SL	Spray	1.5-1.6	533-619	2	0	Root	0.92	1.1*	C035997
2002	530g ai/L					7	Root	0.75	0.9*	
Gudar						14	Root	0.38	0.45*	
						19	Root	0.28	0.34*	
Netherlands	840 SL	Spray	1.3	548-536	2	0	Root	0.80	0.96*	C035997
2002	530g ai/L					7	Root	0.60	0.72*	
Gudar						14	Root	0.36	0.43*	
						19	Root	0.25	0.30*	
Netherlands	840 SL	Spray	1.3	548-543	2	0	Root	1.26	1.5*	C035997
2002	530g ai/L					8	Root	0.30	0.36*	
Gudar						14	Root	0.27	0.32*	
						21	Root	0.17	0.2*	
Netherlands	840 SL	Spray	1.3	452-537	2	0	Root	1.6	1.9*	C035997
2002	530g ai/L					7	Root	0.20	0.24*	
Gudar						14	Root	0.30	0.36*	
						21	Root	0.26	0.31*	

* value reported

Chicory witloof

Twenty greenhouse trials on chicory Witloof with propamocarb hydrochloride were reported from France, Germany and the Netherlands using drip or drench irrigation and foliar spray (Table 45).

Table 45. Residue greenhouse trials with propamocarb hydrochloride conducted in Chicory Witloof.

Country, Year of trial *Variety*	Application Form.	Method	kg ai/ha application	Water L/ha	No.	PHI (Days)	Sample analysed	Residues as Propamocarb mg/kg	Residues Propamocarb HCl, mg/kg	Report no.
France. 1998 *Atlas*	722 SL 722 g ai/l	Spray onto roots	53.8	26700	1	21	Leaves	0.5*		11358[a]
France, 1998 *Atlas*	722 SL 722 g ai/l	Spray onto roots	57.1	28300	1	21	Leaves	0.7*		11358[a]
France, 1998 *Atlas*	722 SL 722 g ai/l	Spray onto roots	58.9	29200	1	21	Leaves	0.6*		11358[a]
France, 1998 *Atlas*	722 SL 722 g ai/l	Spray onto roots	60.5	30000	1	21	Leaves	0.9*		11358[a]
France, 2001 *Bea*	840 SL 530 g ai/l	Nutrient solution	15.8 g/hL		1	21	Leaves	0.18	0.22*	C024398
France, 2001	840 SL 530 g ai/l	Nutrient solution	15.1 g/hL		1	21	Leaves	0.03	0.03*	C024398
France, 2003 *Opal*	840 SL 530 g ai/l	Spray onto roots	106	40000	1	21 21	Leaves Roots	1.0* 12*		RA 2709/03 [a]
France, 2003 *Atlas*	840 SL 530 g ai/l	Spray onto roots	106	40000	1	21 21	Leaves Roots	3.6* 15*		RA 2709/03 [a]
France, 2004 *Mont Blanc*	840 SL 530 g ai/l	Spray onto roots	106	60000	1	21 21	Leaf Root	0.56* 2.8*		RA 2550/04 [a]
France, 2004 *Mont Blanc*	840 SL 530 g ai/l	Spray onto roots	106	60000	1	21 21	Leaf Root	0.41* 3.2*		RA 2550/04 [a]
France, 2004 *Passion*	840 SL 530 g ai/l	Irrigation water	84.8	40000	1	21 21	Leaf Root	0.09* 0.36*		RA 2551/04
Germany 2001, *Atlas*	840 SL 530 g ai/l	Nutrient solution	12.2 g/hL		1	19	Leaves	0.1	0.12*	C024398
Germany 2001, *Focus*	840 SL 530 g ai/l	Nutrient solution	47.1 g/hL		1	22	Leaves	8.0	9.6*	C024398
Germany, 2001, *Focus*	840 SL 530 g ai/l	Nutrient solution	47.1 g/hL		1	21	Leaves	1.6	2.1*	C024398
Germany 2004, *Atlas*	840 SL 530 g ai/l	Spray onto roots	95.0	40000	1	21 21	Leaf Root	0.46* 4.4*		RA 2550/04 [a]
Germany 2004, *Plantine*	840 SL 530 g ai/l	Irrigation water	21.2 g/hL		1	22 22	Leaf Root	5.3* 24*		RA 2551/04
Netherlands 2003, *Vintor*	840 SL 530 g ai/l	Irrigation water	21.2 g/hL		1	21 21	Leaves Roots	0.1* 0.92*		RA 2709/03
Netherlands 2003, *Plantin*	840 SL 530 g ai/l	Irrigation water	21.2 g/hL		1	21 21	Leaves Roots	0.34* 2.3*		RA 2709/03
Netherlands 2004, *Vintor*	840 SL 530 g ai/l	Irrigation water	21.2 g/hL		1	20 20	Leaf Root	0.35* 2.3*		RA 2551/04
Netherlands 2004, *Vintor*	840 SL 530 g ai/l	Spray onto roots	106	50000	1	20 20	Leaf Root	0.69* 12*		RA 2550/04 [a]

*actual value reported; [a.] the roots were treated prior to the forcing step, at forcing after tranplanting of chicory roots into the forcing room as tank dilution.

Ginger

Four trials with propamocarb hydrochloride in ginger were reported from Japan (Table 46).

Table 46: Residue field trials with Propamocarb hydrochloride conducted in ginger in Japan using three drench applications of SL 640 g/kg formulation at 0.213 kg ai/hL (30,000 L/ha).

Location *Variety*	PHI (Days)	Residues as Propamocarb mg/kg	Residue Propamocab* HCl, mg/kg	Location *Variety*	PHI (Days)	Residues as Propamocarb mg/kg	Residue Propamocab* HCl, mg/kg
Ibaraki	14	1.2	1.4	Ibaraki	14	1.7	2.0
Sanshu	30	0.64	0.76	*Sanshu*	30	0.79	0.94
	60	0.19	0.23		60	0.46	0.55

Location *Variety*	PHI (Days)	Residues as Propamocarb mg/kg	Residue Propamocab* HCl, mg/kg	Location *Variety*	PHI (Days)	Residues as Propamocarb mg/kg	Residue Propamocab* HCl, mg/kg
Chiba *Zairaishu*	14 30 60	21 4.3 0.92	25 5.2 1.1	Chiba *Zairaishu*	14 30 60	12 4.5 1.3	14 5.4 1.5

FATE OF RESIDUES DURING PROCESSING

Cabbage

Four field trials were conducted with propamocarb hydrochloride in Germany with cabbage (Pollmann, 2002, C025591). The product was applied twice as a drench treatment (72.2 and 36.1 kg ai/ha 7−10 days before transplanting) with a further 2 foliar applications 14 ± 1 days after transplanting (2.17 kg and 4.33 kg ai/ha), which corresponds to approximately double the maximum label rate. Samples of whole head cabbage were taken 27−31 days after the last application.

The cabbage samples were processed to sauerkraut and cooked cabbage according to industrial processing procedures. In the procedure for sauerkraut, the cabbage heads were cut in an Alexanderwerk mill and compacted by hand into the fermentation jars and salt solution added. The jars were firmly closed so that lactic-acid fermentation would begin for a period of 3 weeks. After opening the jars the fermented sauerkraut was sieved in order to separate it from the sauerkraut juice. For pasteurisation the sauerkraut was put into glass bottles and the sauerkraut juice added and then heated up to 90 ^{o}C. For the cooked cabbage, each cabbage head was cut into 8 parts with further processing steps carried out using two parts from opposite sides of each head. The outer leaves of each cabbage head were removed. The stem was separated into inner and outer stalks and inner leaves. The inner leaves were cut and cooked until the cut cabbage was ‘well done’.

Samples were analysed for residues of propamocarb, calculated as propamocarb hydrochloride, using a validated method (C015449). For this method, recoveries of propamocarb in spiked processed samples were between 89−106%. The LOQ was 0.01 mg/kg. The residue levels and the processing factor for each sample are shown on Table 47.

Table 47. Processing factors for propamocarb in cabbage processed products.

Matrix	Trial 1		Trial 2		Trial 3		Trial 4		PF (mean)
	Residue (mg/kg)	PF	Residue (mg/kg)	PF	Residue (mg/kg)	PF	Residue (mg/kg)	PF	
Cabbage	0.17	-	0.26	-	0.84	-	0.05	-	-
Processing of sauerkraut									
Outer Leaves	0.14	0.82	1.10	4.2	8.5	10.1	0.07	1.4	4.1
Cut Cabbage	0.04	0.24	0.02	0.08	0.04	0.05	0.05	1.00	0.34
Sauerkraut	0.04	0.24	0.01	0.04	0.04	0.05	0.05	1.00	0.33
Sauerkraut Juice	0.09	0.53	0.02	0.08	0.12	0.14	0.06	1.20	0.49
Pasteurised Sauerkraut	0.05	0.29	0.02	0.08	0.06	0.07	0.05	1.00	0.36
Pasteurised Sauerkraut Juice	0.10	0.59	0.05	0.19	0.06	0.07	0.04	0.80	0.41
Cooking process									
Outer Leaves	1.0	6.1	0.86	3.3	7.7	9.2	0.15	3.0	5.4
Inner Leaves	0.03	0.18	0.05	0.19	0.01	0.01	0.04	0.80	0.29
Stem (Inner & outer stalks)	0.09	0.53	0.02	0.08	0.61	0.73	0.03	0.60	0.48
Cooked Cabbage	0.03	0.18	0.04	0.15	0.03	0.04	0.05	1.00	0.34
Cooked Liquid	0.04	0.24	0.03	0.12	0.03	0.04	0.04	0.80	0.30

Potatoes

In one study conducted with potatoes in the USA propamocarb hydrochloride was applied five times as a foliar spray at a rate of 2.4 kg ai/ha (2.5× GAP) (Williams, 1996; A89423). The potatoes were processed into potato flakes, potato chips, wet peel, and dry peel or cleaned by hand, washed, washed and peeled and washed peel. Details of the processing procedures were not given in the report. Samples were analysed by validated methods, and no residues of propamocarb were found in any raw potato or processed product (LOQ of 0.05 mg/kg).

Tomatoes

Propamocarb was applied to tomato five times as a foliar spray at a rate of 6.62 kg ai/ha, corresponding to approximately 5 times the recommended label rate in the USA. Applications were made on a seven day interval and the tomatoes were harvested at normal maturity, 3 days after the last application (Singer, 1999; C002143).

Three sub-samples were taken from the field and processed individually into tomato purée and tomato paste using a procedure that simulates typical commercial practices. The tomatoes were washed twice while being conveyed by flumes and moving belts through flume washers and spray washers. The washed tomatoes were ground and crushed while heated to approximately 93 °C. The juice was passed through a 0.033-inch mesh screen to remove peel and seeds (wet pomace), which was weighed and discarded. The filtered juice was concentrated to puree in a vacuum evaporator. The juice was assayed for the natural tomato soluble solids (NTSS) and was concentrated until it contained 10–11% NTSS. The puree was mixed to homogeneity before a sample was taken for canning. The remainder was condensed further in a steam-jacketed kettle to tomato paste which contained 25–26% NTSS. After further heating, the samples taken for canning contained 29–31% NTSS.

Samples were analysed for residues of propamocarb using a validated analytical method (A85140). The mean recovery for propamocarb in the processed samples spiked at all fortification levels was 91% and the LOQ was 0.05 mg/kg. The level of residues in the samples and the calculated processing factor is shown in Table 49.

Table 48. Processing of tomatoes to tomato purée and paste.

	Subsample A		Subsample B		Subsample C		
Matrix	Residue (mg/kg)	PF	Residue (mg/kg)	PF	Residue (mg/kg)	PF	PF (mean)
Tomato (RAC)	10.9	-	10.3	-	11.0	-	-
Tomato Purée	12.2	1.1	14.8	1.4	14.9	1.4	1.3
Tomato Paste	32.4	3.0	32.8	3.2	33.2	3.0	3.1

RESIDUES IN FOOD IN COMMERCE AND AT CONSUMPTION

No monitoring data for propamocarb/propamocarb HCl on food commodities was submitted.

APPRAISAL

Propamocarb, a carbamate fungicide, was evaluated by JMPR three times in the 1980´s and the last time in 2005, when an ADI of 0–0.4 mg/kg bw and an ARfD of 2 mg/kg bw were established. The residue evaluation of the compound was completed by the current Meeting within the periodic review program.

Data submitted by the manufacturers and evaluated at this Meeting include metabolism in animal and plant, degradation in soil, residues in succeeding crops, analytical methods, residue trials and

processing studies. The Government of Japan submitted GAP information and summary tables of residue trials.

Animal metabolism

A study was conducted with a lactating cow orally dosed twice daily for seven consecutive days at 11.5 mg/kg [^{14}C]-propamocarb HCl equivalents in the diet (2.0 mg/kg bw/day). Over 70% of the administered dose was excreted in the urine and total radioactive residues (TRR) in tissues and bile accounted for 0.7% of the administered dose. Cumulative radioactivity recovered in the milk (0.599 mg/kg) accounted for 0.46% of the administered dose. The residues in the milk were always higher in the afternoon, with a mean of 0.054 ± 0.008 mg/kg propamocarb HCl eq (n = 7), and a maximum of 0.057 mg/kg on day 6 than in the morning (mean: 0.035 ± 0.003 mg/kg propamocarb HCl eq. (n = 7) and the maximum of 0.037 mg/kg on day 5). No residues (< 0.01 mg/kg) were found in milk fat. TRR was higher in liver (0.415 mg/kg) and muscle contained < 0.02 mg/kg.

Propamocarb represented 24.6% TRR in muscle (0.005 mg/kg), 23.5% in kidney (0.025 mg/kg), 6.2% TRR in liver (0.026 mg/kg) and 6.0% TRR in milk (0.003 mg/kg). The compound was either oxidised to form propyl propamocarb N-oxide (Met IV), dimethylated at the di-methyl amine group or hydroxylated at the propyl side chain following cyclisation to form propamocarb oxazolidin-2-one (Met VI). Met IV was the main metabolite found in kidney, liver and muscle (40–49% TRR or 0.008 to 0.203 mg/kg), Met VI was mainly found in urine (59% TRR). 2-hydroxy propamocarb was the main metabolite in milk, with 37.5% TRR (0.022 mg/kg). N-desmethyl propamocarb metabolite was found in milk, muscle and faeces (< 10% TRR), but not in kidney and liver.

Rat metabolism studies provided to the Meeting and extensively reviewed by the 2005 JMPR has shown a pathway and metabolism profile similar to that found in cow.

Plant metabolism

In one study conducted in the USA in 1996 on spinach, 14C-propamocarb was applied twice as a foliar spray at 2.53 kg ai/ha. Samples were harvested immediately following the first application (day 0), just prior to the second application (day 20) and three days after the second application (day 23). Samples were extracted with acidic methanol and extracted filter cake re-extracted with acidic methanol in a Soxhlet system. On average, TRR ranged from 203 to 236 mg/kg propamocarb HCl equivalents, with over 97% TRR being extracted. Propamocarb was the main residue found in the sample extracts, with over 75% TRR. Metabolites IV, VI, 2-hydroxyl and N-desmethyl propamocarb corresponded to < 7.5% TRR

In one study conducted in UK in 2002 in lettuce, [^{14}C]-propamocarb was applied three times to soil at 72.2 kg ai/ha followed by three foliar applications in a greenhouse at 1.08 kg ai/ha. Plants were harvested 38 days after final soil treatment and 21 days after final foliar treatment. Samples were extracted sequentially with methanol and water and the remained plant residues re-extracted by refluxing with 2M HCl and 2M NaOH. TRR in the samples harvested after soil applications was 8.2 mg/kg propamocarb HCl eq., of which only 2.8% TRR (0.23 mg/kg) was the parent compound. Most of the residues (54.4% TRR) was found in an unidentified polar region. Samples harvested 21 days after the foliar treatments had a TRR of 10.7 mg/kg, of which 91% was extracted with methanol and 0.2% remained unextracted. About 90% of the radioactivity found in the methanol and water extracts was identified as propamocarb and three unknown regions accounted each for < 4% TRR. The presence of radioactive residues in the control samples (0.35 mg/kg) suggests the incorporation of volatile radioactive products, probably $^{14}CO_2$, into the structure of the plant.

Three metabolism studies conducted with potato were submitted to the Meeting. In two greenhouse studies conducted in Germany in 1989/1994, plants were treated three times by foliar application, at 2.45 kg ai/ha and potato tubers harvested approximately 6 weeks after the final treatment. In the first study, TRR present in the samples corresponded, on average, to 0.82 mg/kg propamocarb HCl

equivalents, of which 45.5% was extracted with acidic methanol. The ^{14}C residue present was equally distributed between peel and flesh. Propamocarb represented 49.6% TRR, partitioning mainly in the methanol fraction. One metabolite, representing 8.6% TRR or 0.07 mg/kg, had the same chromatographic behaviour as propyl-propamocarb-N-oxide (Met IV). In the second study, 90% of the radiolabelled material was recovered after acidic methanol or acetonitrile extraction followed by alkaline and acid hydrolysis of the remaining material. About 32% TRR was present in the organic extract and 6.6% was unextracted. HPLC analysis using normal and reverse phase showed about 7% TRR of the sample being identified as propamocarb and approximately 50% TRR as d-glucose.

In a field study conducted with potato in UK in 2001, [^{14}C]-Propamocarb was applied six times as a foliar spray at 2.2 kg ai/ha and at 10.8 kg ai/ha. Samples were harvested approximately 7 days after the last treatment and extracted with methanol, water and refluxed in HCl and NaOH base. At the lower spray rate, TRR corresponded to 0.112 mg/kg propamocarb HCl eq. in tuber, 0.05 mg/kg in peel, 0.02 mg/kg in flesh and 85.9 mg/kg in foliage. Values for samples from the higher rate ranged from 0.05 to 476 mg/kg. Unextracted residues ranged from 4.8 to 12.2% TRR. Chromatographic and MS analysis of extracts from the lower rate treatment showed < 2% TRR as propamocarb in tuber and 28.6% TRR in foliage. Residues were mainly found in an unidentified chromatographic region (77.4 and 30% TRR in tuber and foliage). Three metabolites were tentatively identified in both samples: hydroxypropyl propamocarb (0.5% TRR in the tuber), N-desmethyl propamocarb (only detected in foliage at 5.7% TRR) and propyl propamocarb N-oxide (Met IV), present at 3.2% TRR in the tuber (0.004 mg/kg). No unchanged propamocarb was released from the foliage water extract from the higher rate treatment after acid, base and enzyme treatment

In a greenhouse study conducted in Germany in 1998, cucumbers were grown in soil treated once with [^{14}C]-propamocarb HCl applied at 2.9 kg ai/ha (11.8 mg ai/plant) and harvested at 30 days post treatment. Hydroculture-grown cucumbers were treated once at a rate of 53.4 mg ai/plant and sampled with a PHI of 21 days. Samples were extracted using maceration and soxhlet with acidic methanol. Propamocarb residues represented 19.3% TRR in cucumber extracts from the soil treatment and 58.4% TRR in hydroponic treatment. Unextracted residues represented, on average, 6.5% TRR. Polar metabolites represented 59.2 and 32.1% TRR, respectively and the remaining ^{14}C residues detected were incorporated into natural products.

In one greenhouse study conducted with tomato in UK in 2001, [^{14}C]-Propamocarb was applied four times to soil at 0.007 (1×) or 0.036 kg ai/ha (5×) and as a single foliar treatment at 2.2 kg ai/ha. Samples were extracted by maceration with methanol and water, with further acid and basic extraction as necessary. Tomato samples from soil treatments harvested at 14 to 35 days PHI showed, on average, 64.3% TRR present in the methanol extract. From 46.5 to 85.7% TRR of the foliar treated samples harvested after 7 to 28 days were found in the methanol extracts. Propamocarb was not detected in the 14 days 1× soil treated sample, but was the major component of the 7 days foliar treated tomato sample (75.2% TRR; 0.065 mg/kg). The appearance of residues in the control plants, an unknown region observed also in chromatograms of treated plants, suggest the incorporation of volatile ^{14}C into plant natural products.

In summary, in spinach, lettuce and tomato treated with propamocarb as a foliar spray, the parent compound was the main residue (> 70% TRR). Lettuce, cucumber and tomato grown on treated soil showed < 20% TRR as propamocarb, but the majority of the radioactivity found was unidentified polar compounds. The parent propamocarb amounted to 1.9 to 49.6% TRR in potato plants sprayed with propamocarb. In all studies, there was evidence of volatile ^{14}C incorporation into plant material. Results from the spinach and potato studies showed that metabolites are formed by hydroxylation of the terminal propyl chain, N-demethylation and N-oxidation of the parent molecule. No metabolites were found in the samples in larger amounts than the 5% TRR.

Rotational crops

In a confined rotational crop study, bare soil was treated at approximately 6 kg ai/ha, representing 1.2 times the annual maximum application rate for propamocarb. Leafy lettuce, radish and wheat were planted 30 days, 120 days and 365 days after treatment. In crops planted in the 30 day aged soil, total residues ranged from 0.36 (radish roots) to 2.33 mg/kg (wheat straw), and declined sharply in crops planted in soil aged 120 days and 365 days to a maximum of 0.09 mg/kg propamocarb HCl eq. Propamocarb was found in all acidic methanol sample extracts from the 30 day aged soil and was the major component (15.4% TRR in wheat straw to 67.4% TRR in radish tops), except for wheat grain, where the oxazolidine metabolite (Met VI) represented 19.9% TRR. 2-hydroxy propamocarb, N-oxide (Met IV) and desmethyl propamocarb (wheat only) were not present in any sample at levels < 10% TRR. The remainder residue was a complex mixture of highly polar components. Residues released after acid and base hydrolysis (< 10% TRR) indicated a similar pattern of metabolites.

In rotational field studies conducted in 10 American states (11 trials) in 1997, four applications at 1.68 kg ai/ha of propamocarb were made to soil with a five day interval. Wheat, sugar beets, table beets, dry beans and soybeans were planted 30, 60 or 365 days after the final soil treatment. Samples of wheat grain, forage, hay and straw, soybean seed, forage and hay, beets root and tops, and dry bean were harvested at typical sampling times. Wheat was the only crop grown on 30 days aged soils which contained residues at or above LOQ. Therefore, only wheat samples were analysed from all crops grown on 60 days aged soil.

As samples from the 60 day aged soil were generally < LOQ (0.05 mg/kg), samples from the 365-day were not analysed. Residues were detected only in wheat hay and forage samples from the 30 day aged soil. Residues were in the range of 0.051 to 0.229 mg/kg or both hay and forage.

Environmental fate in soil

In five studies conducted from 1978 to 1986 with [^{14}C]-propamocarb hydrochloride incubated under aerobic conditions at 15 or 25^{o}C in loamy sand soil containing 200 mg/kg labelled compound, propamocarb degraded very rapidly with a half life (DT_{50}) ranging from 10 to 28 days. In three studies conducted at 10 or 20^{o}C, clay loam, loamy silt, loamy sand and silty sand soils were incubated with propamocarb incorporated at the rates of 0.00361 or 3.61 kg ai/ha, for 120 days. Degradation of the parent compound was slower in a clay loam soil with a higher clay and organic carbon content, reaching 27.1% TRR at the end of the study at 20^{o}C. Half life determined in the soils ranged from 10.9 days in loamy sand to 29.7 days in silty sand soil. Lower incubation temperature decreased the degradation rate of propamocarb in loamy silt soil with half lives of 11.7 and 25.3 days at 20^{o}C and 10^{o}C, respectively. The study using Borstel soil at 10°C indicated that the rate of degradation slowed with depth, with DT_{50} values ranging from 73.7 days at 20 cm to 267 days at 90 cm, probably due to decreasing microbial activity and organic carbon content in deeper soil layers.

In a study conducted with 4 sandy loam soils and 2 clay loam soils incubated with 250mg/kg and 10 mg/kg [^{14}C]-propamocarb HCl at 20 and 10 ^{o}C for 120 and 365 days, the majority of the radioactivity was assigned to propamocarb, decreasing to a maximum of 22.1% TRR in the soil with the lowest organic carbon and biomass content (sandy loam). This soil also had the highest half life among the soils (87.7 days) while for the others DT_{50} ranged from 14.1 to 42.2 days. Up to ten non-identified metabolites, none of them being present above 10% TRR, were found in the soil extracts from all the studies.

One study conducted in sterilized and non-sterilized German standard soil suggests that soil degradation of propamocarb is mediated by micro-organisms.

Degradation of ^{14}C-propamocarb hydrochloride under anaerobic conditions was much slower than in an aerobic environment, with a half life in loamy sand soil at 25^{o}C of 459 days. The half life of propamocarb in flooded sandy loam soil treated with 250 mg/kg or 10 mg/kg and kept under anaerobic conditions in the dark at 20^{o}C was 308.2 and 65.7 days respectively. Propamocarb was

quickly removed from the water phase (DT_{50} of 14.7 days at 250mg/kg rate). The major degradation product, which was not identified, reached a maximum of 6.6% TRR in the system after 365 days. In one study to investigate the photolysis of propamocarb on soil surface, the estimated half life under irradiated conditions was 35.4 days.

One field dissipation study was conducted in the USA with sandy loam and loamy sandy soils, bare or covered with turf grass, treated four times at 9.35 kg ai/ha rate. DT_{50} in bare soils, thatch and grass ranged from 13.2 to 23.7 days. No propamocarb residues (< 0.002 mg/kg) were detected during the four month period in bare soil layer deeper than 30 cm.

In summary, propamocarb is not expected to accumulate in soil. The compound degrades relatively fast to many unidentified products (each < 10% TRR) under aerobic conditions at 10–25°C, with half life ranging from 10 to 87.7 days, with the longer times occurring in soils with lower organic matter content, possibly due to lower microbial activity. Under anaerobic conditions, propamocarb degradation was very slow in bare or flooded soil (DT_{50} > 300 days). The compound is rapidly transferred from the water to the soil in a flooded system.

Analytical methods

The residue methods used to analyse propamocarb were validated using the free base or the hydrochloride. Plant materials can be extracted with 1% acetic acid and the compound quantified by HPLC/MS/MS (electrospray ionization) at m/z 102 and or 144. Avocado extracts requires a partition step with n-hexane to remove the fat before the chromatography. Some methods also include a C18 SPE clean up step of the acid extract before the final determination. These methods were validated in many laboratories, at levels from 0.01 mg/kg to 10 mg/kg, for lettuce, chicory witloof, peppers, potato, processed potato, spinach, leek, onion, cabbage, cauliflower, Brussels sprout, broccoli, cucumber, avocado, melon and wheat grain. In most cases mean recoveries were within the acceptable levels (70–120%) with a maximum CV of 20% (n = 2–9). LOQ was 0.01 mg/kg, as propamocarb (free base) or propamocarb HCl.

In some laboratories, plant materials were extracted with acidified methanol, the extract basified with NaOH solution and cleaned up with a series of extraction procedures with chloroform, acidic water and di-isopropyl ether. The free base formed was quantified by GC/N/FID or GC/MSD. This method was validated for many crops at levels from 0.05 to 10 mg/kg, with mean recovery and CV falling within the acceptable levels (n = 2–8). LOQ was either 0.05 or 0.1 mg/kg, as propamocarb HCl.

Propamocarb can be extracted from animal products with 1.0% HCl in methanol and residues analyzed by HPLC-MS/MS. Validation at fortification levels of 0.01 mg/kg (LOQ) and 0.10 mg/kg, as propamocarb (free base), for animal tissues, milk and eggs gave recoveries from 83 to 101% and CV < 20% (n = 5).

Residues of propamocarb hydrochloride can be extracted from soil using HCl or acidified methanol, followed by a sequence of clean-up steps of the extract (chloroform/1N HCl/di-isopropyl ether) and the free base determined by GC/N/FID or GC/MSD. The method was successfully validated from 0.026 to 50 mg/kg in four different studies. In another method, propamocarb was extracted with HCl, the extract was cleaned-up on a C18 column, the final extract was basified with ammonia solution and the free base was determined by LC-MS/MS. LOQ was 0.02 mg/kg, with a mean recovery of 89% and CV of 8% (n = 5).

Stability of pesticide residues in stored analytical samples

Propamocarb residues are stable under frozen conditions, up to 26 months of storage in tomato samples fortified at 0.5 mg/kg (> 75% remained). At 5 mg/kg level, the average residue was 67% after 14.5 months of storage. Lettuce samples fortified at 0.5 and 5.0 mg/kg and stored for 14 were stable under frozen conditions (over 85% of the residues remained).

Residue definition

Metabolism studies conducted in spinach, lettuce and tomato treated with propamocarb as a foliar spray have shown that the parent compound was the main residue (> 70% TRR). Lettuce, cucumber and tomato grown on treated soil and potato samples after foliar treatment showed < 50% TRR as propamocarb. In these cases, the majority of the radioactivity (> 50% TRR) was present as unidentified polar metabolites, probably from ^{14}C incorporation into plant material, as d-glucose.

As propamocarb was the major compound present in treated plants, the Meeting agreed that the residue definition in plants for both enforcement and dietary intake purposes is propamocarb (free base).

Propamocarb represented a maximum of 24.6% TRR in cow tissues, while propyl propamocarb N-oxide (Met. IV) was the main compound detected in kidney, liver and muscle (40–49% TRR) and 2-hydroxy propamocarb was the main metabolite in milk (37.5% TRR). No metabolism study on poultry was provided.

Although propamocarb is not the main residue found in animal tissues and milk, no analytical method determining the metabolites is available that would be suitable for enforcement. No residues are expected in feed. The Meeting agreed that the residue definition for animal products for both enforcement and dietary intake purposes is propamocarb.

Propamocarb HCl has a log $P_{OW} < 0$ and animal metabolism studies have shown that it does not concentrate in fat. The Meeting concluded that propamocarb is not fat soluble.

Residues from supervised trials

Formulations containing propamocarb hydrochloride, alone or co-formulated with other active substances were used in the trials. When residues were reported in the studies as propamocarb hydrochloride, the values were multiplied by 0.84 and expressed as propamocarb.

Metabolism studies conducted in lettuce using soil treatment at a rate corresponding to 72.2 kg ai/ha have shown that < 3% TRR represented propamocarb residues in leaves after 38 days. The Meeting agreed that the seedbed drench application is not expected to contribute to final residues in crops treated with additional foliar sprays and or drip irrigation/soil drench. Consequently the trial, in which seedbed drench applications were made at higher or lower than GAP, was considered for MRL estimation.

No residue data was submitted for celery, beetroot, Brussels sprouts and strawberry. The Meeting agreed to withdraw the previous recommendations for these crops

Onion

In Europe, propamocarb is registered for use on onions in Poland (PHI of 7 days), Sweden (PHI of 30 days) and UK (PHI of 133 days). In seven trials conducted in France, Germany, the Netherlands, Spain and UK, propamocarb was applied four times at rates from 0.75–2.9 kg ai/ha and samples collected at 0 and/or 14 days. Residues, as propamocarb, ranged from < 0.008 to 0.29 mg/kg.

As no trials were conducted according to GAP, the Meeting did not recommend a maximum residue level for propamocarb in onions.

Cabbage

Propamocarb is registered to be used in Europe as a foliar application (Germany), as a seedbed or soil drench (Greece, Spain and UK) or both treatments (Italy and Netherlands). In Italy, GAP is 2 applications at 16 kg ai/ha seedbed drench applications and 3 × 1.1–2.2 kg ai/ha foliar treatment, with a PHI of 20 days.

Seventeen trials conducted in France, Germany, Italy and Spain in 2000/2001 using 72 and 36 kg ai/ha seedbed drench followed by two applications at 2.2–3.8 kg ai/ha foliar, head cabbage samples were collected from day 30 up to day 138. In one trial, samples harvested within 22 days PHI gave residues of 0.03 mg/kg.

As only one trial was conducted according to GAP, the Meeting could not recommend a maximum residue level for propamocarb in cabbage.

The Meeting also withdrew its previous recommendation for propamocarb in cabbage of 0.1 mg/kg.

Cauliflower

Propamocarb is registered to be used in Europe as a foliar application (e.g. Belgium and Germany), as a seedbed or soil drench (Greece) or both treatments (Italy, the Netherlands and UK). In Italy, GAP is 2 × 16 kg ai/ha seedbed drench up to 3 × 1.1–2.2 kg ai/ha foliar, with a PHI of 20 days. In the Netherlands, GAP is 2 × 3.61 kg ai/ha seedbed drench and 2 × 2.2–3.6 kg ai/ha foliar, with a PHI of 14 days.

Twenty three trials were conducted in France, Germany, Greece, Italy, Spain and UK from 2000 to 2002 using 72.2 and 36.1 kg ai/ha seedbed drench followed by 2 × 2.2–3.8 kg ai/ha foliar. In four trials, residues in cauliflower heads at 14 or 21 days PHI were 0.008, 0.02, 0.05 and 0.09 mg/kg. In the other trials samples harvested 30 to 138 days after the last application gave residues ranging from < 0.008 to 0.02 mg/kg.

The Meeting confirms the previous recommendation of a maximum residue level of 0.2 mg/kg for propamocarb in cauliflower and also recommends a STMR of 0.035 mg/kg and a HR of 0.09 mg/kg.

Fruiting vegetables, cucurbits

Cucumber

Thirty seven trials were conducted with propamocarb in cucumber in Europe and the USA from 1991 to 2004. In Europe, propamocarb is registered to be used as a seed treatment, soil treatment, within irrigation and/or foliar treatment.

In Spain, one label allows one seedbed drench treatment at 14.4–21.7 kg ai/ha, two soil drench treatments at 0.15 to 0.50 kg ai/hL, one treatment through dripper equipment at 1.4–2.1 kg ai/ha and two foliar treatments at rates of 0.144–0.22 kg ai/hL with a 3 day PHI (F/GH). Five trials were conducted in France, Greece, Italy and Spain using two seedbed drench applications at 72/36 kg ai/ha, followed by one drip irrigation treatment at 1.7–3 kg ai/ha, two spray applications at 1.7–3 kg ai/ha (0.36–0.6 kg ai/hL) and another drip irrigation treatment at the same previously applied rate. Nine trials were conducted in Germany and Spain using one seedbed drench (29 kg ai/ha), two soil drenches (1.7–2.9 kg ai/ha, up to 1.5 kg ai/hL), two spray applications (1.7–3.2 kg ai/ha, approximately 0.5 kg ai/hL) and two more soil drench application at the same rate as previously applied. Four trials used seedbed drench (16 kg ai/ha) and drip irrigation application (1.5–2.7 kg ai/ha). These 18 trials are within the Spanish GAP giving residues within 3 days PHI of 0.40, 0.54, 0.59, 0.60, 0.70, 0.80 (2), 0.83, 1.0 (4), 1.3, 1.4 (2), 1.7, 1.8 and 4.8 mg/kg.

In Germany, propamocarb can be used as a foliar application at 4 × 2.2 kg ai/ha. In eight trials conducted in the country in 1991/1992, within GAP, residues at 4 days PHI were 0.60, 0.68, 0.90 (3), 1.0 and 1.3 mg/kg.

Four trials using drench/drip irrigation treatment conducted in Germany, the Netherlands and Spain did not match any European GAP.

In the USA, propamocarb can be used as a foliar application at 5 × 1.0 kg ai/ha. In seven trials conducted in that country in 1997, according to GAP, residues at 2 days PHI were 0.26, 0.29, 0.32, 0.61, 0.62, 0.69 and 0.75 mg/kg.

In four trials conducted in Japan according to GAP, residues at 21 days were 0.34, 0.37, 0.39 and 0.42 mg/kg. These trials could not be considered by the Meeting as only a summary data was provided.

Residues from 33 trials conducted according to GAP in Europe and USA in cucumber gave residues within the same range and can be combined as 0.26, 0.29, 0.32, 0.40, 0.54 (2), 0.59, 0.60 (2), 0.61, 0.62, 0.68, 0.69, 0.70, 0.75, 0.80 (2), 0.83, 0.90 (3), 1.0 (5), 1.3 (2), 1.4 (2), 1.7, 1.8 and 4.8 mg/kg.

Melons

A total of 48 trials were conducted with propamocarb in melons in Europe, where the compound is registered in many countries. In Spain, the product can be applied up to four times as a seedbed drench at 15.9 kg ai/ha and as a drip irrigation treatment at 1.1–1.6 kg ai/ha with a PHI of 14 days. In nine trials conducted in Germany, Italy, Portugal and Spain from 2001–2004 conforming to Spanish GAP (two seedbed drench application), propamocarb residues in fruit were < 0.008 (3), 0.04, 0.12, 0.21, 0.45, 1.0 and 1.4 mg/kg. In four trials, melon pulp was also analysed, giving residues of 0.06, 0.08, 0.17 and 0.53 mg/kg.

In Italy, the product can be used as a seedbed incorporation after drilling (2 × 57.8–86.6 kg ai/ha) and as a foliar treatment (2 × 1.1–2.2 kg ai/ha) and a PHI of 20 days. In 13 trials conducted in France, Italy, Greece and Spain in 2000/2001 at 2 × 20–24 kg ai/ha (seedbed drench) followed by two foliar applications at 2–2.2 kg ai/ha, residues in fruit 20 days after treatment were 0.04, 0.07, 0.08 (2), 0.17 (3), 0.25, 0.3, 0.59, 0.67 (2) and 2.2 mg/kg. Residues in melon pulp were < 0.01, 0.01 (3), 0.02 (5), 0.03 (2), 0.08 and 0.17 mg/kg. As the seedbed drench application is unlikely to contribute significantly to the final residues after the foliar application, these trials can be considered to be within the Italian GAP. In nine other trials conducted at the same rate, samples harvested up to 14 days after the last application gave residues in the fruit ranging from 0.10 to 0.90 mg/kg.

In Germany, propamocarb can be used up to four times as a foliar application in the field at 2.2 kg ai/ha and a PHI of 4 days. In France, it can be applied up to six times at 1.1 kg ai/ha with a 3 day PHI. Seventeen trials conducted at 3 to 5 applications at 1.1 or 2.2 kg ai/ha can be considered as being within German or French GAP, giving residues in the fruit at a 3 day PHI of 0.10, 0.11, 0.12, 0.14, 0.23, 0.24, 0.28, 0.38 (2), 0.40, 0.44 (2), 0.57, 0.65, 0.92 and 1.1 mg/kg. Melon pulp was analyzed in 15 trials, giving residues of < 0.04 (5), 0.04, < 0.08 (6), 0.07, 0.13 and 0.21 mg/kg.

In seven trials conducted with propamocarb in cantaloupe in the USA in 1997 according to GAP (five foliar applications at 1 kg ai/ha), propamocarb residues at a two day PHI were 0.29 (2), 0.34, 0.44, 0.66, 0.77, and 1.4 mg/kg.

Residues in melon fruit from 39 trials conducted in Europe and in seven trials conducted on cantaloupe in the USA according to GAP can be combined as < 0.008 (3), 0.04 (2), 0.07, 0.08 (2), 0.10, 0.11, 0.12 (2), 0.14, 0.17 (3), 0.21, 0.23, 0.24, 0.25, 0.28, 0.29 (2), 0.34 (2), 0.38 (2), 0.40, 0.44 (3), 0.45, 0.49, 0.57, 0.59, 0.65, 0.66, 0.67 (2), 0.77, 0.92, 1.0, 1.1, 1.4, 1.42 and 2.2 mg/kg.

Residues in melon pulp from 32 trials were < 0.01, 0.01 (3), 0.02 (5), 0.03 (2), < 0.04 (5), 0.04, 0.06, 0.07, < 0.08 (6), 0.08 (2), 0.13, 0.17(2), 0.21 and 0.53 mg/kg.

Summer squash

In six trials conducted with propamocarb in summer squash in the USA in 1997 according to GAP (five foliar applications at 1 kg ai/ha), residues of propamocarb at a 2 day PHI were, 0.37, 0.43, 0.49, 0.64, 0.99 and 1.1 mg/kg.

In the USA and in some European countries, GAP for propamocarb is for the crop group cucurbits. The Meeting, therefore, agreed to combine the residue population of cucumber, melons and summer squash from 85 trials conducted in Europe and USA to make recommendations for the crop group of fruiting vegetables, cucurbits. The residues were, in rank order: < 0.008 (3), 0.04 (2), 0.07, 0.08 (2), 0.1, 0.11, 0.12 (2), 0.14, 0.17 (3), 0.21, 0.23, 0.24, 0.25, 0.26, 0.28, 0.29 (3), 0.32, 0.34 (2), 0.37, 0.38 (2), 0.4 (2), 0.43, 0.44 (3), 0.45, 0.49 (2), 0.54 (2), 0.57, 0.59 (2), 0.60 (2), 0.61, 0.62, 0.64, 0.65, 0.66, 0.67 (2), 0.68, 0.69, 0.7, 0.75, 0.77, 0.8 (2), 0.83, 0.90 (3), 0.92, 0.99, 1.0 (6), 1.1 (2), 1.3 (2), 1.4 (3), 1.42, 1.7, 1.8, 2.2 and 4.8 mg/kg.

The Meeting recommends a maximum residue level of 5 mg/kg for propamocarb in fruiting vegetables, cucurbits.

The Meeting recommends a STMR of 0.59 mg/kg and a HR of 4.8 mg/kg for propamocarb in fruiting vegetables, cucurbits, except melons and watermelons.

Based on the residue data on melon pulp, the Meeting recommends a STMR of 0.04 mg/kg and a HR of 0.53 mg/kg for melons and watermelons.

The Meeting withdraws its previous recommendation for propamocarb in cucumber of 2 mg/kg.

Peppers, sweet

Thirty five trials were conducted with propamocarb hydrochloride in sweet pepper in Europe and the USA from 1997 to 2004 using drench, drip irrigation or foliar treatment.

Propamocarb is registered in Europe and the USA for drench, drip and/or foliar treatment. In Spain, the product can be applied twice as a seedbed treatment after sowing (15.9 kg ai/ha) and up to four times as a drip irrigation treatment (1.1–1.6 kg ai/ha) with a 3 day PHI. In the Netherlands, up to three seedbed drench applications at 36.1 kg ai/ha and up to 5 drench/drip applications at 1.0/0.72 kg ai/ha are allowed, with a three day PHI.

In 18 trials conducted in greenhouses in Belgium, Italy, the Netherlands, Spain and Greece, using two seedbed applications followed by four soil drip or drench applications according to Spanish GAP, residues at three days PHI were < 0.008 (8), 0.008 (2), 0.02, 0.03, 0.06, 0.08, 0.10, 0.14, 0.15, 0.16 mg/kg as propamocarb. In three trials conducted at higher rates, residues at three days PHI ranged from < 0.008 to 0.05 mg/kg.

In the USA, propamocarb can be used in peppers as foliar application at 5 × 1.26 kg ai/ha with a 5 day PHI. In 10 trials conducted in that country according to GAP, residues were 0.07, 0.16, 0.20, 0.23, 0.26, 0.27, 0.32, 0.62, 0.98, 1.8 mg/kg. These trials gave residues at a higher range than trials conducted in Europe using drench/drip applications and the two residue population could not be combined.

The Meeting agreed to recommend a maximum residue level, based on USA trials, of 3 mg/kg, a STMR of 0.265 mg/kg and a HR of 1.8 mg/kg for propamocarb in sweet peppers.

The Meeting withdraws its previous recommendation for propamocarb in sweet peppers of 1 mg/kg.

Eggplant

Propamocarb is not registered for use in eggplant in the USA. In Europe, the compound has the same GAP as for sweet peppers. The Meeting agreed to use the residue trial data for sweet peppers in Europe to recommend a maximum residue level of 0.3 mg/kg, a STMR of 0.008 mg/kg and a HR of 0.16 mg/kg for propamocarb in eggplant.

Tomato

Forty five trials were conducted with propamocarb hydrochloride in tomato in Europe and the USA from 1997 to 2004.

Propamocarb is registered in Europe and USA for drench, drip and/or foliar treatment. In Spain, the product can be applied twice as a seedbed treatment after sowing (15.9 kg ai/ha) and up to four times as a drip irrigation treatment (1.1−1.6 kg ai/ha) with a 3 day PHI. In the Netherlands, up to three seedbed drench applications at 36.1 kg ai/ha and up to five drench/drip applications at 1.0/0.72 kg ai/ha are allowed, with a three day PHI.

In two trials conducted in greenhouses in Spain and Germany using seedbed drench followed by drench or dripping according to Spanish GAP, residues at three days PHI were < 0.008 and 0.08 mg/kg. In 16 trials conducted at higher GAP rates or number of application gave residues from < 0.008 to 0.05 mg/kg three days after the last application. In 9 trials conducted using drench treatment (1 to 2 × 15.9–72.2 kg ai/ha) followed by drench/dripping irrigation (2 to 4 treatments at 0.6–4.3 kg ai/ha), residues after three days ranged from < 0.008 to 0.07 mg/kg.

In the USA, propamocarb can be used in tomato as foliar application at 5 × 1.3 kg ai/ha and 5 days PHI. In 18 trials conducted in the country according to GAP, residues were 0.14, 0.16, 0.23, 0.25, 0.34, 0.37, 0.38, 0.40, 0.51, 0.52 0.60, 0.61 (2), 0.65, 0.68, 0.86, 0.94 and 1.4 mg/kg. Clearly, these trials gave residues at a higher range than the two trials conducted in Europe using drench/drip application and the two residue populations cannot be combined.

The Meeting agreed to recommend maximum residue level based on USA trials of 2 mg/kg, a STMR of 0.515 mg/kg and a HR of 1.4 mg/kg for propamocarb in tomato.

The Meeting withdrew its previous recommendation for propamocarb in tomato of 1 mg/kg.

Lettuce

Propamocarb is registered in lettuce in Europe and the USA. Sixty eight trials were conducted in leaf and head lettuce as a drench and/or foliar spray in France (21), Germany (18), Greece (3), Italy (3), the Netherlands (2), Spain (7) and USA (14) between 1993 and 2002.

GAP in some countries in Europe include two seedbed drench (SD) and two foliar (F) treatments, the rates being 1.6 g ai/m^2 (16 kg ai/ha) (SD) /1.1−1.6 kg ai/ha (Field, F, and Greenhouse, GH) in Italy, with 14 days PHI. In 12 trials (F or GH) conducted in Germany, Greece, Italy, the Netherlands and Spain at Italian GAP, residues at 14 days PHI were 0.92, 1.8, 3.9, 4.0, 4.5, 7.1, 8.1, 9.4, 9.8, 10, 11 and 13 mg/kg. Thirty two field trials were conducted in France, Germany, Greece, Italy and Spain using 2 seedbed drench applications at 72.2 and 36.1 kg ai/ha and 2 foliar applications at 1.66 kg/kg ai/ha. Residues at 14 days PHI determined in 28 trials were 0.7, 1.0 (2), 1.9, 2.0, 2.8, 3.2, 3.3, 4.2, 4.4, 4.7, 6.0, 6.5, 7.4, 7.9, 8.1, 9.2, 13 (2), 14, 15, 16 (2), 17, 21, 29, 31 and 40, mg/kg. In other four trials samples were harvested only after 21 days.

In Belgium, Germany and UK (F/GH), only foliar treatment is recommended, with three applications at 1.1 to 1.4 kg ai/ha and 21 days PHI. In 10 greenhouse trials conducted in France within UK GAP residues at 21 days PHI were 1.2, 1.7, 4.9, 6.5, 14, 15, 20, 24, 39 and 40 mg/kg; In other six trials with four applications, residues ranged from 14 to 40 mg/kg.

The 50 residue trials conducted with propamocarb in Europe can be combined as 0.7, 0.92, 1.0 (2), 1.2, 1.7, 1.8, 1.9, 2.0, 2.8, 3.2, 3.3, 3.9, 4.0, 4.2, 4.4, 4.5, 4.7, 4.9, 6.0, 6.5 (2), 7.1, 7.4, 7.9, 8.1 (2), 9.2, 9.4, 9.8, 10, 11, 13 (3), 14 (2), 15 (2), 16 (2), 17, 20, 21, 24, 29, 31, 39 and 40 (2) mg/kg.

GAP in the USA is four foliar applications at 1.68 kg ai/ha, two days PHI. In 14 trials conducted in the USA in leafy and head lettuce according to GAP, residues were 8.2, 9.7, 10, 11 (2), 17, 19, 31, 41 (2), 48, 51, 60 and 86 mg/kg. Residues in the head without the wrapper leaves of head lettuce ranged from 0.21 to 8.0 mg/kg.

In four trials conducted in Japan at GAP (3 × 1.28 kg ai/ha), residues at 14 days PHI were 0.28, 0.60, 1.6 and 1.8 mg/kg. These trials could not be considered by the Meeting as only summary data was provided.

The 64 European and USA trials can be combined to give one residue population as 0.7, 0.92, 1.0 (2), 1.2, 1.7, 1.8, 1.9, 2.0, 2.8, 3.2, 3.3, 3.9, 4.0, 4.2, 4.4, 4.5, 4.7, 4.9, 6.0, 6.5 (2), 7.1, 7.4, 7.9, 8.1 (2), 8.2, 9.2, 9.4, 9.7, 9.8, 10 (2), 11 (3), 13 (3), 14 (2), 15 (2), 16 (2), 17 (2), 19, 20, 21, 24, 29, 31 (2), 39, 40 (2), 41 (2), 48, 51, 60 and 86 mg/kg.

The Meeting recommended a maximum residue level of 100 mg /kg, a STMR of 9.9 mg/kg and a HR of 86 mg/kg for propamocarb in lettuce, head and leaf.

The Meeting withdrew its previous recommendation for propamocarb in lettuce, head of 10 mg/kg.

Spinach

Propamocarb is registered in Italy as seed, soil and foliar treatments. As a foliar treatment, the label recommends up to three applications at 1.1–2.2 kg ai/ha with a 20 day PHI. Seven trials were conducted in Belgium, Germany, Italy and Spain three applications at 1.3–1.6 kg ai/ha. In four trials, samples were analysed at a 21 day PHI, giving residues of propamocarb of 0.41, 8.4, 14 and 29 mg/kg.

The Meeting recommended a maximum residue level of 40 mg /kg, a STMR of 11.2 mg/kg and a HR of 29 mg/kg for propamocarb in spinach.

Potato

Thirty two trials were conducted with propamocarb HCl between 1990 and 2003 using foliar application in Europe and the USA. In one trial conducted in France and in 11 trials conducted in Germany according to German or UK GAP (6 × 0.94–0.99 kg ai/ha) at a 7 day PHI the residues, as propamocarb, were < 0.01 (2), < 0.08 (8) and 0.17 (2) mg/kg. In two German trials, residues measured in peeled tuber gave residues of < 0.08 (2) mg/kg. Nineteen trials were conducted in the USA, 18 of those at GAP (5 applications at 1.0 kg ai/ha), giving residues at 14 days PHI of < 0.05 (16) and 0.05 (2) mg/kg, measured as propamocarb. One trial conducted at higher rate gave residues of < 0.05 mg/kg.

In summary, residues according to GAP, were < 0.01 (2), ≤ 0.05 (16), 0.05 (2), < 0.08 (8) and 0.17 (2) mg/kg, as propamocarb. The Meeting recommended a maximum residue level of 0.3 mg/kg, a STMR of 0.05 mg/kg and a HR of 0.17 mg/kg for propamocarb in potato.

Radish

In Europe, propamocarb is registered in radish in Germany and the Netherlands with a 14 day PHI. In six trials conducted in Germany using one seed treatment and one foliar treatment at GAP rate (7.22 g/kg seed/0.722 kg ai/ha), root samples were collected from day 21 to 48 days after the last application, giving residues up to 11 mg/kg as propamocarb. In one trial, samples were collected 14 days after the last application, giving residues of 0.33 mg/kg. In four trials conducted in the Netherlands using two foliar applications within the GAP rate (1.08 kg ai/ha) residues at 14 days were 0.27, 0.30, 0.36, 0.42 mg/kg as propamocarb. Two trials conducted at higher or lower rates gave residues within the same range. Residues from five trials conducted according to GAP were 0.27, 0.30, 0.33, 0.36, 0.42 mg/kg

The Meeting recommended a maximum residue level of 1 mg/kg, a STMR of 0.33 mg/kg and a HR of 0.42 mg/kg for propamocarb in radish.

The Meeting withdrew its previous recommendation for propamocarb in radish of 5 mg/kg.

Chicory

In France, propamocarb is approved for application to chicory by spraying onto roots at the start of forcing at 72.2 kg ai/ha with a PHI of 21 days. In five trials conducted according to GAP in France in

1998, residues in the leaves were 0.46, 0.50, 0.60, 0.70 and 0.90 mg/kg as propamocarb. In five trials conducted in France, Germany and Netherlands at 106 kg ai/ha, residues in leaves at 21 days ranged from 0.41 to 3.6 mg/kg.

Propamocarb is also registered in France and Luxembourg to be used via a nutrient solution at 9 g/hL and 21 days PHI. In 10 trials conducted in France, Germany and Netherlands propamocarb was applied using a nutrient solution or irrigation system at 12.2 to 47.1 g/hL rate. Residues in leaves within 21 days PHI ranged from 0.03 to 0.35 mg/kg.

The Meeting recommended a maximum residue level of 2 mg /kg, a STMR of 0.60 mg/kg and a HR of 0.90 mg/kg for propamocarb in chicory.

Ginger

Results from four trials conducted at two sites in Japan with propamocarb HCl in ginger in 1986 were submitted to the Meeting as a summary table. GAP in Japan is up to five drench applications at 0.11–0.16 kg ai/hL with a 30 day PHI. The trials conducted at 3 × 0.213 kg ai/hL gave residues in the tubers at 30 days ranging from 0.64 to 4.5 mg/kg, as propamocarb.

As no trials were conducted according to GAP, the Meeting could not recommend a maximum residue level for propamocarb in ginger.

Fate of residues in processing

Four field trials were conducted in Germany with cabbage in 2001 with propamocarb hydrochloride applied twice as a drench treatment with a further two foliar applications at approximately double the maximum label rate. Samples of whole head cabbage were taken 27–31 days after the last application and processed to sauerkraut and cooked cabbage according to industrial processing procedures. Residues in cabbage head ranged from 0.05 to 0.84 mg/kg propamocarb hydrochloride. Residues decreased in sauerkraut and sauerkraut juice with mean/median processing factors (PF) of 0.33/0.15 and 0.49/0.33. PFs for the pasteurized products were 0.36/0.18 and 0.41/0.32, respectively. Residues also decreased in cooked cabbage, with a mean/median PF of 0.34/0.17.

In one study conducted with potatoes in the USA propamocarb hydrochloride was applied as a foliar spray at an exaggerated rate (2.5 × GAP). Potatoes were processed into potato flakes, potato chips, wet peel, and dry peel. Details of the processing procedures were not given. No residues were found in any raw potato or processed product (< 0.05 mg/kg).

In one study conducted in tomato in the USA in 1996, propamocarb was applied five times as a foliar spray at an exaggerated rate (5 × GAP rate). Tomatoes were harvested at normal maturity three days after the last application and three sub-samples were taken to be processed individually into tomato purée and tomato paste using a procedure that simulates typical commercial practices. Residues in RAC ranged from 10.3 to 11 mg/kg, as propamocarb. Residues concentrated in purée and paste, with a mean/median PF of 1.3/1.4 and 3.1/3, respectively.

Based on the STMR of 0.515 mg/kg for propamocarb in tomato and the median PF, the Meeting recommends a STMR of 0.721 for propamocarb in tomato purée and a STMR of 1.54 for propamocarb in tomato paste.

Residues in animal commodities

Feeding studies

No animal feeding studies were provided to this Meeting. Data from one metabolism study conducted with a lactating cow dosed with propamocarb at 2 mg/kg bw/day for seven days have shown that propyl propamocarb N-oxide (Met. IV) was the main compound detected in kidney, liver and muscle

(40–49% TRR, up to 0.203 mg/kg propamocarb eq. in liver) and 2-hydroxy propamocarb was the main residue in milk (37.5% TRR or 0.022 mg/kg eq.). Propamocarb represented a maximum of 24.6% TRR, present at levels < 0.03 mg/kg.

Dietary burden of farm animals

From all the commodities for which propamocarb uses were considered by the JMPR, potato processed products are the only ones included in the animal diets according to the *FAO Manual* (FAO 2002). In two trials conducted with potato according to GAP, residues in washed peel, were < 0.08 mg/kg. As wet peel represents 75% and 40% of beef cattle and dairy cattle diets respectively, no animal dietary burden is expected from the uses of propamocarb considered by the JMPR.

Residues in animal commodities

The Meeting recommended a maximum residue level of 0.01(*) mg/kg and a STMR of 0 mg/kg for propamocarb in eggs, milks, edible offal (mammalian), poultry edible offal, poultry meat and meat (from mammals other than marine mammals). The Meeting also recommends an HR of 0.01 mg/kg for eggs, edible offal (mammalian), poultry edible offal, poultry meat and meat (from mammals other than marine mammals).

RECOMMENDATIONS

Residue definition for compliance with MRLs and estimation of dietary intake for plant commodities: *propamocarb*

Summary of the recommendation for the MRL, STMR and HR for propamocarb.

		Recommended MRL (mg/kg)		STMR (P)	HR (P)
CCN	**Commodity name**	**New**	**Previous**	**mg/kg**	**mg/kg**
VB 0041	Cabbages, Head	W	0.1		
VB 0404	Cauliflower	0.2	0.2	0.035	0.09
VX 0624	Celery	W	0.2		
VS 0469	Chicory witloof (sprouts)	2		0.60	0.90
VC 0424	Cucumber	W	2		
VR 0574	Beetroot	W	0.2		
VB 0402	Brussels sprouts	W	1		
VC 0045	Fruiting vegetables, cucurbits	5			
	Fruiting vegetables, cucurbits, except melons, and watermelons			0.59	4.8
VL 0482	Lettuce, head	100	10	9.9	86
VL 0483	Lettuce, leaf	100	10	9.9	86
MO 0105	Edible offal (mammalians)	0.01*		0	0.01
VO0440	Eggplant	0.3		0.008	0.16
PE 0012	Eggs	0.01*		0	0.01
MM 0095	Meat (from mammals other tha	0.01*		0	0.01

CCN	Commodity name	Recommended MRL (mg/kg) New	Recommended MRL (mg/kg) Previous	STMR (P) mg/kg	HR (P) mg/kg
	marine mammals)				
VC 0046	Melons, except watermelon			0.04	0.53
ML 0106	Milks	0.01*		0	
VO 0485	Peppers, sweet	3	1	0.265	1.8
VR 0589	Potato	0.3		0.05	0.17
PM 0111	Poultry, edible offal of	0.01*		0	0.01
PM 0110	Poultry meat	0.01*		0	0.01
VR 0494	Radish	1	5	0.33	0.42
VL 0502	Spinach	40		11.2	29
FB 0275	Strawberry	W	0.1		
VO 0448	Tomato	2	1	0.515	1.4
	Tomato purée			0.721	
	Tomato paste			1.54	
VC 0432	Watermelon			0.04	0.53

DIETARY RISK ASSESSMENT

Long-term intake

The ADI for propamocarb is 0-0.4 mg/kg bw. The International Estimated Daily Intake (IEDI) for propamocarb was estimated for the 13 GEMS/Food cluster diets using the STMR or STMR-P values estimated by the current Meeting for 11 plant commodities. The results are shown in Annex 3 of the 2006 JMPR Report. The IEDI ranged from 0 to 1% ADI. The Meeting concluded that the long-term intake of residues of propamocarb from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The ARfD for propamocarb is 2 mg/kg bw. The International Estimated Short Term Intake (IESTI) for propamocarb was calculated for the plant commodities for which STMRs and HRs were estimated and for which consumption data were available. The results are shown in Annex 4 of the 2006 JMPR Report. The IESTI ranged from 0 to 40% ARfD for the general population and from 0 to 80% ARfD for children. In both populations, the highest intake came from the consumption of lettuce. The Meeting concluded that the short-term intake of residues of propamocarb from uses that have been considered by the JMPR is unlikely to present a public health concern.

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PROPARGITE (113)

First draft prepared by David Lunn, New Zealand Food Safety Authority, Wellington, New Zealand.

EXPLANATION

Propargite was first evaluated by the JMPR for residues in 1977 and toxicology in 1978, with subsequent evaluations in 1978, 1979, 1980 and 1982. A periodic review (toxicology) was conducted by JMPR in 1999 when an ADI of 0-0.01 mg/kg bw was estimated and it was concluded that an acute reference dose was not necessary. A periodic review of residues was conducted in 2002 and MRLs were recommended by JMPR for many crops and animal commodities. MRLs for beans (dry), pears, potato, pears, strawberry and walnuts were among those not proposed because of insufficient data or GAP information.

The CCPR (36th Session, 2004, paragraph 141, ALINORM 04/27/24) retained the existing Codex MRLs for beans (dry), pear, potato, strawberry and walnuts for 4 years under the periodic review procedure so that additional data could be provided to support these MRLs.

Information on registered uses and data from supervised residue trials on beans (dry), potato and walnuts, together with additional information on methods of analysis and residue stability in stored analytical samples, were provided to the Meeting by the USA manufacturer. Advice was also received from The Netherlands that propargite was not authorised for use on agricultural crops in that country.

Additional GAP information and summaries of residue trials conducted in Japan on tea, citrus, apples, grapes, peaches and cherries were provided by the Japanese manufacturer.

METHODS OF RESIDUE ANALYSIS

Animal metabolism

The meeting received analytical method descriptions and validation data for propargite in beans (dry), walnuts and potato. These methods involved minor variations on a method ME-208, capable of determining both propargite and the metabolite t-butyl phenoxy cyclohexanol (TBPC).

The reported methods involved the acetonitrile extraction of residues in homogenized plant matrices, with the filtered extract being washed with hexane before being mixed with 10% NaCl and partitioned into either petroleum ether or hexane. After concentration and clean-up using a Florisil® column, the extracts were evaporated to dryness or near dryness. In some studies (potato and dry beans), the primary metabolite (TBPC) was then derivatised by incubation with triethylamine and heptafluorobutyric anhydride. Extracts were then resuspended in either hexane or toluene and analyzed for propargite using a gas chromatograph equipped with a mass selective detector (GC-MSD) in the selected ion monitoring mode.

Table 1. Propargite analytical methods.

Beans (dry), Potato			
Dzialo, 2006 [Ref: 2005-006], Korpalski, 2006 [Ref: RP-04002]			
Analytes:	Propargite and metabolite TBPC	GC-MSD	ME-208 ver 5
LOQ:	0.02 mg/kg (dry beans), 0.01 mg/kg (potato)		
Description	Samples were extracted with acetonitrile, filtered, washed with hexane and back-extracted with hexane-saturated acetonitrile. After evaporation of the acetonitrile extract to near dryness and the addition of 10% NaCl solution, residues were		

partitioned into hexane. Clean-up involved elution with 10% acetone in hexane through a Florasil® column. After evaporation to dryness, extracts were incubated in triethylamine and heptafluorobutyric anhydride (to derivatise the TBPC metabolite) and extracted into hexane. Analysis for propargite was by GC using a mass selective detector (ions 135 or 350, 173 or 335).

Potato

Korpalski, 2006 [Ref: RP-97013]

Analytes: Propargite and metabolite TBPC GC-MSD CAL 004-50

LOQ: 0.01 mg/kg

Description Samples were extracted with acetonitrile, filtered and washed with hexane. After evaporation of the acetonitrile extract to about 50ml, residues were partitioned into petroleum ether/10% NaCl solution. Clean-up was by elution with 15% acetone/petroleum ether through a Florasil column. After evaporation and resuspension in toluene residues of propargite were analysed by GC using a mass selective detector (ions 135 or 350, 173 or 335).

Walnut (nutmeat)

Russell & Dzialo, 2006 [Ref: 2005-008]

Analytes: Propargite GC-MSD GRL-12307

LOQ: 0.02 mg/kg

Description Samples were extracted with acetonitrile, filtered, washed with hexane and back-extracted with hexane-saturated acetonitrile. After evaporation of the acetonitrile extract to near dryness and the addition of 10% NaCl solution, residues were partitioned into hexane. Clean-up involved elution with 10% acetone in hexane through a Florasil® column. After evaporation to dryness, extracts were resuspended in hexane. Analysis for propargite was by GC using a mass selective detector (ions 350, 335).

Method validation studies were provided for all the reported analytical methods, with samples of beans (dry), walnut (nutmeat) and potato being fortified with propargite at levels ranging from 0.01 mg/kg to 10 mg/kg. Average recovery rates ranged from 89% to 112% in the various substates. Data from these validation studies are summarised in Table 2.

Table 2. Analytical recoveries for spiked propargite in various substrates.

Commodity	Analyte	Spike conc, mg/kg	n	Recovery%		Method	Reference
				mean	range		
Beans (dry)	propargite	0.02	6	91	81-97	ME-208 ver 5	GRL-12308
Beans (dry)	propargite	0.05	4	94	81-110	ME-208 ver 5	GRL-12308
Beans (dry)	propargite	0.2	2	89	78-107	ME-208 ver 5	GRL-12308
		Overall	12	91	RSD 14%		
Potato	propargite	0.01	8	89	86-96	CAL 0400-50	RP-97019
Potato	propargite	0.5	4	101	92-113	CAL 0400-50	RP-97019
Potato	propargite	10	4	98	95-101	CAL 0400-50	RP-97019
Potato	propargite	Overall	16	94	RSD 7.3%		
Potato	propargite	0.1	8	106	96-120	ME-208 ver 5	RP-04029
Potato	propargite	1.0	3	102	95-111	ME-208 ver 5	RP-04029
Potato	propargite	10	3	96	93-98	ME-208 ver 5	RP-04029
		Overall	14	103	RSD 7.4%		
Walnut (nutmeat)	propargite	0.02	6	102	86-120	GRL-12307	GRL-12307
Walnut (nutmeat)	propargite	0.05	3	90	78-109	GRL-12307	GRL-12307

Commodity	Analyte	Spike conc, mg/kg	n	Recovery%		Method	Reference
				mean	range		
Walnut (nutmeat)	propargite	0.1	3	112	102-121	GRL-12307	GRL-12307
		Overall	12	102	RSD 14.5%		

Stability of residues in stored analytical samples

The Meeting received information on the stability of residues of propargite in various alfalfa, almond, barley, carrot and lettuce substrates stored frozen and analysed at various intervals. In these studies, samples were extracted with hexane/2-propanol and analysed for propargite by GC-FPD (sulphur mode) after Florasil® column and gel permeation chromatography clean-up. The results of these studies are summarised in Table 3. Values were uncorrected for analytical method recovery.

Table 3. Frozen storage stability of propargite residues.

Matrix	Temp (°C)	Storage period[1]	Fortification level (mg/kg)	Method recovery%	% Residues remaining		Reference
						Mean	
Barley (grain)	-20	0	0.1	74	71, 83	77	RP-89064
		1m		69	54, 57	55	
		2m		90	56, 68	62	
		7m		71	51, 63	52	
		9m		89	78, 81	80	
		12m		72	49, 57	53	
Barley (straw)	-20	0d	0.1	84	83, 79	81	RP-89064
		1m		75	78. 74	76	
		2m		106	92, 91	91	
		7m		113	100, 103	102	
		9m		114	99, 87	93	
		12m		83	48, 54	51	
Carrot (root)	-20	0d	0.1	121	106, 104	105	RP-89064
		1m		112	87, 85	86	
		2m		123	81, 76	78	
		7m		113	88, 87	87	
		9m		107	72, 83	78	
		12m		120	76, 76	76	
Carrot (top)	-20	0	0.1	113	97, 94	95	RP-89064
		1m		94	85, 76	80	
		2m		113	84, 96	90	
		7m		103	91, 95	93	
		9m		80	85, 70	77	
		13m		70	44, 42	43	
Lettuce	-20	0d	0.1	111	101, 112	107	RP-89064
		1m		114	87, 109	98	
		2m		97	93, 104	99	
		7m		103	106, 104	105	
		9m		109	102, 103	103	
		12m		112	93, 95	94	
Almond hulls	-20	0d	1.0	108[1]	104, 104	104	RP-91057.
		1m		84[1]	85, 78	82	
		3m		101[1]	87, 85	86	
		6m		89[1]	63, 70	67	
		8m		87[1]	83, 82	83	
		12m		98[1]	93, 90	92	
Almond kernels	-20	0d	1.0	105[1]	104, 104	104	RP-91057
		1m		97[1]	88, 95	92	
		3m		94[1]	88, 88	88	
		6m		85[1]	87, 67	77	
		8m		99[1]	88, 93	91	
		12m		99[1]	82, 87	85	

Matrix	Temp (°C)	Storage period[1]	Fortification level (mg/kg)	Method recovery%	% Residues remaining		Reference
						Mean	
Alfalfa regrowth hay (dry)	-20	0d	1.0	95[1]	100, 100	100	RP-91062
		1m		99[1]	90, 104	97	
		3m		80[1]	93, 90	92	
		6m		87[1]	88, 77	83	
		8m		93[1]	90, 78	84	
		12m		98[1]	92, 82	87	
Alfalfa regrowth hay (fresh)	-20	0d	1.0	102[1]	112, 104	108	RP-91062
		1m		113[1]	117, 104	111	
		3m		90[1]	92, 92	92	
		6m		90[1]	88, 88	88	
		8m		102[1]	100, 100	100	
		12m,		97[1]	97, 85	91	
Alfalfa seed screenings	-20	0d	1.0	106[1]	112, 108	110[3]	RP-91062
		1m		101[1]	108, 104	106	
		3m		110[1]	72, 112	92	
		6m		90[1]	88, 92	90	
		8m		108[1]	112, 112	112	
		12m		101[1]	100, 90	95	
Alfalfa seed	-20	0d	1.0	108[1,2]	121, 104	113[2]	RP-91062
		1m		108[1]	100, 98	99	
		3m		89[1]	88, 92	90	
		6m		112[1]	92, 97	95	
		8m		108[1]	98, 83	91	
		12m		104[1]	88, 85	87	

[1]) Average of two replicates

USE PATTERN

Propargite is registered in a number of countries for control of mites on a wide range of fruit trees, nuts, vines, vegetables, ornamentals and field crops such as cotton, maize and peanuts. The manufacturer provided USA labels for two emulsifiable concentrate formulations and updated information on GAP. Uses relevant to this evaluation are summarised in Table 4 below.

Table 4. Propargite: Registered uses of relevance to this evaluation.

Crop	Country	Form (EC - g/litre) (WP – g/kg)	Application				PHI (days)	Comments
			Method	kg ai/ha (kg ai/hL)	Water, L/ha	No		
Beans (dry)	USA	EC 785	foliar	1.8 – 2.8	190 min (ground) 47 min (air)	2[1]	14	West of the Rocky Mountains only. Do not use vines or trash for feed or forage
Potato	USA	EC 785	foliar	1.4 – 2.3	190 - 470 (ground) 94 min (air)	2[1]	14	Pacific Northwest only. NOT through irrigation
Potato	USA	EC 719	foliar	1.7 – 2.5	190 - 470 (ground) 94 min (air)	2[1]	14	Pacific Northwest only. May be applied through sprinkler irrigation
Walnuts	USA	EC 719	foliar	1.7 – 5.0	935 min	2[1]	21	ground application
Walnuts	USA	EC 719	foliar	2.5 – 3.4	190 min	2[1]		aerial application
Tea	Japan	EC 569	foliar	0.43 – 1.5	1500 - 4000	2	14	No label sighted[3]
Citrus	Japan	WP 300	foliar	0.8 – 2.8 (0.04)	2000 - 7000	2	14	No label sighted[3]
Apple	Japan	WP 300	foliar	0.8 – 2.8 (0.04)	2000 - 7000	2	14	No label sighted[3]
Grapes	Japan	WP 300	foliar	0.6 – 2.1 (0.03)	2000 - 7000	2	14-21[2]	No label sighted[3]
Peach	Japan	WP 300	foliar	0.8 – 2.8 (0.04)	2000 - 7000	2	21	No label sighted[3]

Crop	Country	Form (EC - g/litre) (WP – g/kg)	Application				PHI (days)	Comments
			Method	kg ai/ha (kg ai/hL)	Water, L/ha	No		
Cherries	Japan	WP 300	foliar	0.8 – 2.8 (0.04)	2000 - 7000		after harvest	No label sighted[3]

[1]) Minimum spray interval of 21 days

[2]) PHI of 14 days for large berries; 21 days for small berries

[3]) GAP information provided by the manufacturer

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials involving foliar applications of propargite on the following crops:

Citrus	Japan	Table 5
Apples	Japan	Table 6
Peaches	Japan	Table 7
Cherries	Japan	Table 8
Grapes	Japan	Table 9
Beans (dry)	USA	Table10
Potato	USA	Table 11
Walnut	USA	Table 12
Tea	Japan	Table 13

Trials from the USA were well documented with laboratory and field reports. Laboratory reports included method validation including procedural recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no residues in control samples exceeded the LOQ. Control data have therefore not been included in the following tables. Residue data are recorded unadjusted for recovery.

The summaries of the Japanese trials included application details and the propargite residues found, but no information on the analytical methods or whether the reported results had been corrected for recovery.

When residues were not detected they are shown as below the LOQ (e.g. < 0.01 mg/kg). Residues, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. These results are double underlined.

Citrus

Table 5. Residue data summary of supervised trials on citrus in Japan, involving foliar applications of propargite.

CITRUS Country, year (variety)	Application				PHI, (days)	Residues (mg/kg)		Reference & Comments
	Form	kg ai/ha	kg ai/hL	no.		Pulp	Whole fruit	
Mandarins								
Japan, Osaka, 1979 (Unshu)	WP 300	1.6	0.04	2	14	< 0.04		NN-0510 (S55-3-5-NNC.1)
Japan, Kagawa, 1979 (Unshu)	WP 300	2.0	0.04	2	14	< 0.04		NN-0510 (S55-3-5-NNC.2)
Japan, Osaka, 1979 (Unshu)	WP 300	1.6	0.04	2	14	0.02		NN-0510 (S55-3-5-IET.3)
Japan, Kagawa, 1979 (Unshu)	WP 300	2.0	0.04	2	14	0.01		NN-0510 (S55-3-5-IET.4)
Japan, Saga, 1973 (Unshu)	WP 300	1.32	0.04	2	14	< 0.04		NN-0510 (S55-3-5-IET.5)
Japan, Wakayama, 1973 (Unshu)	WP 300	2.4	0.04	2	14	0.02		NN-0510 (S55-3-5-IET.6)
Japan, Saga, 1973 (Unshu)	WP 300	1.32	0.04	2	14	0.08		NN-0510 (S55-3-5-NNC.7)
Japan, Wakayama, 1973 (Unshu)	WP 300	2.4	0.04	2	14	< 0.08		NN-0510 (S55-3-5-NNC.8)
Natsudaidai								
Japan, Yamaguchi, 1994 (Shin-Amanatsu)	WP 300	2.0	0.04	2	14		1.4	NN-0510 (H7.5.25-IET.1)
Japan, Tokushima, 1994 (Amanatsu)	WP 300	1.6	0.04	2	14		2.4	NN-0510 (H7.5.25-IET.2)
Japan, Yamaguchi, 1995 (Common-Natsu)	WP 300	2.0	0.04	2	14		1.9	NN-0510 (H8.7.1-IET.3)
Japan, Ohita, 1995 (Amanatsu)	WP 300	1.6	0.04	2	14		0.94	NN-0510 (H8.7.1-IET.4)
Japan, Yamaguchi, 1995 (Common-Natsu)	WP 300	2.0	0.04	2	14		2.1	NN-0510 (H8.5.10-JEO.5)
Japan, Ohita, 1995 (Amanatsu)	WP 300	1.6	0.04	2	14		1.1	NN-0510 (H8.5.10-JEO.6)
Japan, Aichi, 1997 (Kawano-Natsudaidai)	WP 300	1.6	0.04	2	14		0.7	NN-0510 (H10.3.10-JEO.7)
Japan, Mie, 1997 (Shin-Amanatsu)	WP 300	2.0	0.04	2	14		0.61	NN-0510 (H10.3.10-JEO.8)
Japan, Wakayama, 1997 (Kawano-Natsudaidai)	WP 300	1.6	0.04	2	14		1.6	NN-0510 (H10.3.10-JEO.9)
Japan, Ehime, 1997 (Kawano-Natsudaidai)	WP 300	1.6	0.04	2	14		1.2	NN-0510 (H10.3.10-JEO.10)
Japan, Fukuoka, 1997 (Kawano-Natsudaidai)	WP 300	1.6	0.04	2	14		1.8	NN-0510 (H10.3.10-JEO.11)

Apple

Table 6. Residue data summary of supervised trials on apples in Japan, involving foliar applications of propargite.

APPLE Country, year (variety)	Application				PHI, (days)	Residues (mg/kg)	Reference & Comments
	Form	kg ai/ha	kg ai/hL	no.			
Japan, Aomori, 1997 (Fuji)	WP 300	2.0	0.04	1	14	1.24	NN-0510 (H9.12.18-IET.1)
Japan, Nagano, 1997 (Fuji)	WP 300	2.0	0.04	1	14	0.74	NN-0510 (H9.12.18-IET.2)
Japan, Aomori, 1997 (Fuji)	WP 300	2.0	0.04	1	14	1.13	NN-0510 (H10.2.23-JEO.3)
Japan, Nagano, 1997 (Fuji)	WP 300	2.0	0.04	1	14	0.7	NN-0510 (H10.2.23-JEO.4)

Grape

Table 7. Residue data summary of supervised trials on grapes in Japan, involving foliar applications of propargite.

GRAPE Country, year (variety)	Application				PHI, (days)	Residues (mg/kg)	Reference & Comments
	Form	kg ai/ha	kg ai/hL	no.			
Japan, Osaka, 1996 (Delaware)	WP 300	0.6	0.03	1	28	1.12	NN-0510 (H9.1.27-IFES.1)
Japan, Okayama, 1996 (Muscat of Alexandria)	WP 300	0.6	0.03	1	28	0.46	NN-0510 (H9.1.27-IFES.2)
Japan, Osaka, 1996 (Delaware)	WP 300	0.6	0.03	1	28	1.22	NN-0510 (H8.12.18-ARSO.3)
Japan, Okayama, 1996 (Muscat of Alexandria)	WP 300	0.6	0.03	1	14	0.7	NN-0510 (H8.12.18-ARSO.4)
Japan, Yamanashi, 1971 (Neo-Muscat)	WP 300	0.6	0.03	4	19	1.94	NN-0510 (S46-NNC.5)
Japan, Yamanashi, 1971 (Neo-Muscat)	WP 300	0.6	0.03	7	19	2.83	NN-0510 (S46-NNC.5)

Peach

Table 8. Residue data summary of supervised trials on peaches in Japan, involving foliar applications of propargite.

PEACH Country, year (variety)	Application				PHI, (days)	Residues (mg/kg)	Reference & Comments
	Form	kg ai/ha	kg ai/hL	no.			
Japan, Fukushima, 1981 (Hakuhou)	WP 300	1.6	0.03	2	21	0.02	NN-0510 (S56.10.27-JFRL.1)
Japan, Nagano, 1981 (Ohkubo)	WP 300	2.0	0.03	2	21	< 0.02	NN-0510 (S56.10.27-JFRL.2)
Japan, Fukushima, 1981 (Hakuhou)	WP 300	1.6	0.03	2	21	0.02	NN-0510 (S56.10.15-SAL.3)
Japan, Nagano, 1981 (Ohkubo)	WP 300	2.0	0.03	2	21	< 0.02	NN-0510 (S56.10.15-SAL.4)

PEACH Country, year (variety)	Application				PHI, (days)	Residues (mg/kg)	Reference & Comments
	Form	kg ai/ha	kg ai/hL	no.			
Japan, Kagawa, 1973 (Sunago-wase)	WP 300	0.8	0.03	2	20	< 0.02	NN-0510 (S48.1.7-JFRL.5)

Residues reported in fruit without skin

Cherry

Table 9. Residue data summary of supervised trials on cherries in Japan, involving foliar applications of propargite after harvesting.

CHERRY Country, year (variety)	Application				PHI, (days)	Residues (mg/kg)	Reference & Comments
	Form	kg ai/ha	kg ai/hL	no.			
Japan, Yamagata, 1995 (Satonishiki)	WP 300	1.6	0.04	2	311	< 0.01	NN-0510 (H8.9.1-IET.1)
Japan, Fukushima, 1995 (Satonishiki)	WP 300	2.0	0.04	2	297	< 0.01	NN-0510 (H8.9.1-IET.2)
Japan, Yamagata, 1995 (Satonishiki)	WP 300	1.6	0.04	2	311	< 0.01	NN-0510 (H8.7.29-JEO.3)
Japan, Fukushima, 1995 (Satonishiki)	WP 300	2.0	0.04	2	297	< 0.01	NN-0510 (H8.7.29-JEO.4)

Beans (dry)

In six trials in the USA, involving a range of dry bean varieties, propargite was applied by either tractor mounted or backpack boom sprayers to single replicate plots of 81 to 110 square metres. Two treatments were made to maturing crops, 21 days apart, with the second treatment being about 14 days before commercial harvest. Application rates were all within 5% of the target rates. Three duplicate samples (min 1 kg) of mature dry beans were taken from each treatment plot (with one sample from the control plots) and these were frozen within 2.5 hours of sampling. After frozen storage for 73-137 days, the samples were analysed for propargite using method ME-208 ver 5. Recovery rates in these trials ranged from 73 – 115% (fortification at 0.023 and 0.23 mg/kg, n=4) and the reported LOQ was 0.02 mg/kg.

Table 10. Residue data summary of supervised trials on beans (dry) in the USA, involving foliar treatments with propargite.

BEANS (DRY) Country, year (variety)	Application				PHI, (days)	Residues[1] (mg/kg)	Reference & Comments
	Form	kg ai/ha	kg ai/hL	no.			
USA, South Dakota, 2005 (Navy bean - Norstar)	EC 785	2.8	1.5	2	15	0.08	CC 2005-006 (DGD-05-001)
USA, Washington, 2005 (Pinto bean - Othello)	EC 785	2.8+ 2.7	1.5+ 1.5	1+ 1	7+7[2]	0.02	CC 2005-006 (DGD-05-002)
USA, California, 2005 (Garbanzo bean – Sierra)	EC 785	2.8+ 2.7	1.5+ 1.5	1+ 1	14	0.21	CC 2005-006 (DGD-05-003)
USA, Michigan, 2005 (Navy bean - Vista)	EC 785	2.8	1.5	2	13	0.16	CC 2005-006 (DGD-05-004)
USA, Michigan, 2005 (Red kidney bean – CA Early Light)	EC 785	2.8	1.5	2	14	0.07	CC 2005-006 (DGD-05-005)

BEANS (DRY) Country, year (variety)	Application				PHI, (days)	Residues[1] (mg/kg)	Reference & Comments
	Form	kg ai/ha	kg ai/hL	no.			
USA, Pennsylvania, 2005 (??? – Great Northern)	EC 785	2.8	1.5	2	9+5[3]	0.1	CC 2005-006 (DGD-05-006)

1) Results are the mean values from analysis of three duplicate samples

2) Bean plants harvested 7 days after treatment and allowed to dry for 7 days before manual threshing

3) Bean plants harvested 9 days after treatment and allowed to dry for 5 days before manual threshing

Potato

In eight trials conducted in the USA in 1997 and 1998, propargite was applied by either tractor mounted or backpack boom sprayers to single replicate plots of 72 to 140 square metres. Two treatments were made to maturing crops, 21 days apart, with the second treatment being about 14 days before commercial harvest. Application rates were all within 5% of the target rates. Three duplicate samples (min 24 tubers or 4 kg) were taken from each treatment plot (with one sample from the control plots). In one trial, three additional samples were taken at 7 day intervals after treatment. Samples were frozen within 1.5 hours of sampling and after frozen storage for 28-160 days, were analysed for propargite using method CAL 004-50. Recovery rates in these trials ranged from 94 – 100% (fortification at 0.01 and 1.0 mg/kg) and the reported LOQ was 0.01 mg/kg.

In four additional trials conducted in the USA in 2004, propargite was applied by either tractor mounted or backpack boom sprayers to single replicate plots of 72 to 110 square metres. Two treatments were made to maturing crops, 14 days apart, with the second treatment being about 14 days before commercial harvest. Application rates were all within 5% of the target rates. Three duplicate samples (min 3 kg) were taken from each treatment plot (with one sample from the control plots) and these were frozen within 3 hours of sampling. After frozen storage for 137-154 days, the samples were analysed for propargite using method ME-208 version 5. The mean recovery rate in these trials was 91% (RSD 13%, n=16) following fortification at 0.01 mg/kg and 10 mg/kg. The reported LOQ was 0.01 mg/kg.

Table 11. Residue data summary of supervised trials on potato in the USA, involving foliar applications of propargite.

POTATO Country, year (variety)	Application				PHI, (days)	Residues[1] (mg/kg)	Reference & Comments
	Form	kg ai/ha	kg ai/hL	no.			
USA, Idaho, 2004 (Russet Burbank)	EC 785	2.3	1.2	2	15	≤ 0.01	RP-04002 (DNJ-04103)
USA, Idaho, 2004 (Russet Burbank)	EC 785	2.3	1.2	2	14	≤ 0.01	RP-04002 (DNJ-04102)
USA, Oregon, 2004 (Russet Burbank)	EC 785	2.3	1.2	2	14	≤ 0.01	RP-04002 (DNJ-04101)
USA, Washington, 2004 (Ranger Russet)	EC 785	2.3	1.2	2	14	≤ 0.01	RP-04002 (DNJ-04100)
USA, Colorado, 1997 (Norkota)	EC 785	2.3	1.2	2	14	≤ 0.01	RP-97013 (SWF-97-500)
USA, Washington, 1997 (Norkota)	EC 785	2.3	1.2	2	14	≤ 0.01	RP-97013 (DNJ-97-104)
USA, Washington, 1997 Russet Burbank	EC 785	2.3	1.2	2	14	≤ 0.01	RP-97013 (DNJ-97-105)

POTATO Country, year (variety)	Application				PHI, (days)	Residues[1] (mg/kg)	Reference & Comments
	Form	kg ai/ha	kg ai/hL	no.			
USA, Idaho, 1997 (Russet Burbank)	EC 785	2.3+ 2.3	1.3+ 1.2	1+ 1	7 14 21 28	< 0.01 ≤ 0.01 < 0.01 < 0.01	RP-97013 (DNJ-97-106)
USA, California, 1998 (White Rose)	EC 785	2.3+ 2.3	1.3+ 1.2	1+ 1	14	0.015	RP-97013 (CEJ-98-107)
USA, Washington, 1998 (Russet Burbank)	EC 785	2.3	1.2	2	14	0.01	RP-97013 (DNJ-98-115)
USA, Idaho, 1998 (Russet Burbank)	EC 785	2.3	1.2	2	14	≤ 0.01	RP-97013 (DNJ-98-116)
USA, Idaho, 1998 (Russet Burbank)	EC 785	2.3	1.2	2	14	≤ 0.01	RP-97013 (DNJ-98-117)

1) Results are the mean values from analysis of three duplicate samples

Walnuts

In six trials in the USA, propargite was applied by airblast sprayers to single replicate plots of 16–18 trees, ranging in height from 4–9 metres. Two treatments were applied, 20-21 days apart, with the second treatment being about 21 days before commercial harvest. Application rates were all within 5% of the target rates. Three duplicate samples of nuts were hand harvested from each treatment plot (with one sample from the control plots) and the kernels removed by hand, to give at least 1kg kernels per sample. These samples were frozen within 5 hours of sampling and stored frozen for 75–104 days before analysis using method GRL-12307. Recovery rates in these trials ranged from 90–120% (fortification at 0.023 and 0.12 mg/kg, n=4) and the reported LOQ was 0.02 mg/kg.

Table 12. Residue data summary of supervised trials on walnuts in the USA, involving foliar applications of propargite.

WALNUTS Country, year (variety)	Application				PHI, (days)	Residues (mg/kg)	Reference & Comments
	Form	kg ai/ha	kg ai/hL	no.			
USA, California, 2005 (Howard)	EC 719	5.1+ 4.9	0.54+ 0.52	1+ 1	20	≤ 0.02	CC-2005-008 (DGD-05-008)
USA, California, 2005 (Hartley)	EC 719	5+ 5.1	0.63+ 0.66	1+ 1	21	≤ 0.02	CC-2005-008 (DGD-05-009)
USA, California, 2005 (Chandlers)	EC 719	5.1+ 5	0.54+ 0.54	2	20	0.14	CC-2005-008 (DGD-05-010)
USA, California, 2005 (Chandlers)	EC 719	5.1+ 4.8	0.54+ 0.54	1+ 1	20	0.13	CC-2005-008 (DGD-05-011)
USA, California, 2005 (Chandlers)	EC 719	5	0.54	2	21	0.02	CC-2005-008 (DGD-05-012)
USA, California, 2005 (Hartley)	EC 719	5.1	0.54	2	21	0.08	CC-2005-008 (DGD-05-013)

Tea

Table 13. Residue data summary of supervised trials on tea in Japan, involving foliar applications of propargite.

TEA Country, year (variety)	Application				PHI, (days)	Residues (mg/kg)	Reference & Comments
	Form	kg ai/ha	kg ai/hL	no.			
Japan, Miyazaki, 1974 (Yabukita)	EC 596	1.52	0.038	2	14	0.24	NN-0510 (S50-7-3-JFRL.1)
Japan, Shizuoka, 1974 (Yabukita)	EC 596	1.52	0.038	2	14	1.0	NN-0510 (S50-7-3-JFRL.2)
Japan, Miyazaki, 1974 (Yabukita)	EC 596	1.52	0.038	2	14	0.3	NN-0510 (S50-7-3-SAL.3)
Japan, Shizuoka, 1974 (Yabukita)	EC 596	1.52	0.038	2	14	0.72	NN-0510 (S50-7-3-SAL.4)

APPRAISAL

Propargite [2-(4-*tert*-butylphenoxy)cyclohexyl prop-2-ynyl sulfite] was first evaluated by the JMPR for residues in 1977 and toxicology in 1978, with subsequent evaluations in 1978, 1979, 1980 and 1982. A periodic review (toxicology) was conducted by JMPR in 1999 when an ADI of 0-0.01 mg/kg bw was estimated and it was concluded that an acute reference dose was not necessary. A periodic review of residues was conducted in 2002 and MRLs were recommended for many crops and animal commodities. MRLs for beans (dry), pears, potato, pears, strawberry and walnuts were among those not proposed because of insufficient data or GAP information.

The CCPR (36th Session, 2004, paragraph 141, ALINORM 04/27/24) retained the existing Codex MRLs for beans (dry), pear, potato, strawberry and walnuts for 4 years under the periodic review procedure so that additional data could be provided to support these MRLs.

Information on registered uses and data from supervised residue trials in USA on beans (dry), potato and walnuts, together with additional information on methods of analysis and residue stability in stored analytical samples, were provided to the Meeting by the USA manufacturer and additional information on GAP and summaries of residue trials in Japan on citrus, apples, peaches, cherries, grapes and tea were provided by the Japanese manufacturer. No information was provided for pears or strawberries.

Analytical methods

The meeting received analytical method descriptions and validation data for propargite in beans (dry), walnuts and potato. These methods involved minor variations on a method ME-208, capable of determining both propargite and the metabolite t-butyl phenoxy cyclohexanol (TBPC). These methods were previously evaluated by JMPR in 2002.

The reported methods involved the acetonitrile extraction of residues in homogenized plant matrices, with the filtered extract being washed with hexane before being mixed with 10% NaCl and partitioned into either petroleum ether or hexane. After concentration and clean-up using a Florisil® column, the extracts were evaporated to dryness or near dryness. In some studies (potato and dry beans), the primary metabolite (TBPC) was then derivatised by incubation with triethylamine and heptafluorobutyric anhydride. Extracts were then resuspended in either hexane or toluene and analyzed for propargite using a gas chromatograph equipped with a mass selective detector (GC-MSD) in the selected ion monitoring mode. Residues of the TBPC metabolite were not reported as

this is not included in the residue definition for either compliance with MRLs or for dietary intake estimation. Limits of quantification (LOQs) for these methods were 0.02 mg/kg for beans (dry) and walnuts (nutmeat) and 0.01 mg/kg for potato.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the stability of propargite in various commodities under freezer storage (-16 to -20 °C). These studies were among those previously assessed by JMPR in 2002, where it was noted that with the exception of forage and fodder crops, more than 70% of propargite residues remained in frozen samples stored for about one year.

The Meeting agreed with the previous JMPR conclusion that propargite residues were stable in most frozen plant commodities for about one year, and could be applied to beans (dry), walnuts and potato, based on studies provided for alfalfa (seed, foliage and alfalfa hay), barley grain, almond kernels and on the potato study previously evaluated by the 2002 JMPR.

Results of supervised trials on crops

The Meeting received reports of supervised trials conducted in USA, where propargite was applied to a range of bean varieties grown for dry bean production, chick-peas, potato and walnuts and also received summaries of residue trials in Japan on citrus, apples, peaches, cherries, grapes and tea.

The Meeting agreed that the MRLs recommended at the 2002 Meeting for these latter commodities (citrus, apples, peaches, cherries, grapes and tea) were sufficient to accommodate residues arising from the Japanese GAPs, and that no further evaluation was required.

Beans (dry), chick-peas (dry)

Field trials conducted in USA on a range of bean varieties grown for dry bean production, and on chick-peas, involving two foliar applications of propargite were made available to the Meeting. Information on one additional trial on red kidney beans was provided to the JMPR 2002.

GAP in USA for *Lupinus, Phaseolus and Vicia spp* grown for dry bean production and for chick-peas, i.e., crops within the USA 'beans (dry)' group, is for up to two applications per season, with a maximum rate of 2.8 kg ai/ha, a 21 day interval between treatments with a PHI of 14 days. In six of the bean trials from USA matching the USA GAP, residues in beans (dry) were: 0.02, 0.07, 0.08, 0.1, 0.11 and 0.16 mg/kg and in one chick-pea trial, residues were 0.21 mg/kg.

The Meeting agreed that the results of the above trials could be combined and used to mutually support recommendations for beans (dry) and chick-peas, and could be extrapolated to similar commodities for which GAP existed in USA. The combined results were: 0.02, 0.07, 0.08, <u>0.1</u>, 0.11, 0.16 and 0.21 mg/kg.

The Meeting estimated maximum residue levels of 0.3 mg/kg and STMRs of 0.1 mg/kg for beans (dry), lupin (dry), broad bean (dry) and chick-pea (dry).

Potato

Field trials conducted in USA involving two foliar applications of propargite on potatoes, were made available to the Meeting.

GAP in USA for potatoes is for up to two applications per season, with a maximum rate of 2.5 kg ai/ha, a 21 day interval between treatments and a PHI of 14 days. In eight USA trials matching USA GAP, reported residue levels in potato tubers were: < 0.01 (6), 0.01 and 0.02 mg/kg. In a further four trials with a shorter treatment interval of 14 days, but otherwise matching the USA

GAP, residue levels were all < 0.01 mg/kg. The Meeting agreed to combine the data from these two sets of trials, as the different spray intervals were not expected to influence the residue levels present in the tubers. The residue levels in the combined set of 12 trials were: < 0.01 (10), 0.01 and 0.02 mg/kg.

The Meeting estimated a maximum residue level of 0.03 mg/kg for potato and estimated an STMR of 0.01 mg/kg.

Walnuts

Field trials conducted in USA on walnuts, involving two foliar (air blast) applications of propargite were made available to the Meeting.

GAP in the USA for walnuts allows up to two applications per season, with a maximum rate of 2.5 kg ai/ha, a 21 day interval between treatments and a PHI of 21 days. In six USA trials matching USA GAP, reported residue levels in walnut kernels (nutmeat) were: < 0.02(2), 0.02, 0.08, 0.13 and 0.14 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg for walnuts and estimated an STMR of 0.05 mg/kg.

Animal commodity maximum residue levels

The Meeting noted that the GAP for dry bean varieties in USA included a condition that treated vines or trash should not be used for animal forage or fodder, and that the potential contribution to the farm animal dietary burden from residues in potato culls would be insignificant (when compared to the contribution from other fruit and vegetable by-products such as citrus pulp).

The Meeting therefore concluded that the farm animal dietary burden estimated by the JMPR in 2002 was still valid and confirmed the existing maximum residue limits and STMRs estimated for propargite in animal commodities.

RECOMMENDATIONS

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

Definition of the residue (for compliance with MRLs and for estimation of dietary intake): *propargite.* This definition applies to both plant and animal commodities.

The residue is fat-soluble.

CNN	Commodity Name	MRL (mg/kg)		STMR or STMR-P (mg/kg)
		New	Previous	
VD0071	Beans (dry)	0.3	0.2	0.1
VD0523	Broad bean (dry)	0.3		0.1
VD0524	Chick-pea (dry)	0.3		0.1
VD0545	Lupin (dry)	0.3		0.1
VR0589	Potato	0.03	0.1*	0.01
TN0678	Walnuts	0.3	0.1*	0.05

DIETARY RISK ASSESSMENT

Long-term intake

The evaluation of propargite has resulted in recommendations for new maximum residue limits and STMR values for several new commodities. Data on consumption were available for 32 commodities from this and previous evaluations, and this data was used to calculate dietary intake. The results are shown in Annex 3 of the 2006 JMPR Report.

The IEDIs in the 13 GEMS/Food cluster diets, based on the estimated STMRs were 3–30% of the maximum ADI (0.01 mg/kg bw). The Meeting concluded that the long-term dietary intake of residues of propargite from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The 2002 JMPR concluded that it was unnecessary to establish an ARfD for propargite. The Meeting therefore concluded that the short-term dietary intake of residues of propargite from uses that have been considered by the JMPR is unlikely to present a risk to consumers.

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PROPICONAZOLE (160)

First draft was prepared by Arpad Ambrus, Hungary

EXPLANATION

Propiconazole was evaluated by the JMPR in 1987, 1991 and 1994 when an ADI of 0-0.07 mg/kg bw and an ARfD of 0.3 mg/kg bw were established, and a number of maximum residue levels were estimated.

The cranberry industry performed a number of supervised trials within the Interregional Research Project No. 4 to provide data for the establishment of US tolerances for propiconazole residues in cranberry. The relevant labels and reports of supervised trials were submitted for evaluation by the 2006 JMPR.

RESIDUE ANALYSIS

Analytical methods

The harvested fruit samples were analyzed by a total residue method determining propiconazole and its metabolite as 2,4 dichlorobenzoic acid by capillary gas chromatography (Toth and Manuli, 1989). The method validation recoveries from test portions spiked at 0.05, 0.5 and 1 mg/kg ranged from 63.6 to 86.2% with an overall average of 77.5% and standard deviation of 6.2. Concurrent recoveries, performed at 0.05 and 1 mg/kg levels during the analyses of samples ranged from 71 to 120% with an average of 93% and standard deviation of 14.6%.

No quantifiable residues were observed in the control samples.

Stability of pesticide residues in stored analytical samples

The maximum storage interval for field-treated samples was 78 days. To evaluate storage stability, control samples were fortified with 1.0 mg/kg propiconazole, stored at < -20oC and analyzed after 92 days of frozen storage. The average residues that survived in fortified samples (86%) were not significantly different from the analytical average recovery (77.5–93%).

The JMPR reported the results of numerous studies on various plant commodities that indicated that propiconazole residues were stable for more than 6 months (FAO 2004).

USE PATTERN

The Orbit 3.6E containing 41.8% and Tilt 250E containing 250g/L propiconazole are registered in Canada and the USA to control various fungal diseases of cranberry. They can be applied to protect cranberry as shown in Table 1.

Table 1. Use pattern of Propiconazole on cranberry.

Application						PHI
Method		**Product**	**No**	**Interval, days**	**Rate kg ai/ha**	**day**
Ground	USA	3.6E	Max 4	10	0.3-0.42	45
Ground[1]	Canada	250E	Max 2	10-14	0.31	45

[1] Apply with a minimum of 200 L water/ha to runoff

RESIDUES RESULTING FROM SUPERVISED TRIALS

During the 1995–1999 growing seasons' three field trials were conducted on cranberries in two geographical regions of the USA (Thompson 1997, Thompson and Chen 1999).

The cranberry crops were grown and maintained according to typical agricultural practices for each geographical region.

Each treated plot received four foliar broadcast applications of the test substance at a rate of approximately 0.4–0.42 kg ai/ha each. The first two applications were made at bud break and 14 days later, the third and fourth applications were made at early bloom and 10 or 14 days later.

One control and duplicate treated samples were collected at 43–44 days after the last (fourth) application.

The residue results from the supervised field trials are presented in Table 2.

Table 2. Residues of propiconazole in/on cranberry fruits following four foliar broadcast applications 14 days apart.

Field Trial Location	Dosage kg ai/ha	PHI (days)	Residue mg/kg	
			Total	Average
Warrens, WI	0.42	44	0.59, 0.46	0.53
Wisconsin Rapids, WI	0.42	44	0.18, 0.22	0.20
Oregon	0.42	43	0.23, 0.23	0.23

APPRAISAL

Propiconazole was last evaluated by the JMPR in 1987 1991,1994 and 2004 when an ADI of 0-0.07 mg/kg bw and ARfD of 0.3 mg/kg bw were established, and a number of maximum residue levels were estimated. The residue was defined as propiconazole for regulatory and dietary intake assessment purposes.

Results of supervised trials, carried out on cranberry according the US registered uses, were submitted for evaluation.

Results of supervised trials on crops

During the 1995 and 1999 growing seasons three field trials were conducted with maximum dosage on cranberries in two geographical regions of the USA.

For each trial, four broadcast foliar applications of propiconazole were made with single application rates of 0.42 kg ai/ha. The samples were collected at 43 and 44 days (US GAP: max application rate is 0.42 kg ai/ha with a PHI of 45 days)

The cranberry samples were analysed with a total residue method determining propiconazole and its metabolite as 2,4 dichlorobenzoic acid by capillary gas chromatography. The concurrent recoveries ranged from 71 to 120% with an average of 93% and standard deviation of 14.6%.

Concurrent storage stability tests indicated that the residues in samples spiked at 1.0 mg/kg level and stored at < -20^{o}C for 92 days did not degrade.

The residues measured in samples at harvest and expressed as propiconazole equivalent were: 0.2, 0.23 and 0.53 mg/kg.

The 1994 JMPR reported that over 30 days after the last application the proportion of the parent compound in the total residue was 8–11%, 21% and 18–23% in peanut, grapes and grape leaves, respectively (Evaluation 1994, part 1, vol 2. p1048). The residue composition obtained in various kinds of plant matrices indicate that the parent compound would not amount to more than 25% of the total residue in plant commodities including cranberry 45 days after last application.

Taking into account the proportion of parent compound in propiconazole residues in plant commodities, and the minimum data requirement (3 trials) specified for commodities which are insignificant in trade and do not raise any intake concern (2004 JMPR Report, pp. 30-31), the Meeting estimated a maximum residue level of 0.3 mg/kg an HR of 0.13 mg/kg and STMR of 0.058 mg/kg.

RECOMMENDATION

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

Summary of the recommendation for the MRL, STMR and HR for propiconazole.

CCN	Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR/P mg/kg
		New	Previous		
FB 0265	Cranberry	0.3		0.13	0.058

DIETARY RISK ASSESSMENT

Long-term intake

The GEMS Food specifies the following long-term cranberry consumption (g/day/person) for various cluster diets: A (0.1); D (0.3); F (0.6); M (2.5). The consumption of cranberry in the other regions is nil.

The highest IEDI in the 13 GEMS/Food regional diets, based on estimated STMR, was < 0.01% of the maximum ADI (0.07 mg/kg bw).

The Meeting concluded that the long-term intake of residues of propiconazole use on cranberry will not practically increase the intake of residues from other uses considered earlier by the JMPR.

Short-term intake

The GEMS/Food regional diet specifies the large portion sizes of cranberry of 3.53 g/kg bw for adults and 6.78 g/kg bw for children (both from the USA).

The IESTIs of propiconazole calculated on the basis of the large portion size and the estimated HR of 0.13 mg/kg are 0.15% and 0.3% of the ARfD for adults and children, respectively.

The Meeting concluded that the short-term intake of residues resulting from the use of propiconazole on cranberry that have been considered by the JMPR is unlikely to present a public health concern.

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PYRACLOSTROBIN (210)

The first draft was prepared by Mr. Arpad Ambrus, Hungary

EXPLANATION

The first evaluation of pyraclostrobin residues was carried out by the 2004 JMPR, when maximum residue levels were recommended for a large number of fruits, vegetables, cereals and products of animal origin. The residue was defined as pyraclostrobin for commodities of plant and animal origin.

Due to insufficient number of trials or registered uses no maximum residue levels could be estimated for apple, beans, coffee, cucumber, head lettuce, peppers, raspberry and soybean. Further information and results of supervised trials conducted with these commodities were submitted for evaluation to this Meeting.

Furthermore, trial data for a number of additional crops including broccoli, Brussels sprouts, cauliflower, head cabbage, hops, kale, leek, cantaloupe, sunflower and vining peas were provided for evaluation.

The Meeting evaluated the new data together with those included in the 2004 evaluation. The relevant data reported by the 2004 JMPR are also listed in this evaluation for convenience.

USE PATTERN

Pyraclostrobin is used as fungicide with foliar application alone or in combination with other active substances (e.g., boscalid is a new fungicide that extended the scope of the use of pyraclostrobin). Information provided on registered uses (Regenstein 2006/1009448, Bross M., Mackenroth C. 2005/1023124) on crops evaluated by the Meeting is summarized in Tables 1 and 2.

Table 1. Registered uses in Europe (mix formulation: BAS 516 07 F, WG 6.7% ai for foliar application by spraying).

Crop	Country	Application rate per treatment					PHI (days)	Total amount within a season [kg ai/ha]
		Number min max	Interval between applications (days)	kg ai/hL min max	water L/ha min max	kg ai/ha min max		
Apple[1]	Belgium	3	7-12	0.022	300	0.067	7	
Beans, dry	Denmark	2	14-21	0.017-0.033	200-400	0.067	21	0.134
Broccoli	Denmark	3	14-21	0.023	300	0.067	14	
Broccoli	Germany	3	14-28	0.011-0.017	400-600	0.067	14	
Brassica[2]	UK	3				0.067	14	0.201
Brussels sprout	Cyprus	2	8-12	0.007	1000	0.067	14	0.134
Brussels sprout	UK	3	21 - 28	0.007 - 0.04	200 - 1000	0.067	14	0.201
Brussels sprouts	Denmark	3	14-21	0.023	300	0.067	14	
Brussels sprouts	Germany	3	14-28	0.011-0.017	400-600	0.067	14	
Cabbage	Belgium	3	21 – 28 (7 - 14)*	0.017	400	0.067	14	0.201
Cabbage	Cyprus	2	8 - 12	0.007	1000	0.067	14	0.134
Cabbage	Nether-lands	3	14	0.033 -0.067	200-400	0.067	14	0.201
Cabbage	Nether-lands	3	21 - 28	0.033 -0.067	200-400	0.067	14	0.201
Cabbage	UK	3	21 - 28	0.007 - 0.04	200 - 1000	0.067	14	0.201
Cabbages	Poland	3	7 - 10	0.008 - 0.011	600 - 800	0.050 - 0.067	14	0.150 - 0.201
Cauliflower	Cyprus	2	8 - 12	0.007	1000	0.067	14	0.135
Cauliflower	UK	3	21 - 28	0.007 - 0.033	200 - 1000	0.067	14	0.135
Hops[1]	France	3	8-14	0.01-0.042	600-2700	0.057-0.252	21	

Crop	Country	Application rate per treatment					PHI (days)	Total amount within a season [kg ai/ha]
		Number min max	Interval between applications (days)	kg ai/hL min max	water L/ha min max	kg ai/ha min max		
Leek	Belgium	3	21 - 28 (10 - 14)*	0.017	400	0.100	14	0.400
Leek	Netherlands	2-3	10 - 14	0.025 -0.040	250-400	0.100	14	0.200
Lettuce	Belgium	2	14 - 21	0.013	500	0.100	14	0.200
Lettuce (outdoor and protected)	UK	2	10 - 14	0.007 - 0.033	200 - 900	0.100	14	0.200
Pepper	Italy	3	7-10	0.007-0.01		0.067-0.1	3	
Pome fruit	Belgium	3	7-12	0.022	300	0.067	7	
Pome fruit[1]	France	4	8-14	0.01	1000	0.1	7	4
Pome fruit[1]	Greece	4	10-14	0.006-0.01	1000-1500	0.1	7	4
Pome fruit[1]	Italy	3	8-14	0.0067	1500	0.1	7	3
Pome fruit[1],[4]	Italy	1-2	7-14	0.0033	1500	0.1	7	1-2
Pome fruit[1]	Netherlands	4	7-12	0.007-0.01	1000-1500	0.1	7	4
Pome fruit[1]	UK	4	10-14	0.007-0.067	300-1500	0.1	7	4
Root vegetables[5]	UK	2				0.100	21	0.200
Solanaceae (Tomato)	Poland	3	7 - 10	0.008 - 0.017	600 - 800	0.067– 0.10	3	0.200 – 0.300
Tomato	Italy							
Spelt[6]	Belgium	2				0.027	2	
	Luxemburg	2				0.027	2	
Stonefruit[7]	Hungary	2 - 3	7 - 10	0.017	400	0.05-0.07	7	0.200
Stonefruit[7]	Italy	3	7 - 14			0.04-0.06	3	0.200
Strawberry[8]	Belgium	2	7 - 10	0.012	1000	0.120	3	0.240
Strawberry	Cyprus	2	8 - 12	0.012	1000	0.120	3	0.240
Strawberry	Netherlands	4	7	0.020 -0.04	300-600	0.120	3	0.480
Strawberry[9]	Poland	2		0.024	1200– 500	0.120	3	0.480
Strawberry (outdoor)	UK	2	7 - 10	0.006 - 0.012	1000 - 2000	0.120	3	0.240
Strawberry (protected)[10]	UK	2				0.120	3	0.240
Vining peas[6]	France	2				0.1	35	2

1. WG formulation containing 12.8% active ingredient in combination with boscalid 25.2%
2. Outdoor crops of kale, collards (including spring greens), Chinese cabbage, leafy brassica crops grown for baby leaf production (i.e. crops harvested up to 8 true leaf stage), pak choi and komatsuna
3. Leaf herbs (outdoor and protected), leafy brassica crops (protected) grown for baby leaf production (i.e. harvested up to 8 true leaf stage)
4. Storage disease control
5. Parsnip, horseradish; both outdoor
6. SE formulation with 133g ai/L
7. Plums cherries, apricots, peaches, nectarines
8. Both F and G
9. Using pressure sprayer with field beam; 500 – 700 L/ha using beam “Fragraria III” or fan sprayer.
10. G

Table 2. Registered uses of pyraclostrobin outside of Europe.

Crop	Country	Formulation		Application					PHI day
		Type	Conc.	No	Interval	Rate kg/hL	Water L/ha	Rate kg ai/ha	
Apple	Brazil	EC	250g/L	4	7-14	0.010	1000	0.100	14
Beans, dry	Canada	EC	250 g/L	2	10-14	0.1	100	0.1	
Beans, dry	USA	EG	20%	2	7-14			0.169	21
Bell pepper	USA	WG	20%	6	7-14			0.224	0
Blackberry	USA	WG	20%	4	7-14			0.196	0
Blueberry	USA	WG	20%	4	7-14			0.196	0
Brassica head and	USA	WG	20%	4	7-14			0.21-0.28	0

Crop	Country	Formulation		Application					PHI day
		Type	Conc.	No	Interval	Rate kg/hL	Water L/ha	Rate kg ai/ha	
stem vegetables									
Brassica leafy vegetables	USA	WG	20%	4	7-10			0.21-0.28	3
Cantaloupe	USA	WG	20%	4	7-10			0.224	0
Chilli pepper	USA	WG	20%	6	7-14			0.224	0
Coffee	Brazil	EC	250g/L	2	60	0.040	500	0.15-0.2	45
Coffee	Brazil	SE	133g/L	2	90	0.040	500	0.2	45
Cucumber	USA	WG	20%	4	7-14			0.224	0
Cucumber	Brazil	EC	250g/L	4	7-14	0.01	1000	0.100	7
Cucurbits	Canada	WG	20%	4	7-14	0.05-0.07	350	0.112-0.168	3
Cucurbits	USA	EG	20%		7-14			0.168	3
Eggplant	USA	WG	20%	6	7-14			0.224	0
Fruiting vegetables	Canada	WG	20%	6	7-14	0.05-0.08	225	0.112-0.2	0
Fruiting vegetables	USA	EG	20%	6	7-		225	0.2	0
Leafy vegetables except brassicas	USA	EG	20%	4	7-14			0.17-0.23	0
Leek	USA	WG	20%	6	14			0.168	7
Pepper red	Korea	WG	6.3%	3	10	0.006	1500	0.095	7
Pepper red	Brazil	EC	250g/L	3	10	0.01	500-1000	0.1	3
Pome fruit	USA	WG	20%	4	7-10			0.16-0.21	0
Raspberry	USA	WG	20%	4	7-14			0.196	0
Snap beans	USA	WG	20%	2	7-14			0.087-0.13	7
Soybean	Argentina	EC	250g/L	1		0.025	200	0.050	15
Soybean	Brazil	EC	250g/L	2	15	0.03	300	0.09-0.1	14
Soybean[1]	USA	EC	250g/L	2	7-21			0.21-0.42	21
Stone fruits	Canada	WG	20%	5	7-14	0.013	1000	0.134	10
Stone fruits	USA	WG	20%	5	7-14			0.134	0
Sunflower	USA	EC	250g/L	2	7-14			0.21-0.42	21
Tomato	USA	WG	20%	6	7-14			0.224	0
Tomato	USA	WG	6.3%	5	7-14			0.36	4

1. Do not feed forage before 14 days after application.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The composition of the formulations which were used in the residue trials (Regenstein 2006/1009448) are described in Table 3.

Table 3. Formulations used in residue trials.

BAS-Code	Type	ai	Other active substances		Use
BAS 500 00 F	EC	250 g/L	none		Grapes
BAS 500 01 F	EC	250 g/L	none		Cereals
BAS 500 02 F	WG	20%	none		
BAS 512 00 F	SE	133 g/L	epoxiconazole	50 g/L	Cereals
BAS 513 00 F	SE	133 g/L	Epoxiconazole; kresoxim-methyl	50 g/L 67 g/L	Cereals
BAS 518 01 F	WG	5%	metiram	55%	Grapes and vegetables
BAS 516 00 F	WG	6.7%	boscalid	26.7%	Fruit and vegetables
BAS 516 01 F	SE	100 g/L	boscalid	200 g/L	
BAS 516 04 F	WG	12.8%	boscalid	25.3%	
BAS 516 GA F	WG	6.7%	boscalid	50 g/L	
BAS 518 00 F	WG	5%	metiram	55%	Grapes
BAS 528 00 F	EC	100 g/L	fenpropimorph	375 g/L	Cereals
BAS 529 00 F	SE	114.3 g/L	fenpropimorph,	214 g/L	Cereals

BAS-Code	Type	ai	Other active substances		Use
			epoxiconazole	43 g/L	
BAS 531 00 F	SE	100 g/L	fenpropimorph, epoxiconazole, quinoxyfen	187.5 g/L, 37.5 g/L, 37.5 g/L	Cereals
BAS 533 00 F	SE	133 g/L	epoxiconazole, quinoxyfen	50 g/L, 50 g/L	Cereals
BAS 536 00 F	WG	6.7%	dimethomorph	12%	Grapes[1]
BAS 537 00 F	SE	40 g/L	folpet	400 g/L	Grapes

1. No longer supported by BASF

The meeting reviewed information on supervised trials for the following crops:

Table No(s)	Crop	Page
4,5	Apple	4
8	Broccoli	10
9	Brussels sprouts	12
10	Cabbage, head	14
13	Cantaloupe	17
8	Cauliflower	10
28	Coffee	31
11,12	Cucumber	16
29	Hops	31
19	Kale	23
7	Leek	8
20-22	Lettuce, head	23
14, 15,17	Pepper	18
6	Raspberry	8
23	Snap beans	26
25, 26	Soybean	28
27	Sunflower	30
16, 18	Tomato	18
24	Vining peas	27

The samples were analysed with analytical methods based on LC/MS/MS detection providing an LOQ of 0.02 mg/kg (Jones J., 2001, D9908, Benz A. 2000BASF 445/0). The methods are described in detail in the 2004 Evaluations (FAO 2005). The applicability of the methods was confirmed with concurrent recovery tests in each study. The average recoveries were typically between 80 and 99% for pyraclostrobin and 500M07.

No interference of plant matrices was observed in most of the studies. Where low levels of apparent residues were detected, they were taken into consideration.

The storage intervals of samples from sampling to analysis were within the period covered by the storage stability tests reported by the 2004 JMPR.

The total residue was calculated as the sum of the parent pyraclostrobin and the major metabolite 500M07 [BF500-3, methyl-N-[[[1-(4-chlorophenyl)-pyrazol-3-yl]oxy]-o-tolyl]carbamate] and expressed as parent pyraclostrobin in this evaluation.

Apple

During the 2000 and 2001 growing seasons, four studies (Raunft E., BASF 2001/1006135 2001/1015029, and Schulz H., BASF2001/1000946 and 2001/1015046) with a total of 18 field trials were conducted in different representative apple growing areas in Belgium, Germany, France, Italy and the Netherlands. Four applications were made about 5, 4, 3 and 2 weeks before commercial

harvest of the crop in each trial at a rate of 1.0 L/ha in a spray volume of 1000 L/ha. Apple fruits were taken directly after the last application (day 0) as well as about 1, 2, 3 and 4 weeks thereafter.

During the 2003 growing season, one bridging study (Schulz H., BASF 2003/1001291) with another 4 field trials was conducted in the representative apple growing areas in Germany, Northern and Southern France and Italy. The BAS 516 01 F (100 g/L pyraclostrobin, 200 g/L boscalid, SE) and BAS 516 04 F (12.8% pyraclostrobin, 25.2% boscalid, WG) were compared, both with four applications at growth stages BBCH 74-78, 75-81, 76-85 and 77-87. In both variants, the application rates were about 100 g ai/ha of pyraclostrobin for all treatments and the spray volumes were 1000 L/ha.

The results are summarized in Table 4.

Table 4. Residues in apples derived from supervised trials carried out with BAS 516 01 F.

CROP	Application				Residues [mg/kg]			Ref. Report No
Country/ year trial code	No.	kg ai/ha	kg ai/hL	Day	Parent	500M07	Total	
GAP in European countries: 3 × 0.067-0.1 kg ai/ha, PHI=7 days								
Belgium	4	0.100	0.01	0	0.134	< 0.02	0.154	#2001/
2001				6	0.118	< 0.02	0.138	1015029
(AGR/15/01)				13	0.063	< 0.02	0.083	
				22	0.083	< 0.02	0.103	
				27	0.029	< 0.02	0.049	
Germany	4	0.100	0.01	0	0.087	0.026	0.113	#2001/
2000				6	0.034	< 0.02	0.054	1006135
(ACK/06/00)				14	0.022	< 0.02	0.042	
				21	0.024	< 0.02	0.044	
				28	< 0.02	< 0.02	< 0.04	
Germany	4	0.100	0.01	0	0.111	0.022	0.133	#2001/
2000				7	0.081	0.034	0.115	1006135
(DU2/12/00)				14	0.034	< 0.02	0.054	
				21	0.034	< 0.02	0.054	
				28	0.031	< 0.02	0.051	
Germany	4	0.100	0.01	0	0.115	< 0.02	0.135	#2001/
2000				7	0.058	< 0.02	0.078	1006135
(DU4/11/00)				14	0.041	< 0.02	0.061	
				21	0.032	< 0.02	0.052	
				28	0.034	< 0.02	0.054	
Germany	4	0.100	0.01	0	0.265	0.03	0.297	#2001/
2001				7	0.131	0.03	0.162	1015029
(DU2/07/01)				14	0.124	0.04	0.161	
				21	0.082	0.03	0.111	
				27	0.091	0.03	0.120	
France	4	0.100	0.01	0	0.182	< 0.02	0.202	#2001/
2000				6	0.095	< 0.02	0.115	1000946
(X006203)				13	0.085	0.023	0.108	
				21	0.051	< 0.02	0.071	
				28	0.036	< 0.02	0.056	
France	4	0.100	0.01	0	0.205	0.037	0.242	#2001/
2000				7	0.163	0.053	0.216	1000946
(X006204)				15	0.088	0.040	0.129	
				22	0.117	0.034	0.151	
				28	0.113	0.038	0.151	

CROP	Application				Residues [mg/kg]			Ref. Report No
Country/ year trial code	No.	kg ai/ha	kg ai/hL	Day	Parent	500M07	Total	
France 2000 (X006205)	4	0.100	0.01	0 7 14 21 28	0.205 0.256 0.290 0.200 0.176	< 0.02 0.025 0.038 0.022 0.025	0.225 0.281 0.328 0.222 0.202	#2001/ 1000946
France 2000 (X006206)	4	0.100	0.01	0 7 14 21 28	0.208 0.142 0.143 0.084 0.075	0.028 0.033 0.034 < 0.02 < 0.02	0.236 0.175 0.177 0.104 0.095	#2001/ 1000946
F - France 2001 (FBM/02/01)	4	0.100	0.01	0 8 14 20 28	0.124 0.070 0.050 0.042 0.030	< 0.02 0.04 0.03 0.03 < 0.02	0.144 0.107 0.078 0.073 0.050	#2001/ 1015029
France 2001 (X 01 062 08)	4	0.100	0.01	0 7 14 21 28	0.233 0.143 0.068 0.058 0.063	< 0.02 0.025 < 0.02 < 0.02 < 0.02	0.253 0.170 0.088 0.078 0.083	#2001/ 1015046
France 2001 (X 01 062 09)	4	0.100	0.01	0 7 14 21 28	0.227 0.120 0.091 0.061 0.039	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.247 0.140 0.111 0.081 0.059	#2001/ 1015046
Italy 2000 (0025R)	4	0.100	0.01	0 7 13 20 27	0.118 0.064 0.024 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.138 0.084 0.044 < 0.04 < 0.04	#2001/ 1000946
Italy 2000 (0026R)	4	0.100	0.01	0 8 14 22 28	0.124 0.066 0.070 0.043 0.036	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.144 0.086 0.090 0.063 0.056	#2001/ 1000946
Italy 2001 (0148R)	4	0.100	0.01	0 7 14 21 27	0.036 0.041 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.056 0.061 < 0.04 < 0.04 < 0.04	#2001/ 1015046
Italy 2001 (0149R)	4	0.100	0.01	0 7 14 21 28	0.107 0.070 0.060 0.070 0.046	< 0.02 0.021 < 0.02 < 0.02 < 0.02	0.127 0.092 0.080 0.090 0.066	#2001/ 1015046
Italy 2001 (0150R)	4	0.100	0.01	0 6 13 20 27	0.069 0.030 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.089 0.050 < 0.04 < 0.04 < 0.04	#2001/ 1015046
Netherlands 2001 (AGR/16/01)	4	0.100	0.01	0 8 13 21 29	0.106 0.101 0.066 0.064 0.039	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.126 0.121 0.086 0.084 0.059	#2001/ 1015029

CROP	Application				Residues [mg/kg]			Ref. Report No
Country/ year trial code	No.	kg ai/ha	kg ai/hL	Day	Parent	500M07	Total	
Germany	4	0.100	0.01	0	0.133	0.025	0.158	#2003/
2003				8	0.057	0.029	0.086	1001291
(ACK/11/03)				15	< 0.02	< 0.02	< 0.04	
				21	< 0.02	< 0.02	< 0.04	
				28	< 0.02	< 0.02	< 0.04	
	4[a]	0.100	0.01	0	0.098	< 0.02	0.120	
				8	0.051	< 0.02	0.073	
				15	0.035	< 0.02	0.057	
				21	0.025	< 0.02	0.047	
				28	< 0.02	< 0.02	< 0.04	
France	4	0.100	0.01	0	0.123	< 0.02	0.145	#2003/
2003				8	0.139	< 0.02	0.161	1001291
(FAN/18/03)				15	0.116	< 0.02	0.138	
				22	0.093	< 0.02	0.115	
				29	0.061	< 0.02	0.083	
	4[a]	0.100	0.01	0	0.201	< 0.02	0.223	
				8	0.104	< 0.02	0.126	
				15	0.074	< 0.02	0.096	
				22	0.097	< 0.02	0.119	
				29	0.051	< 0.02	0.073	
France	4	0.100	0.01	0	0.358	0.023	0.381	#2003/
2003				7	0.289	0.037	0.326	1001291
(FTL/15/03)				14	0.191	0.023	0.214	
				21	0.159	0.023	0.182	
				28	0.222	0.036	0.258	
	4[a]	0.100	0.01	0	0.373	< 0.02	0.395	
				7	0.276	0.028	0.304	
				14	0.234	0.029	0.263	
				21	0.191	0.031	0.222	
				28	0.165	0.025	0.190	
Italy	4	0.100	0.01	0	0.184	< 0.02	0.206	#2003/
2003				7	0.167	0.023	0.190	1001291
(ITA/09/03)				15	0.142	0.030	0.172	
				21	0.129	0.024	0.153	
				28	0.068	< 0.02	0.090	
	4[a]	0.100	0.01	0	0.222	< 0.02	0.244	
				7	0.184	< 0.02	0.206	
				15	0.066	< 0.02	0.088	
				21	0.081	< 0.02	0.103	
				28	0.074	< 0.02	0.096	

(a) BAS 516 04 F was used

The 2004 JMPR reported the results of supervised trials carried out in Brazil which are copied into Table 5.

Table 5. Pyraclostrobin residues in apple derived from supervised trials in Brazil (reported by the 2004 JMPR).

	Appl. per treatment						Residues [mg/kg]				Trials
Location	kg ai/ha	Water L/ha	kg ai/hL	No of tr.	Growth stage	Portion analysed	Parent	500M07	Total	PHI days	method
Brazilian GAP: 4 × 0.1 kg ai/ha, PHI =14days											
BR/Santagro	0.150	1000	0.015	4	72	apples	0.34	0.08	0.42	14	#2000/5241
2000/049	0.300	1000	0.030	03/02/00			1.00	0.19	1.19	14	D9908

	Appl. per treatment						Residues [mg/kg]				Trials
Location	kg ai/ha	Water L/ha	kg ai/hL	No of tr.	Growth stage	Portion analysed	Parent	500M07	Total	PHI days	method
BR/Santagro 2000/905	0.150	1000	0.015	4 03/02/00	72	apples	0.35	0.09	0.44	14	#2000/5241 D9908
	0.300	1000	0.030			apples	0.93	0.21	1.14	14	
BR Fitopesquisa 2000/050	0.150	1000	0.015	4 10/03/00	ripening	apples	0.16	0.02	0.18	0	#2000/5241 D9908
							0.19	0.05	0.24	7	
							0.15	0.03	0.18	14	
BR Fitopesquisa 2000/050							0.11	< 0.02	0.13	21	
							0.04	< 0.02	0.06	28	
							< 0.02	< 0.02	< 0.04	35	
BR/BR5 2000/051	0.150	1000	0.015	4 18/02/00	coloured fruits	apples	0.11	0.04	0.16	14	#2000/5241 D9908
	0.300	1000	0.030				0.30	0.11	0.41	14	
BR/BR5 2000/052	0.150	1000	0.015	4 18/02/00	coloured fruits	apples	0.06	0.02	0.08	14	#2001/500242 D9908
	0.300	1000	0.030				0.09	0.03	0.12	14	
BR Fitopesquisa CDR/F 2000/053	0.150	1000	0.015	4 10/03/00	ripening	apples	< 0.02	< 0.02	< 0.04	0	#2001/500242 D9908
							0.12	< 0.02	0.14	7	
							0.14	< 0.02	0.16	14	
							0.03	< 0.02	0.05	21	
							0.04	< 0.02	0.06	28	
							0.04	< 0.02	0.06	35	
BR Santagro 2000/054	0.150	1000	0.015	4 03/02/00	87	apple	0.38	0.05	0.43	14	#2001/5002427 D9908
	0.300	1000	0.030				0.94	0.07	1.01	14	
BR Santagro 2000/906	0.150	1000	0.015	4 03/02/00	87	apple	0.25	0.05	0.30	14	#2001/5002427 D9908
	0.300	1000	0.030				0.57	0.08	0.65	14	

Raspberry

During the 1999 and 2004 growing seasons five trials were performed in USA and one in Canada (Versoi P.L., *et al*. BASF 1999/5143, Leonard R. and Gooding R. BASF 2005/5000144). In each trial four broadcast foliar applications were made 6-7 days apart with a WG formulation containing 12.8% pyraclostrobin using 267–798 L/ha water. An adjuvant was added to all spray solutions for all applications. The samples of mature fruits were collected on the day of last application (day 0). The maximum storage interval for the samples was 182 days.

The trial conditions and results are given in Table 6.

Table 6. Pyraclostrobin residues in raspberries from supervised trials in USA.

	Appl. per treatment[1]					Residues[2] [mg/kg]				Trial number
Location	kg ai/ha	Water L/ha	kg ai/hL	No of tr[2].	Portion analyzed	Parent	500M07	Total	PHI days	Method
US GAP: 4 times 0.16-0.21 kg ai/ha with 0-day PHI										
Penn Yau. Yates Co.. New York (RCN 99277)[3]	0.20	570	0.035	4	Mature fruit	0.78	0.03	0.81	0	# 1999/5143 421/0
						0.53	0.02	0.55	2	
						0.52	0.03	0.55	4	
						0.41	0.03	0.44	6	
						0.30	< 0.02	0.32	8	
Oregon Washington[3]	0.20	546	0.037	4	Mature fruit	0.50	< 0.02	0.52	0	# 1999/5143 421/0
Oregon Washington[3]	0.20	522	0.038	4	Mature fruit	0.63	0.03	0.66	0	# 1999/5143 421/0
Nodine, MN RCN 20044143	0.21	565-575	0.037	4	Mature fruit	1.18, 1.28	0.05, 0.48	1.23, 1.33	0	2005/5000144/ D9908
Corvallis, OR RCN 20044144	0.21	702	0.03	4	Mature fruit	0.82, 0.89	0.051, 0.049	0.87, 0.94	0	2005/5000144/ D9908

	Appl. per treatment[1]					Residues[2] [mg/kg]				Trial number
Location	kg ai/ha	Water L/ha	kg ai/hL	No of tr[2].	Portion analyzed	Parent	500M07	Total	PHI days	Method
US GAP: 4 times 0.16-0.21 kg ai/ha with 0-day PHI										
Abbotsford, QC Canada RCN 20044145	0.19-0.21	606-798	0.026-0.032	4	Mature fruit	0.73, 1.03	0.035, 0.051	0.76, 1.08	0	2005/5000144/ D9908
Yates, NY 99277	0.2			4	Mature fruit	0.94, 0.62	0.03, < 0.02	0.97, 0.64	0	1999/5143/ D9808
Washington, OR 99280	0.2			4	Mature fruit	0.51, 0.44	< 0.02, < 0.02	0.53, 0.46	0	1999/5143/ D9808
Washington, OR 99281	0.2			4	Mature fruit	0.78, 0.47	0.04, < 0.02	0.82, 0.49	0	1999/5143/ D9808

1. Application rates and spray volumes are rounded
2. Treatments were made at intervals of 6-7 days
3. Trials reported by the 2004 JMPR

Leek

During the 1999 and 2003 growing seasons, three studies with a total of 11 field trials on five varieties of leek were conducted in different representative growing areas in Belgium, Germany, Great Britain, France and The Netherlands (Raunft E. BASF 2001/1006130 and BASF 2001/1006131, Schulz H. BASF 2004/1015937). The applications were done about 5, 3 and 2 weeks before commercial harvest of the crop and the intended PHI was 14 days. For the analysis, plants without roots were sampled immediately after the last application as well as about 7, 14 and 21 days thereafter.

The results are summarized in Table 7.

Table 7. Results of supervised trials performed on leek[1] with BAS 516 GA F containing 6.7% pyraclostrobin.

CROP	Application			Residues [mg/kg]				Ref. Report No
Country/ year trial code	No.	kg ai/ha	kg ai/hL	Day	Parent	500M07	Total	
GAP (The Netherlands and Belgium): 2-3 × 0.1 kg ai/ha with a PHI of 14 days								
Belgium 1999 (AGR/19/99)	3	0.100	0.025	0	1.04	< 0.02	1.06	#2001/ 1006130
				7	0.41	< 0.02	0.43	
				14	0.24	< 0.02	0.26	
				21	0.23	< 0.02	0.25	
Belgium 2000 (AGR/08/00)	3	0.100	0.025	0	1.15	0.02	1.17	#2001/ 1006131
				7	0.26	< 0.02	0.28	
				14	0.18	< 0.02	0.20	
				20	0.19	< 0.02	0.21	
Germany 1999 (ACK/09/99)	3	0.100	0.025	0	0.51	< 0.02	0.53	#2001/ 1006130
				7	0.31	< 0.02	0.33	
				14	0.25	< 0.02	0.27	
				20	0.14	< 0.02	0.16	
Germany 1999 (DU2/14/99)	3	0.100	0.025	0	0.98	0.04	1.02	#2001/ 1006130
				7	0.59	0.05	0.64	
				14	0.42	0.04	0.46	
				21	0.26	0.02	0.28	
Germany 2000 (DU2/09/00)	3	0.100	0.025	0	0.90	0.03	0.93	#2001/ 1006131
				7	0.24	< 0.02	0.26	
				14	0.22	< 0.02	0.24	
				21	0.15	< 0.02	0.17	

CROP	Application			Residues [mg/kg]				Ref. Report No
Country/ year trial code	No.	kg ai/ha	kg ai/hL	Day	Parent	500M07	Total	
Germany	3	0.100	0.025	0	0.60	< 0.02	0.62	#2001/
2000				7	0.31	< 0.02	0.33	1006131
(DU4/08/00)				14	0.22	< 0.02	0.24	
				21	0.17	< 0.02	0.19	
Great Britain	3	0.100	0.025	0	0.68	< 0.02	0.70	#2001/
2000				7	0.18	< 0.02	0.20	1006131
(OAT/10/00)				14	0.12	< 0.02	0.14	
				21	0.09	< 0.02	0.11	
The Netherlands	3	0.100	0.025	0	1.04	< 0.02	1.06	#2001/
1999				7	0.55	< 0.02	0.57	1006130
(AGR/18/99)				14	0.24	< 0.02	0.26	
				21	0.29	< 0.02	0.31	
The Netherlands	3	0.100	0.025	0	0.53	< 0.02	0.55	#2001/
2000				7	0.23	< 0.02	0.25	1006131
(AGR/09/00)				13	0.16	< 0.02	0.18	
				20	< 0.02	< 0.02	< 0.04	
France	3	0.100	0.025	0	0.81	0.02	0.83	#2004/
2003				8	0.10	< 0.02	0.12	1015937
(FAN/12/03)				14	0.05	< 0.02	0.07	
				21	0.02	< 0.02	0.04	
France	3	0.100	0.025	0	0.60	0.03	0.63	#2004/
2003				7	0.25	0.04	0.29	1015937
(FBM/06/03)				14	0.15	0.03	0.18	
				20	0.16	0.02	0.18	

Brassica vegetables

Broccoli and cauliflower

During the 2003 and 2004 growing seasons, two studies (Schulz H., BASF 2004/1015910, Johnston R. L., BASF 2004/7007476) with a total of 13 field trials were conducted in different representative cauliflower and broccoli growing areas in Europe. Eleven trials were performed in the Northern EU (Great Britain, Netherlands, Denmark, Germany, Sweden and France) and two trials in the Southern EU (France).

The WG formulation BAS 516 00F was applied three times at a rate of 1.0 kg formulated product/ha, resulting in a dosage of 0.067 kg ai/ha of pyraclostrobin. The applications took place at about 28, 21 and 14 days before harvest. The intended PHI was 14 days. The product was applied with a spray volume of 300 L/ha.

In all trials, samples were taken directly after the last application (day 0) as well as about 7, 14 and 21 days thereafter.

During the 1999 and 2000 growing seasons, a total of 11 field trials (Beck J., BASF 2001/1001001 and BASF 2001/1001000, Funk H., BASF 2001/1009065 and 2001/1009066, Schulz H., BASF 2001/1000932) were conducted in different representative brassica growing areas in Germany, Denmark, France, Great Britain, the Netherlands and Sweden. The BAS 516 GA F was tested in cauliflower and broccoli using four applications at 1.5 kg product/ha each in a spray volume of 300 L/ha. The applications were done about 5, 4, 3 and 2 weeks before commercial harvest of the crop. The samples were taken directly after the last application (day 0) as well as about 1, 2, and 3 weeks thereafter. The mix formulation BAS 516 GA F was replaced by the formulations BAS 516 00 F and BAS 516 07 F later on in the development phase.

The results are summarized in Table 8

Table 8. Summary of results of field trials carried out with BAS 516 on flowering brassica.

CROP	Application					Residues [mg/kg]			Ref. Report No
Country/ year trial code	Formulation	No.	kg ai/ha	kg ai/hL	Day	Parent	500M07	Total	
CAULIFLOWER (GAP: 2-3 × 0.067 kg ai/ha with a PHI of 14 days)									
Netherlands	BAS 516 00 F	3	0.067	0.022	0	< 0.02	0.02	0.04	#2004/
2003					8	< 0.02	< 0.02	< 0.04	1015910
(AGR/26/03)					15	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
Denmark	BAS 516 00 F	3	0.067	0.022	0	0.02	< 0.02	0.04	#2004/
2003					7	< 0.02	< 0.02	< 0.04	1015910
(ALB/16/03)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
England	BAS 516 00 F	3	0.067	0.022	0	0.24	< 0.02	0.26	#2004/
2003					7	0.10	< 0.02	0.12	1015910
(OAT/22/03)					13	0.04	< 0.02	0.06	
					20	0.04	< 0.02	0.06	
France	BAS 516 00 F	3	0.067	0.022	0	< 0.02	< 0.02	< 0.04	#2004/
2003					6	< 0.02	< 0.02	< 0.04	1015910
(FBD/15/03)					13	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
France	BAS 516 00 F	3	0.067	0.022	0	0.11	< 0.02	0.13	#2004/
2004					7	< 0.02	< 0.02	< 0.04	7007476
(FAN/09/04)					15	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
Germany	BAS 516 00 F	3	0.067	0.022	0	< 0.02	< 0.02	< 0.04	#2004/
2004					7	< 0.02	< 0.02	< 0.04	7007476
(DU4/04/04)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
France	BAS 516 00 F	3	0.067	0.022	0	< 0.02	< 0.02	< 0.04	#2004/
2004					6	< 0.02	< 0.02	< 0.04	7007476
(FTL/11/04)					13	< 0.02	< 0.02	< 0.04	
					20	< 0.02	< 0.02	< 0.04	
Germany	BAS 516 GA F	4	0.100	0.033	0	0.07	< 0.02	0.09	#2001/
1999					6	< 0.02	< 0.02	< 0.04	1001001
(DU4/05/99)					13	< 0.02	< 0.02	< 0.04	
					20	< 0.02	< 0.02	< 0.04	
France	BAS 516 GA F	4	0.100	0.033	0	0.34	< 0.02	0.36	#2001/
2000					7	< 0.02	< 0.02	< 0.04	1009065
(F00W027R)					14	< 0.02	< 0.02	< 0.04	
					22	< 0.02	< 0.02	< 0.04	
France	BAS 516 GA F	4	0.100	0.033	0	< 0.02	< 0.02	< 0.04	#2001/
2000					7	< 0.02	< 0.02	< 0.04	1009065
(F00W031R)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
Great Britain	BAS 516 GA F	4	0.100	0.033	0	0.16	< 0.02	0.18	#2001/
1999					7	< 0.02	< 0.02	< 0.04	1001001
(OAT/16/99)					14	< 0.02	< 0.02	< 0.04	
					20	< 0.02	< 0.02	< 0.04	
Netherlands	BAS 516 GA F	4	0.100	0.033	0	0.04	< 0.02	0.06	#2001/
2000					7	0.04	< 0.02	0.06	1001000
(AGR/05/00)					13	0.04	0.03	0.07	
					20	0.05	0.04	0.09	
Sweden	BAS 516 GA F	4	0.100	0.033	0	1.30	0.03	1.32	#2001/
2000					7	0.04	< 0.02	0.06	1001000
(HUS/07/00)					15	< 0.02	< 0.02	< 0.04	

CROP	Application					Residues [mg/kg]			Ref. Report No
Country/ year trial code	Formulation	No.	kg ai/ha	kg ai/hL	Day	Parent	500M07	Total	
					21	< 0.02	< 0.02	< 0.04	
BROCCOLI (GAP: 3 × 0.067 kg ai/ha with a PHI of 14 days)									
Germany 2003 (ACK/17/03)	BAS 516 00 F	3	0.067	0.022	0 7 14 21	0.27 0.05 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.29 0.07 < 0.04 < 0.04	#2004/ 1015910
France 2003 (FAN/24/03)	BAS 516 00 F	3	0.067	0.022	0 7 14 20 28	0.83 0.14 < 0.02 < 0.02 < 0.02	< 0.02 0.03 < 0.02 < 0.02 < 0.02	0.85 0.17 < 0.04 < 0.04 < 0.04	#2004/ 1015910
France 2003 (FTL/19/03)	BAS 516 00 F	3	0.067	0.022	0 7 15 22	0.13 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.15 0.04 < 0.04 < 0.04	#2004/ 1015910
Sweden 2004 (HUS/02/04)	BAS 516 00 F	3	0.067	0.022	0 7 14 21	0.28 0.06 < 0.02 0.06	< 0.02 < 0.02 < 0.02 < 0.02	0.30 0.08 < 0.04 0.08	#2004/ 7007476
Denmark 2004 (ALB/05/04)	BAS 516 00 F	3	0.067	0.022	0 8 15 23	0.21 0.10 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.23 0.12 0.04 < 0.04	#2004/ 7007476
France 2004 (FBD/10/04)	BAS 516 00 F	3	0.067	0.022	0 7 14 21	0.18 0.05 0.03 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.20 0.07 0.05 0.04	#2004/ 7007476
Denmark 1999 (ALB/10/99)	BAS 516 GA F	4	0.100	0.033	0 7 14 21	1.72 0.08 < 0.02 < 0.02	0.08 < 0.02 < 0.02 < 0.02	1.80 0.10 < 0.04 < 0.04	#2001/ 1001001
France 1999 (X 99 62 01)	BAS 516 GA F	4	0.100	0.033	0 7 13 20	0.72 0.09 0.04 0.03	< 0.02 < 0.02 < 0.02 < 0.02	0.74 0.11 0.06 0.05	#2001/ 1000932
France 2000 (F00W034R)	BAS 516 GA F	4	0.100	0.033	0 7 14 21	0.60 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.62 0.04 < 0.04 < 0.04	#2001/ 1009066
Great Britain 2000 (OAT/20/00)	BAS 516 GA F	4	0.100	0.033	0 8 14 21	1.65 0.31 0.18 0.11	0.03 0.03 < 0.02 < 0.02	1.68 0.34 0.20 0.13	#2001/ 1001000
The Netherlands 1999 (AGR/08/99)	BAS 516 GA F	4	0.100	0.033	0 8 14 21	0.52 0.05 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.54 0.07 < 0.04 < 0.04	#2001/ 1001001

Brussels sprouts

During the 2003 and 2004 growing seasons, three studies (Schulz H., BASF 2004/1015912, Johnston R.L., BASF 2004/7007478 and 2004/7007477) with a total of nine field trials were conducted in Brussels sprouts. The trials were performed in Great Britain, Netherlands, Denmark, Germany, Sweden and France.

The WG formulation BAS 516 00 F was applied three times with an application rate of 1.0 kg formulated product/ha, resulting in application rates of 0.067 kg ai/ha for pyraclostrobin. The applications took place at about 28, 21 and 14 days before harvest. The intended PHI was 14 days. The product was applied with a spray volume of 300 L/ha.

In all trials, samples were taken directly after the last application (day 0) as well as about 7, 14 and 21 days thereafter.

During the 1999 and 2000 growing seasons, two studies (Beck J., BASF 2001/1001001 and BASF 2001/1001000,) with a total of nine field trials was conducted in Brussels sprouts in Germany, Denmark, Great Britain, the Netherlands and Sweden. The BAS 516 GA F was applied four times with 1.5 kg product/ha each in a spray volume of 300 L/ha. The applications were done about 5, 4, 3 and 2 weeks before commercial harvest of the crop.

The results are summarized in Table 9.

Table 9. Residues derived from supervised field trials with BAS 516 00 F on Brussels sprouts.

CROP	Application			Residues [mg/kg]				Ref. Report No
Country/ year trial code	No.	kg ai/ha	kg ai/hL	Day	Parent	500M07	Total	
(GAP: 2-3 × 0.067 kg ai/ha with a PHI of 14 days)								
Germany 2003 (ACK/16/03)	3	0.067	0.022	0 8 13 20	0.05 0.07 0.05 <u>0.10</u>	< 0.02 < 0.02 < 0.02 < 0.02	0.07 0.09 0.07 0.12	#2004/ 1015912
The Netherlands 2003 (AGR/25/03)	3	0.067	0.022	0 6 13 20	0.09 0.10 0.05 <u>0.08</u>	< 0.02 < 0.02 < 0.02 < 0.02	0.11 0.12 0.07 0.10	#2004/ 1015912
Denmark 2003 (ALB/15/03)	3	0.067	0.022	0 7 14 21	0.10 0.03 <u>0.03</u> < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.12 0.05 0.05 < 0.04	#2004/ 1015912
Sweden 2003 (HUS/07/03)	3	0.067	0.022	0 8 15 22	0.05 < 0.02 <u>0.03</u> < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.07 < 0.04 0.05 < 0.04	#2004/ 1015912
England 2003 (OAT/19/03)	3	0.067	0.022	0 7 14 21	< 0.02 < 0.02 <u>< 0.02</u> < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	< 0.04 < 0.04 < 0.04 < 0.04	#2004/ 1015912
Germany 2004 (DU2/04/04)	3	0.067	0.022	0 7 14 21	0.22 0.17 <u>0.14</u> 0.09	< 0.02 < 0.02 < 0.02 < 0.02	0.24 0.19 0.16 0.11	#2004/ 7007478
France 2004 (FAN/08/04)	3	0.067	0.022	0 7 13 21	0.07 0.06 0.05 <u>0.06</u>	< 0.02 < 0.02 < 0.02 < 0.02	0.09 0.08 0.07 0.08	#2004/ 7007478
Sweden 2004 (HUS/01/04)	3	0.067	0.022	0 7 15 21	0.07 0.02 <u>< 0.02</u> < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.09 0.04 < 0.04 < 0.04	#2004/ 7007478

CROP	Application			Residues [mg/kg]				Ref. Report No
Country/ year trial code	No.	kg ai/ha	kg ai/hL	Day	Parent	500M07	Total	
England	3	0.067	0.022	0	0.05	< 0.02	0.07	#2004/
2004				7	0.03	< 0.02	0.05	7007477
(OAT/08/04)				13	< 0.02	< 0.02	< 0.04	
				21	< 0.02	< 0.02	< 0.04	
Germany	4	0.100	0.033	0	0.28	< 0.02	0.30	#2001/
2000				-	-	-	-	1001000
(DU4/10/00)				15	0.21	< 0.02	0.23	
				21	0.21	< 0.02	0.23	
Denmark	4	0.100	0.033	0	0.61	0.04	0.65	#2001/
1999				7	0.09	< 0.02	0.11	1001001
(ALB/11/99)				14	0.11	< 0.02	0.13	
				21	0.06	< 0.02	0.08	
Denmark	4	0.100	0.033	0	0.29	< 0.02	0.31	#2001/
2000				7	0.08	< 0.02	0.10	1001000
(ALB/06/00)				14	0.07	< 0.02	0.09	
				21	0.05	< 0.02	0.07	
Great Britain	4	0.100	0.033	0	0.15	< 0.02	0.17	#2001/
1999				7	0.12	< 0.02	0.14	1001001
(OAT/17/99)				14	0.06	< 0.02	0.08	
				21	0.08	< 0.02	0.10	
Great Britain	4	0.100	0.033	0	0.12	< 0.02	0.14	#2001/
1999				8	0.05	< 0.02	0.07	1001001
(OAT/18/99)				15	0.04	< 0.02	0.06	
				22	0.08	0.02	0.10	
Great Britain	4	0.100	0.033	0	0.16	< 0.02	0.18	#2001/
2000				7	0.07	< 0.02	0.09	1001000
(OAT/05/00)				13	0.06	< 0.02	0.08	
				22	0.05	< 0.02	0.07	
The Netherlands	4	0.100	0.033	0	0.29	< 0.02	0.31	#2001/
1999				7	0.19	< 0.02	0.21	1001001
(AGR/09/99)				15	0.13	< 0.02	0.15	
				22	0.08	< 0.02	0.10	
The Netherlands	4	0.100	0.033	0	0.20	< 0.02	0.22	#2001/
2000				7	0.19	< 0.02	0.21	1001000
(AGR/06/00)				14	0.13	< 0.02	0.15	
				21	0.09	< 0.02	0.11	
Sweden	4	0.100	0.033	0	0.14	< 0.02	0.16	#2001/
1999				7	0.12	< 0.02	0.14	1001001
(HUS/07/99)				14	0.12	< 0.02	0.14	
				21	0.11	< 0.02	0.13	

Cabbage

During the 2003 and 2004 growing seasons, two studies (Schulz H., BASF 2004/1015911, Johnston R.L., BASF 2004/7007477) with a total of 11 field trials were conducted in different representative head cabbage growing areas in the EU to determine the residue levels of pyraclostrobin. Nine trials were performed in the Northern EU (Germany, Sweden, Denmark, Great Britain, Netherlands and France) and two trials in the Southern EU (France).

The WG formulation BAS 516 00 F was applied three times with a rate of 1.0 kg formulated product/ha (0.067 kg ai/ha for pyraclostrobin). The applications took place at about 28, 21 and 14 days before harvest. The intended PHI was 14 days. The product was applied with a spray volume of 300 L/ha.

In all trials, samples were taken directly after the last application (0 day) as well as about 7, 14 and 21 days thereafter.

During the 1999 and 2000 growing seasons, five studies (Beck J, BASF 2001/1001000 and 2001/1001001 Schulz H., 2001/1000932 Funk H., BASF 2001/1000945 and 2001/1009064) with a total of 12 field trials were conducted in different representative brassica growing areas in Germany, Denmark, France, Great Britain, the Netherlands and Sweden to determine the residue levels of pyraclostrobin. The BAS 516 GA F was applied four times with 1.5 kg product/ha each in a spray volume of 300 L/ha. The applications were done about 5, 4, 3 and 2 weeks before commercial harvest of the crop.

The results are summarized in Table 10

Table 10. Summary of residues derived from field trials carried out with BAS 516 F on cabbages.

CROP	Application			Residues [mg/kg]					Ref. Report No
Country/ year trial code	No.	kg ai/ha	kg ai/hL	Matrix	Day	Parent	500M07	Total	
(GAP: 3 × 0.067 kg ai/ha with a PHI of 14 days)									
France	3	0.067	0.022	White	0	0.43	< 0.02	0.45	#2004/
2003				Cabbage	7	0.11	< 0.02	0.13	1015911
(FAN/23/03)					15	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
France	3	0.067	0.022	White	0	0.73	0.04	0.77	#2004/
2003				Cabbage	7	0.27	0.05	0.32	1015911
(FTL/18/03)					14	0.05	< 0.02	0.07	
					21	0.04	< 0.02	0.06	
England	3	0.067	0.022	White	0	0.03	< 0.02	0.05	#2004/
2004				Cabbage	7	< 0.02	< 0.02	< 0.04	7007477
(OAT/13/04)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
Sweden	3	0.067	0.022	White	0	< 0.02	< 0.02	< 0.04	#2004/
2004				Cabbage	6	< 0.02	< 0.02	< 0.04	7007477
(HUS/05/04)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
France	4	0.100	0.033	White	0	0.17	< 0.02	0.19	#2001/
1999				Cabbage	7	< 0.02	< 0.02	< 0.04	1000932
(X 99 62 02)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
France	4	0.100	0.033	White	0	0.38	< 0.02	0.40	#2001/
2000				Cabbage	7	0.06	< 0.02	0.08	1000945
(F00W033R)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
Great Britain	4	0.100	0.033	White	0	0.02	< 0.02	0.04	#2001/
1999				Cabbage	8	< 0.02	< 0.02	< 0.04	1001001
(OAT/20/99)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
The Netherlands	4	0.100	0.033	White	0	< 0.02	< 0.02	< 0.04	#2001/
1999				Cabbage	7	< 0.02	< 0.02	< 0.04	1001001
(AGR/10/99)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
Sweden	3	0.067	0.022	Red	0	0.06	< 0.02	0.08	#2004/
2003				Cabbage	7	< 0.02	< 0.02	< 0.04	1015911
(HUS/08/03)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	

CROP	Application			Residues [mg/kg]					Ref. Report No
Country/ year trial code	No.	kg ai/ha	kg ai/hL	Matrix	Day	Parent	500M07	Total	
France	3	0.067	0.022	Red	0	0.37	< 0.02	0.39	#2004/
2004				Cabbage	7	0.14	< 0.02	0.16	7007477
(FBM/09/04)					14	0.06	< 0.02	0.08	
					21	0.09	< 0.02	0.11	
France	3	0.067	0.022	Red	0	< 0.02	< 0.02	< 0.04	#2004/
2004				Cabbage	7	< 0.02	< 0.02	< 0.04	7007477
(FBD/15/04)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
Germany	4	0.100	0.033	Red	0	0.49	< 0.02	0.51	#2001/
1999				Cabbage	8	0.10	< 0.02	0.12	1001001
(DU4/06/99)					14	< 0.02	< 0.02	< 0.04	
					20	< 0.02	< 0.02	< 0.04	
France	4	0.100	0.033	Red	0	< 0.02	< 0.02	< 0.04	#2001/
2000				Cabbage	7	< 0.02	< 0.02	< 0.04	1009064
(F00W026R)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
France	4	0.100	0.033	Red	0	0.06	< 0.02	0.08	#2001/
2000				Cabbage	7	< 0.02	< 0.02	< 0.04	1009064
(F00W032R)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
Great Britain	4	0.100	0.033	Red	0	0.06	< 0.02	0.08	#2001/
2000				Cabbage	7	0.02	< 0.02	0.04	1001000
(OAT/06/00)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
Great Britain	4	0.100	0.033	Red	0	0.05	< 0.02	0.07	#2001/
2000				Cabbage	7	< 0.02	< 0.02	< 0.04	1001000
(OAT/07/00)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
Sweden	4	0.100	0.033	Red	0	< 0.02	< 0.02	< 0.04	#2001/
1999				Cabbage	8	< 0.02	< 0.02	< 0.04	1001001
(HUS/05/99)					15	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
Denmark	3	0.067	0.022	Savoy	0	0.28	< 0.02	0.30	#2004/
2003				Cabbage	7	0.10	< 0.02	0.12	1015911
(ALB/13/03)					14	0.09	< 0.02	0.11	
					21	0.02	< 0.02	0.04	
England	3	0.067	0.022	Savoy	0	0.16	< 0.02	0.18	#2004/
2003				Cabbage	7	0.07	< 0.02	0.09	1015911
(OAT/20/03)					14	0.04	< 0.02	0.06	
					21	0.05	< 0.02	0.07	
Germany	3	0.067	0.022	Savoy	0	0.13	< 0.02	0.15	#2004/
2004				Cabbage	8	0.03	< 0.02	0.05	7007477
(DU4/09/04)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	
The Netherlands	3	0.067	0.022	Savoy	0	0.12	< 0.02	0.14	#2004/
2004				Cabbage	7	< 0.02	< 0.02	< 0.04	7007477
(AGR/14/04)					14	< 0.02	< 0.02	< 0.04	
					21	< 0.02	< 0.02	< 0.04	

CROP	Application			Residues [mg/kg]					Ref. Report No
Country/ year trial code	No.	kg ai/ha	kg ai/hL	Matrix	Day	Parent	500M07	Total	
Denmark	4	0.100	0.033		0	0.59	< 0.02	0.61	#2001/
2000					6	0.23	< 0.02	0.25	1001000
(ALB/07/00)					13	0.07	< 0.02	0.09	
					20	0.03	< 0.02	0.05	
The Netherlands	4	0.100	0.033		0	0.70	< 0.02	0.72	#2001/
2000					7	0.20	< 0.02	0.22	1001000
(AGR/07/00)					13	0.06	< 0.02	0.08	
					20	0.08	< 0.02	0.10	

Fruiting vegetables

Cucumber

Supervised field trials were conducted at eight sites in USA (Wofford T. *et al*., BASF 1999/5083). Each plot received 6 sequential applications (7 ± 1 day apart) with 0.224 kg ai/ha and a total seasonal rate of 1.34 kg ai/ha according to the US GAP. Duplicate samples were collected from each site at day 0. In addition decline studies were performed at two sites. The results are given in Table 11.

The residues in cucumber from Brazilian trials reported by the 2004 JMPR are given in Table 12.

Table 11. Pyraclostrobin residues in cucumber resulting from supervised trials with BAS 500 F in USA.

Location/ trial code	Application		Residues (mg/kg)				Ref. Report No
	No.	kg ai/ha	Day	Parent	500M07	Total residue	
(GAP: 4 × 0.224 kg ai/ha with a PHI of 0 day)							
Macon County, GA, 98003	6	0.224	0	0.36, 0.41	< 0.02, < 0.02	0.38, 0.43	1999/5083/ D9908
Barnwell County, SC 98004	6	0.224	0	0.06, 0.05	< 0.02, < 0.02	0.08, 0.07	1999/5083/ D9908
Tift County, GA	6	0.224	0	< 0.02, 0.05	< 0.02, < 0.02	0.04, 0.07	1999/5083/
98005			3	< 0.02, < 0.02	< 0.02, < 0.02	0.04, 0.04	D9908
			7	< 0.02, < 0.02	< 0.02, < 0.02	0.04, 0.04	
			10	< 0.02, < 0.02	< 0.02, < 0.02	0.04, 0.04	
			15	< 0.02, < 0.02	< 0.02, < 0.02	0.04, 0.04	
Seminole County, FL 98008	6	0.224		0.09, 0.08	< 0.02, < 0.02	0.11, 0.10	1999/5083/ D9908
Ottawa County, MI 98010	6	0.224		0.03, 0.02	< 0.02, < 0.02	0.05, 0.04	0999/5083/ D9908
Pepin County, WI 98011	6	0.224		0.06, 0.07	< 0.02, < 0.02	0.08, 0.09	1999/5083/ D9908
Uvalde county, TX 98014	6	0.224		0.14, 0.11	< 0.02, < 0.02	0.16, 0.13	1999/5083/ D9908
Tulare county, CA	6	0.224	0	0.12, 0.09	< 0.02, < 0.02	0.14, 0.11	1999/5083/
98016			3	0.03, 0.03	< 0.02, < 0.02	0.05, 0.05	D9908
			7	< 0.02, < 0.02	< 0.02, < 0.02	0.04, 0.04	
			10	< 0.02, < 0.02	< 0.02, < 0.02	0.04, 0.04	
			15	< 0.02, < 0.02	< 0.02, < 0.02	0.04, 0.04	

Table 12. Pyraclostrobin residues in cucumber resulting from supervised trials in Brazil (Reported by the 2004 JMPR).

	Appl.. per treatment				Residues, [mg/kg]				Trial number
Location	kg ai/ha	Water (L/ha)	kg ai/hL	No of tr.	Parent	500M07	Total	PHI days (d)	Method
(GAP: 4 × 0.1 kg ai/ha with a PHI of 7 days)									
BR/BRV Tapuirama-2000/155/	0.100	400	0.025	4	< 0.02	0.02	< 0.04	7	#2001/5002342 D9908
	0.200	400	0.050		< 0.02	0.02	< 0.04	7	
BR/BRU Elias Fausto 2000/156	0.100	400	0.025	4	< 0.02	< 0.02	< 0.04	7	#2001/5002342 D9908
	0.200	400	0.050		0.03	< 0.02	0.05	7	
BR/BRT Marilia-SP 2000/157/	0.100	400	0.025	4	0.02	< 0.02	< 0.04	0	#2001/5002342 D9908
					0.02	< 0.02	< 0.04	3	
					0.02	< 0.02	< 0.04	7	
					0.02	< 0.02	< 0.04	14	
BR/BRT Morretes-PR 2000/158/	0.100	400	0.025	4	< 0.02	< 0.02	< 0.04	7	#2001/5002342 D9908
	0.200	400	0.050		< 0.02	< 0.02	< 0.04	7	

Cantaloupe

In the six trials performed in cantaloupe, BAS 500 00 F was applied six times at a use rate of 0.224 kg ai/ha (Wofford T. et al. BASF 1999/5083). The crops were harvested directly after last application (day 0). This use pattern corresponds to agricultural practice in USA.

The results are summarized in Table 13.

Table 13. Residues in cantaloupe treated with BAS 500 00 F in supervised trials conducted in USA.

Location/ trial code	Application		Residues (mg/kg)				Ref. Report No
	No.	kg ai/ha	Day	BAS 500 F	500M07	Total residue	
(GAP: 4 × 0.224 kg ai/ha with a PHI of 0 days)							
Henry county, AL 98006	6	0.224	0	0.10, 0.11	< 0.02, < 0.02	0.12, 0.13	995083/ D9908
Ottawa County, MI 98012	6	0.224	0	0.10, 0.10	< 0.02, < 0.02	0.12, 0.12	
Caddo County, OK 98015	6	0.224	0	0.08, 0.06	0.03, 0.02	0.11, 0.08	
Tulare County, CA 98017	6	0.224	0	0.13, 0.12	< 0.02, < 0.02	0.15, 0.14	
Glenn County, CA 98018	6	0.224	0	0.12, 0.08	< 0.02, < 0.02	0.14, 0.10	
Fresno County, CA	6	0.224	0	0.09, 0.09	< 0.02, < 0.02	0.11, 0.11	
Tift County, GA 98005	6	0.224	0	< 0.02, 0.05	< 0.02, < 0.02	0.04, 0.07	995083/ D9908
			3	< 0.02, < 0.02	< 0.02, < 0.02	0.04, 0.04	
			7	< 0.02, < 0.02	< 0.02, < 0.02	0.04, 0.04	
			10	< 0.02, < 0.02	< 0.02, < 0.02	0.04, 0.04	
			15	< 0.02, < 0.02	< 0.02, < 0.02	0.04, 0.04	
Tulare county, CA 98016	6	0.224	0	0.12, 0.09	< 0.02, < 0.02	0.14, 0.11	995083/ D9908
			3	0.03, 0.03	< 0.02, < 0.02	0.05, 0.05	
			7	< 0.02, < 0.02	< 0.02, < 0.02	0.04, 0.04	
			10	< 0.02, < 0.02	< 0.02, < 0.02	0.04, 0.04	
			15	< 0.02, < 0.02	< 0.02, < 0.02	0.04, 0.04	

Peppers and tomato

During the growing seasons from 1999 to 2003, eleven studies with a total of 23 field and 20 greenhouse trials were conducted in different representative areas for pepper and tomato cultivation in France, Greece, Italy and Spain (Balluf M., BASF 2001/1009067 and BASF 2001/1009060, Treiber S., 2001/1006129, Schulz H., BASF 2004/1015938).

The applications were done about 17, 10 and 3 days before commercial harvest of the crop. The intended PHI was 3 days. For the analysis, fruits were sampled immediately after the last application as well as about 3, 7 and 10 days thereafter.

The 2004 JMPR reported field trials on peppers from Brazil and USA. The latter ones were performed according to GAP.

The results of European trials are summarized in Tables 14 and 16-18 and 17-18. Table 15 contains the trial data reported by the 2004 JMPR.

Table 14. Results of supervised trials conducted on field peppers in Europe.

Country/ year trial code	Application[1]			Residues[2] (mg/kg)				Ref. Report No
	No.	kg ai/ha	kg ai/hL	Day	BAS 500 F	500M07	Total residue	
Italy 2000 (I00W018R)	3	0.100	0.01	0	0.03	< 0.02	0.05	#2001/ 1009087
				3	0.03	< 0.02	0.05	
				7	< 0.02	< 0.02	< 0.04	
				14	< 0.02	< 0.02	< 0.04	
Italy 2000 (I00W019R)	3	0.100	0.01	0	0.03	< 0.02	0.05	#2001/ 1009087
				3	0.03	< 0.02	0.05	
				7	< 0.02	< 0.02	< 0.04	
				14	< 0.02	< 0.02	< 0.04	
Italy 2000 (I00W020R)	3	0.100	0.01	0	0.04	< 0.02	0.06	#2001/ 1009087
				3	0.02	< 0.02	0.04	
				7	< 0.02	< 0.02	< 0.04	
				14	0.03	< 0.02	0.05	
I Italy 2000 (I00W021R)	3	0.100	0.01	0	0.11	< 0.02	0.13	#2001/ 1009087
				3	0.13	< 0.02	0.15	
				7	0.08	< 0.02	0.10	
				14	0.04	< 0.02	0.06	
Spain[1] 2001 (ALO/05/01)	3	0.100	0.01	0	0.22	< 0.02	0.24	#2001/ 1015036
				3	0.13	< 0.02	0.15	
				7	0.10	< 0.02	0.12	
				14	0.04	< 0.02	0.06	
Spain[1] 2001 (ALO/05/01)	3	0.100	0.01	0	0.36	0.02	0.38	#2001/ 1015036
				2	0.25	0.03	0.28	
				7	0.10	< 0.02	0.12	
				14	0.04	< 0.02	0.06	
Spain[1] 2001 (ALO/05/01)	3	0.100	0.01	0	0.06	< 0.02	0.08	#2001/ 1015036
				3	0.03	< 0.02	0.05	
				7	0.02	< 0.02	0.04	
				14	< 0.02	< 0.02	< 0.04	
Spain[1] 2001 (ALO/05/01)	3	0.100	0.01	0	0.09	< 0.02	0.11	#2001/ 1015036
				3	0.09	< 0.02	0.11	
				6	0.07	< 0.02	0.09	
				14	0.04	< 0.02	0.06	

1 BAS 516 GA F formulation is not indicated, BAS 518 01 F is marked with superscript 1.

2 Residues were measured in pepper fruits

Table 15. Pyraclostrobin residues in pepper resulting from supervised trials in Brazil and USA (reported by the 2004 JMPR).

Location	Application		Residues, mg/kg			PHI days	Ref Report No.
	No.	kg ai/ha	BAS 500F	500M07	Total		Methods
(GAP: 3 × 0.1 kg ai/ha with a PHI of 7 days)							
BR/BRX 2000/577	4	0.15	0.12	< 0.02	0.14	0	#2001/5002342 D9908
			0.11	0.02	0.13	1	
			0.04	0.02	0.07	3	
			< 0.02	0.02	< 0.04	7	
			< 0.02	0.02	< 0.04	10	
BR/BRU 2000/502	4	0.15	0.17	< 0.02	0.19	3	#2001/5002342 D9908
		0.30	0.52	< 0.02	0.54	3	
BR/BRT 2000/574	4	0.15	0.22	< 0.02	0.24	3	#2001/5002342 D9908
		0.30	0.17	< 0.02	0.19	3	
BR/BRV 2000/576	4	0.15	0.32	0.07	0.39	3	#2001/5002342 D9908
		0.30	0.28	0.05	0.33	3	
USA Oklahoma Dill City.	6	0.224	0.82	0.04	0.86	0	# 1999/5151 421/0 (g)
USA Texas Claude.	6	0.224	0.22	< 0.02	0.24	0	# 1999/5151 421/0 (g)
USA New Mexico Hatch.	6	0.224	0.14	< 0.02	0.16	0	# 1999/5151 421/0 (g)

Table 16. Results of supervised trials conducted on field tomato in Europe.

Country/ year trial code	Application[1]			Residues[2] (mg/kg)				Ref. Report No
	No.	kg ai/ha	kg ai/hL	Day	BAS 500 F	500M07	Total residue	
GAP: 3 × 0.067-0.1 kg ai/ha with a PHI of 3 days								
Italy 2000 (I00W022R)	3	0.100	0.025	0	0.07	< 0.02	0.09	#2001/ 1009086
				3	< 0.02	< 0.02	< 0.04	
				7	0.02	< 0.02	0.04	
				13	< 0.02	< 0.02	< 0.04	
Italy 2000 (I00W023R)	3	0.100	0.025	0	0.06	< 0.02	0.08	#2001/ 1009086
				3	0.04	< 0.02	0.06	
				7	< 0.02	< 0.02	< 0.04	
				14	< 0.02	< 0.02	< 0.04	
Italy 2000 (I00W024R)	3	0.100	0.025	0	0.04	< 0.02	0.06	#2001/ 1009086
				3	< 0.02	< 0.02	< 0.04	
				7	< 0.02	< 0.02	< 0.04	
				14	< 0.02	< 0.02	< 0.04	
Italy 2000 (I00W025R)	3	0.100	0.025	0	0.19	< 0.02	0.21	#2001/ 1009086
				3	0.13	< 0.02	0.15	
				7	0.09	< 0.02	0.11	
				14	0.09	< 0.02	0.11	
Spain 2001 (ALO/07/01)	3	0.100	0.025	0	0.07	< 0.02	0.09	#2001/ 1015035
				4	0.04	< 0.02	0.06	
				7	0.03	< 0.02	0.05	
				14	0.02	< 0.02	0.04	
Spain 2001 (ALO/08/01)	3	0.100	0.025	0	0.12	< 0.02	0.14	#2001/ 1015035
				4	0.09	< 0.02	0.11	
				7	0.07	< 0.02	0.09	
				14	0.04	< 0.02	0.06	

Country/ year trial code	Application[1]			Residues[2] (mg/kg)				Ref. Report No
	No.	kg ai/ha	kg ai/hL	Day	BAS 500 F	500M07	Total residue	
Spain 2001 (AYE/08/01)	3	0.100	0.025	0 4 7 14	0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.04 < 0.04 < 0.04 < 0.04	#2001/ 1015035
Spain 2001 (AYE/09/01)	3	0.100	0.025	0 3 7 14	0.04 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.06 0.04 < 0.04 < 0.04	#2001/ 1015035
Greece 2002 (02RF030/1)	3	0.100	0.025	0 2 7 14	0.11 0.04 0.04 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.13 0.06 0.06 < 0.04	#2004/ 1024744
France 2003 (FTL/06/03)	3	0.100	0.025	0 3 9 14	0.11 0.10 0.06 0.05	0.05 0.08 0.04 0.05	0.16 0.18 0.10 0.10	#2004/ 1015936
France 2003 (FBD/06/03)	3	0.100	0.025	0 3 7 14	0.10 0.07 0.04 0.07	< 0.02 0.04 < 0.02 < 0.02	0.12 0.11 0.06 0.09	#2004/ 1015936
Italy[1] 2003 (ITA/06/02)	3	0.100	0.01	0 2 7 13	0.07 0.03 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.09 0.05 < 0.04 < 0.04	#2003/ 1001360
Italy[1] 2003 (ITA/07/02)	3	0.100	0.01	0 3 7 14	0.11 0.06 0.04 0.05	< 0.02 < 0.02 < 0.02 < 0.02	0.13 0.08 0.06 0.07	#2003/ 1001360
Italy[1] 2003 (ITA/08/02)	3	0.100	0.01	0 3 7 14	0.07 0.03 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.09 0.05 < 0.04 < 0.04	#2003/ 1001360
Italy[1] 2003 (ITA/09/02)	3	0.100	0.01	0 3 7 13	0.14 0.11 0.08 0.05	< 0.02 < 0.02 < 0.02 < 0.02	0.16 0.13 0.10 0.07	#2003/ 1001360

1. BAS 516 GA F formulation is not indicated, BAS 518 01 F is marked with superscript 1.
2. Residues were measured in tomato fruits

Table 17. Results of supervised trials conducted with BAS 516 GA F on greenhouse peppers in Europe.

Country/ year trial code	Application			Residues (mg/kg)				Ref. Report No
	No.	kg ai/ha	kg ai/hL	Day	BAS 500 F	500M07	Total residue	
GAP: 3 × 0.067-0.1 kg ai/ha with a PHI of 3 days								
Spain 1999 (S99018R)	3	0.100	0.025	0 3 7 14	0.16 0.13 0.07 0.08	< 0.02 < 0.02 < 0.02 < 0.02	0.18 0.15 0.09 0.10	#2001/ 1009060

Country/ year trial code	Application			Residues (mg/kg)				Ref. Report No
	No.	kg ai/ha	kg ai/hL	Day	BAS 500 F	500M07	Total residue	
Spain 1999 (S99019R)	3	0.100	0.025	0	0.28	< 0.02	0.30	#2001/ 1009060
				2	0.16	< 0.02	0.18	
				7	0.17	< 0.02	0.19	
				14	0.14	< 0.02	0.16	
Spain 1999 (S99020R)	3	0.100	0.025	0	0.11	< 0.02	0.13	#2001/ 1009060
				3	0.07	< 0.02	0.09	
				8	0.03	< 0.02	0.05	
				14	< 0.02	< 0.02	< 0.04	
Spain 1999 (S99021R)	3	0.100	0.025	0	0.52	< 0.02	0.54	#2001/ 1009060
				3	0.24	< 0.02	0.26	
				7	0.30	< 0.02	0.32	
				14	0.26	< 0.02	0.28	
Spain 2000 (AC/03/00)	3	0.100	0.025	0	0.11	< 0.02	0.13	#2001/ 1006129
				3	0.08	< 0.02	0.10	
				7	0.04	< 0.02	0.06	
				14	< 0.02	< 0.02	< 0.04	
Spain 2000 (AC/04/00)	3	0.100	0.025	0	0.06	< 0.02	0.08	#2001/ 1006129
				4	0.08	< 0.02	0.10	
				7	0.07	< 0.02	0.09	
				14	0.03	< 0.02	0.05	
Spain 2000 (AC/05/00)	3	0.100	0.025	0	0.14	< 0.02	0.16	#2001/ 1006129
				4	0.17	< 0.02	0.19	
				7	0.09	< 0.02	0.11	
				14	0.07	< 0.02	0.09	
Spain 2000 (AC/06/00)	3	0.100	0.025	0	0.15	< 0.02	0.17	#2001/ 1006129
				3	0.13	< 0.02	0.15	
				7	0.12	< 0.02	0.14	
				14	0.08	< 0.02	0.10	
Greece 2002 (02RF030/3)	3	0.100	0.025	0	0.17	< 0.02	0.19	#2004/ 1024744
				3	0.06	< 0.02	0.08	
				7	0.04	< 0.02	0.06	
				14	0.02	< 0.02	0.04	

Table 18. Results of supervised trials conducted with BAS 516 GA F on greenhouse tomato in Europe.

Country/ year trial code	Application			Residues (mg/kg)				Ref. Report No
	No.	kg ai/ha	kg ai/hL	Day	BAS 500 F	500M07	Total residue	
Spain 1999 (S99014R)	3	0.100	0.025	0	0.04	< 0.02	0.06	#2001/ 1009067
				3	0.06	< 0.02	0.08	
				7	0.03	< 0.02	0.05	
				14	< 0.02	< 0.02	< 0.04	
Spain 1999 (S99015R)	3	0.100	0.025	0	0.09	< 0.02	0.11	#2001/ 1009067
				2	0.06	< 0.02	0.08	
				7	0.03	< 0.02	0.05	
				15	0.04	< 0.02	0.06	
Spain 1999 (S99016R)	3	0.100	0.025	0	0.11	< 0.02	0.13	#2001/ 1009067
				3	0.09	< 0.02	0.11	
				7	0.04	< 0.02	0.06	
				14	< 0.02	< 0.02	< 0.04	

Country/ year trial code	Application			Residues (mg/kg)				Ref. Report No
	No.	kg ai/ha	kg ai/hL	Day	BAS 500 F	500M07	Total residue	
Spain	3	0.100	0.025	0	0.11	< 0.02	0.13	#2001/
1999				3	0.12	< 0.02	0.14	1009067
(S99017R)				8	0.05	< 0.02	0.07	
				14	0.09	< 0.02	0.11	
Spain	3	0.100	0.025	0	0.05	< 0.02	0.07	#2001/
2000				3	0.04	< 0.02	0.06	1006129
(AC/07/00)				7	0.04	< 0.02	0.06	
				14	0.06	< 0.02	0.08	
Spain	3	0.100	0.025	0	0.04	< 0.02	0.06	#2001/
2000				4	0.03	< 0.02	0.05	1006129
(AC/08/00)				7	0.04	< 0.02	0.06	
				13	0.03	< 0.02	0.05	
Spain	3	0.100	0.025	0	0.05	< 0.02	0.07	#2001/
2000				4	0.07	< 0.02	0.09	1006129
(AC/09/00)				7	0.04	< 0.02	0.06	
				14	0.03	< 0.02	0.05	
Spain	3	0.100	0.025	0	0.05	< 0.02	0.07	#2001/
2000				4	0.06	< 0.02	0.08	1006129
(AC/10/00)				7	0.04	< 0.02	0.06	
				14	0.05	< 0.02	0.07	
Greece	3	0.100	0.025	0	0.14	< 0.02	0.16	#2004/
2002				3	0.03	< 0.02	0.05	1024744
(02RF030/2)				7	0.06	< 0.02	0.08	
				14	0.04	< 0.02	0.06	
France	3	0.100	0.025	0	0.05	< 0.02	0.07	#2004/
2003				3	0.07	< 0.02	0.09	1015938
(FAN/13/03)				7	0.09	< 0.02	0.11	
				13	0.07	< 0.02	0.09	
France	3	0.100	0.025	0	0.11	< 0.02	0.13	#2004/
2003				3	0.11	< 0.02	0.13	1015938
(FTL/07/03)				7	0.04	< 0.02	0.06	
				14	0.06	< 0.02	0.08	

Kale

During the 1999 and 2000 growing seasons, two studies (Beck J., BASF 2001/1001000 and 2001/1001001) with a total of six field trials were conducted in curly kale in Denmark, Great Britain, the Netherlands and Sweden. The BAS 516 GA F was applied four times at 1.5 kg/ha each, in a spray volume of 300 L/ha. The applications were done about 5, 4, 3 and 2 weeks before anticipated commercial harvest of the crop. Samples were taken from 0 to 20–21 days after last application. The product is registered in UK with a GAP of 3 applications at 0.067 kg ai/ha with a PHI of 14 days.

The results are summarized in Table 19.

Table 19. Summary of residues of pyraclostrobin in kale leaves derived from treatments carried out with BAS 516 GA F.

CROP	Application				Residues [mg/kg]			Ref. Report No
Country/ year trial code	No.	kg ai/ha	kg ai/hL	Day	Parent	500M07	Total	
GAP: 3 × 0.067-0.1 kg ai/ha with a PHI of 14 days								
Denmark	4	0.100	0.033	0	1.25	0.05	1.30	#2001/
2000				7	1.10	0.07	1.17	1001000
(ALB/08/00)				14	0.07	< 0.02	0.09	
				20	0.09	< 0.02	0.11	
Great Britain	4	0.100	0.033	0	0.07	< 0.02	0.09	#2001/
1999				6	< 0.02	< 0.02	< 0.04	1001001
(OAT/19/99)				13	< 0.02	< 0.02	< 0.04	
				21	< 0.02	< 0.02	< 0.04	
Great Britain	4	0.100	0.033	0	0.67	0.14	0.81	#2001/
1999				7	0.07	< 0.02	0.09	1001001
(OAT/21/99)				13	0.06	< 0.02	0.08	
				21	0.02	< 0.02	0.04	
Great Britain	4	0.100	0.033	0	1.83	< 0.02	1.85	#2001/
2000				8	0.38	< 0.02	0.40	1001000
(OAT/08/00)				14	0.18	< 0.02	0.20	
				21	0.26	< 0.02	0.28	
The Netherlands	4	0.100	0.033	0	1.87	0.06	1.93	#2001/
1999				7	< 0.02	< 0.02	< 0.04	1001001
(AGR/11/99)				15	0.31	< 0.02	0.33	
				22	0.11	< 0.02	0.13	
Sweden	4	0.100	0.033	0	0.87	0.03	0.90	#2001/
1999				8	0.40	< 0.02	0.42	1001001
(HUS/06/99)				14	0.49	0.03	0.52	
				21	0.61	0.04	0.65	

Lettuce, head

A pesticide product containing 20% pyraclostrobin has been registered for Brassica vegetables (head and stem), Brassica leafy vegetables and leafy vegetables (except Brassica) in the USA. For lettuce, 4 applications at 0.117–0.23 kg ai/ ha are authorised with PHI of 0 day.

The results of trials reported by the 2004 JMPR are shown in Table 20.

Table 20. Pyraclostrobin residues at Day 0 in lettuce resulting from supervised trials in USA.

	Application				Residues, [mg/kg]			Trials number
Location	kg ai/ha	Water L/ha	kg ai/hL	No.	Parent	500M07	total	Method
GAP: 4 × 0.23 kg ai/ha with a PHI of 0 day								
USA California Salinas	0.224	625	0.036	4	3.69	0.14	3.83	# 2002/5003764 D9908
USA Florida Gainesville	0.224	375	0.060	4	13.70	0.34	14.0	# 2002/5003764 D9908
USA California El Centro	0.224	510	0.044	4	1.95	0.09	2.04	# 2002/5003764 D9908
USA Columbia Cloverdale	0.224	719	0.031	4	4.96	0.21	5.17	# 2002/5003764 D9908
USA California Parlier	0.224	346	0.065	4	19.70	0.35	20.1	# 2002/5003764 D9908
USA California Parlier	0.224	341	0.066	4	14.90	0.36	15.3	# 2002/5003764 D9908

Further 18 trials were carried out in typical growing regions of Europe (Beck J., BASF Doc ID 2001/1000998 and2001/1000999, Schulz H., BASF 2001/1000933) during 1999 and 200 according to the GAP (2 × 0.1 kg ai/ha at 10–14 day interval and PHI of 14 days).

The two applications with 0.1 kg ai/ha were made about 4 and 2 weeks before commercial harvest of the crop. For the analysis, lettuce heads were sampled immediately after the last application as well as about 7, 14 and 21 days thereafter.

During the 2002 growing season, one study (Young H. and Atkinson S., BASF 2003/1001259) with eight greenhouse trials was conducted in Germany, Spain, France and the Netherlands. The BAS 516 00 F was tested performing two applications with 1.5 kg/ha each in a spray volume of 500 L/ha resulting in application rates of 0.1 kg ai/ha of pyraclostrobin. The applications were done about 4 and 2 weeks before commercial harvest of the crop. The intended PHI was 14 days. For the analysis, lettuce heads were sampled immediately after the last application as well as about 7, 14 and 21 days thereafter.

The results are summarized in Tables 21 and 22.

Table 21. Results of supervised field trials conducted with BAS 516 GA F on head lettuce.

Country/ year trial code	Application			Residues [mg/kg]				Ref. Report No
	No.	kg ai/ha	kg ai/hL	Day	Parent	500M07	Total	
GAP: 2 × 0.1 kg ai/ha with a PHI of 14 days								
Germany 1999 (ACK/06/99)	2	0.100	0.025	0 7 14 21	2.39 0.15 < 0.02 < 0.02	0.06 < 0.02 < 0.02 < 0.02	2.44 0.17 < 0.04 < 0.04	#2001/ 1000998
Germany 1999 (DU2/11/99)	2	0.100	0.025	0 7 14 21	2.73 0.13 0.06 < 0.02	0.024 < 0.02 < 0.02 < 0.02	2.76 0.15 0.08 < 0.04	#2001/ 1000998
Germany 1999 (DU4/08/99)	2	0.100	0.025	0 5 13 20	4.65 0.39 0.08 0.03	0.044 0.045 < 0.02 < 0.02	4.69 0.44 0.10 0.05	#2001/ 1000998
Germany 2000 (ACK/03/00)	2	0.100	0.025	0 6 14 22	1.76 < 0.02 0.28 < 0.02	< 0.02 < 0.02 0.03 < 0.02	1.78 < 0.04 0.32 < 0.04	#2001/ 1000999
Germany 2000 (DU2/05/00)	2	0.100	0.025	0 7 14 21	2.39 0.06 < 0.02 < 0.02	0.03 < 0.02 < 0.02 < 0.02	2.42 0.08 < 0.04 < 0.04	#2001/ 1000999
Spain 1999 (AC/14/99)	2	0.100	0.025	0 7 13 20	1.78 0.43 0.04 0.04	< 0.02 0.03 < 0.02 < 0.02	1.80 0.46 0.06 0.06	#2001/ 1000998
Spain 1999 (AC/15/99)	2	0.100	0.025	0 7 13 20	1.81 0.41 0.04 < 0.02	0.02 0.03 < 0.02 < 0.02	1.83 0.44 0.06 < 0.04	#2001/ 1000998
Spain 1999 (AC/16/99)	2	0.100	0.025	0 7 13 20	2.45 0.29 0.08 < 0.02	0.05 0.05 < 0.02 < 0.02	2.50 0.34 0.10 < 0.04	#2001/ 1000998

Country/ year trial code	Application			Residues [mg/kg]				Ref. Report No
	No.	kg ai/ha	kg ai/hL	Day	Parent	500M07	Total	
Spain 2000 (AC/15/00)	2	0.100	0.025	0 6 14 20	4.05 0.14 < 0.02 < 0.02	0.03 0.02 < 0.02 < 0.02	4.09 0.16 < 0.04 < 0.04	#2001/ 1000999
Spain 2000 (AC/16/00)	2	0.100	0.025	0 6 14 21	1.77 0.19 0.03 < 0.02	< 0.02 0.03 < 0.02 < 0.02	1.79 0.22 0.05 < 0.04	#2001/ 1000999
France 1999 (X 99 62 11)	2	0.100	0.025	0 7 14 21	2.53 0.08 < 0.02 < 0.02	0.05 0.02 < 0.02 < 0.02	2.58 0.10 < 0.04 < 0.04	#2001/ 1000933
France 1999 (X 99 62 12)	2	0.100	0.025	0 7 14 21	3.09 0.07 < 0.02 < 0.02	0.11 0.03 < 0.02 < 0.02	3.20 0.10 < 0.04 < 0.04	#2001/ 1000933
France 1999 (FR4/01/99)	2	0.100	0.025	0 7 14 21	2.76 0.24 0.04 < 0.02	< 0.02 0.03 < 0.02 < 0.02	2.78 0.27 0.06 < 0.04	#2001/ 1000998
France 2000 (FR3/06/00)	2	0.100	0.025	0 7 14 21	2.21 0.36 0.38 0.25	0.06 0.05 0.04 0.03	2.27 0.40 0.42 0.29	#2001/ 1000999
France 2000 (FR4/06/00)	2	0.100	0.025	0 7 13 20	2.03 0.11 0.04 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	2.05 0.13 0.06 < 0.04	#2001/ 1000999
France 2000 (FR8/05/00)	2	0.100	0.025	0 7 14 21	1.62 0.28 0.08 0.13	0.03 0.03 < 0.02 < 0.02	1.65 0.31 0.10 0.15	#2001/ 1000999
The Netherlands 1999 (AGR/13/99)	2	0.100	0.025	0 6 14 22	1.75 0.11 < 0.02 < 0.02	0.04 < 0.02 < 0.02 < 0.02	1.78 0.13 < 0.04 < 0.04	#2001/ 1000998
The Netherlands 2000 (AGR/03/00)	2	0.100	0.025	0 7 13 20	1.74 0.11 0.04 < 0.02	0.02 < 0.02 < 0.02 < 0.02	1.77 0.13 0.06 < 0.04	#2001/ 1000999

Table 22. Results of supervised trials conducted with BAS 516 00 F on head lettuce in greenhouse.

CROP	Application				Residues [mg/kg]			Ref. Report No
Country/ year trial code	No.	kg ai/ha	kg ai/hL	Day	Parent	500M07	Total	
GAP: 2 × 0.1 kg ai/ha with a PHI of 14 days								
Germany 2002 (ACK/03/02)	2	0.100	0.025	0 7 13 20	6.71 0.18 0.03 < 0.02	0.03 < 0.02 < 0.02 < 0.02	6.74 0.20 0.05 < 0.04	#2003/ 1001259

CROP	Application				Residues [mg/kg]			Ref. Report No
Country/ year trial code	No.	kg ai/ha	kg ai/hL	Day	Parent	500M07	Total	
The Netherlands 2002 (AGR/08/02)	2	0.100	0.025	0 6 14 20	5.87 1.98 0.81 0.19	< 0.02 0.05 0.03 < 0.02	5.89 2.03 0.84 0.21	#2003/ 1001259
Spain 2002 (ALO/04/02)	2	0.100	0.025	0 7 14 21	3.86 0.61 0.75 0.32	0.04 0.04 0.07 0.04	3.90 0.65 0.82 0.36	#2003/ 1001259
Spain 2002 (AYE/03/02)	2	0.100	0.025	0 7 14 21	6.31 0.50 0.04 < 0.02	0.11 0.04 < 0.02 < 0.02	6.42 0.54 0.06 < 0.04	#2003/ 1001259
France 2002 (FAN/04/02)	2	0.100	0.025	0 7 14 21	3.30 0.53 0.23 0.09	0.02 < 0.02 < 0.02 < 0.02	3.32 0.55 0.25 0.11	#2003/ 1001259
France 2002 (FBD/04/02)	2	0.100	0.025	0 7 13 21	4.88 1.09 0.29 0.07	0.04 0.02 < 0.02 < 0.02	4.92 1.11 0.31 0.09	#2003/ 1001259
France 2002 (FBM/02/02)	2	0.100	0.025	0 7 14 21	1.89 0.99 0.33 0.19	0.03 0.03 < 0.02 < 0.02	1.92 1.02 0.35 0.21	#2003/ 1001259
France 2002 (FTL/21/02)	2	0.100	0.025	0 7 14 21	3.79 0.26 0.13 < 0.02	0.04 0.03 < 0.02 < 0.02	3.83 0.29 0.15 < 0.04	#2003/ 1001259

Snap beans

Nine trials were carried out at various locations of the USA during the 2000 growing season. Two foliar applications were made to each treated plot at a target rate of 0.224 kg ai/ha. The intervals between the two applications were not reported.

Samples of snap beans of normal maturity were collected 7 days after the last application. The results of the trials, reported by the 2004 JMPR, are shown in Table 23.

Table 23. Pyraclostrobin residues in snap beans resulting from supervised trials in USA

	Application per treatment				Residues [mg/kg]				trials number
Location USA	kg ai/ha	Water L/ha	kg ai/hL	No of tr.	Parent	500M07	Total	PHI days	Method
GAP: 4 × 0.13 kg ai/ha with a PHI of 7 days									
Germansville, PA	0.230	287	0.08	2	0.10	0.04	0.14	7	# y2001/5000906 D9808
Athens CA	0.225	259	0.08	2	0.10	0.04	0.14	7	# 2001/5000906 D9608
Geneva MN	0.224	170	0.13	2	0.13	0.03	0.15	7	# 2001/5000906 D9808
Arkansaw W	0.227	189	0.12	2	< 0.02	< 0.02	0.04	14	# 2001/5000906
Madera CA	0.224	280	0.08	2	0.08	0.06	0.13	7	# 2001/5000906 D9808
Jerome ID	0.222	308	0.07	2	0.04	0.03	0.07	7	# 2001/5000906
Kings county	0.225	248	0.091	2	0.11	0.02	0.13	7	# 2001/5000906

	Application per treatment				Residues [mg/kg]				trials number
Location USA	kg ai/ha	Water L/ha	kg ai/hL	No of tr.	Parent	500M07	Total	PHI days	Method
GAP: 4 × 0.13 kg ai/ha with a PHI of 7 days									
NS									D9808
St. Cesaire .QC	0.222	274	0.08	2	0.12	0.03	0.15	7	# 2001/5000906 D9808
St. Cesaire. QC	0.226	283	0.08	2	0.16	0.03	0.19	7	# 2001/5000906 D9808

Vining peas

Four studies (Jones S., BASF 2003/1012652, Smalley R., BASF 2003/1004355, Schulz H., BASF 2004/1010544 and 2004/1006472) with a total of 21 field trials were conducted in vining peas in France (N/S), the United Kingdom, Germany, Denmark and Sweden in 2002 and 2003. The BAS 512 00 F and BAS 516 00 F were applied twice with a target rate of 100 to 67 g ai/ha. The samples were taken at earliest commercial harvest (corresponding to approximately 8–14 days after the last application). The results are summarized in Table 24. In green peas the residues were below the limit of quantitation of 0.02 mg/kg.

Table 24. Residue ranges of pyraclostrobin derived from supervised field trials conducted on vining peas.

Crop	No. of trials	Application		DALA	Residues (mg/kg)		
		Rate (kg ai/ha)	No.		Parent	500M07	Total
GAP: 2 × 0.1 kg ai/ha with a PHI of 35 days							
BAS 512 00 F (2002, BASF Doc ID 2003/1012652)							
Vining peas	6	0.100	2	0 8 -18* 8 - 18**	0.15 - 6.54 < 0.02 0.14 - 1.18	0.06 - 0.77 < 0.02 0.15 - 0.73	0.22 - 7.31 < 0.04 0.29 - 1.78
BAS 512 00 F (2003, BASF Doc ID 2004/1010544)							
Vining peas	6	0.100	2	0 3 - 21* 3 - 21**	1.17 - 3.67 < 0.02 0.12 - 3.81	0.11 - 0.27 < 0.02 0.10 - 1.26	1.31 - 3.94 < 0.04 0.22 - 5.06
BAS 516 00 F (2002, BASF Doc ID 2003/1004355)							
Vining peas	5	0.067	1	0 11 - 15* 11 - 15** 14 - 22* 14 - 22**	0.37 - 1.50 < 0.02 0.12 - 0.63 < 0.02 0.08 - 0.48	< 0.02 - 0.02 < 0.02 0.04 - 0.16 < 0.02 0.02 - 0.13	0.39 - 1.52 < 0.04 0.16 - 0.79 < 0.04 0.10 - 0.61
BAS 516 00 F (2003, BASF Doc ID 2004/1006472)							
Vining peas	4	0.067	1	0 14* 14** 21* 21**	0.60 - 1.23 < 0.02 0.08 - 0.88 < 0.02 0.07 - 0.95	< 0.02 < 0.02 < 0.02 - 0.19 < 0.02 < 0.02 - 0.31	0.62 - 1.25 < 0.04 0.10 - 1.07 < 0.04 0.09 - 1.26

* green peas ** rest of plant

Soybean

In 2002, 17 field trials were conducted in the major growing regions in the US (Leonard R.C., BASF 2002/5004272). Two sequential applications were performed 7 ± 1 day apart with BAS 500 02 F at a rate of 0.224 kg as/ha. There was a 7 day target interval between the two applications. Duplicate

samples were taken 5 days after last application (immature seed) and at day 28 after the last application (dry seed). In immature seeds, the pyraclostrobin residue levels ranged from < 0.02 to 0.30 mg/kg. In ripe soybean seed, no residues above the limit of quantitation were found in any of the samples.

Soybean forage samples were collected 14 day after last application. The results are summarised in Table 25.

The supervised trials were conducted in Brazil (Abdel-Baky S., BASF 2001/5002354). The residues detected in soybean seeds are summarized in Table 26 together with those which were reported by the 2004 JMPR.

Table 25. Residues in immature soybean seeds, soybean forage and hay following two applications of pyraclostrobin at a total rate of 0.448 kg (0.437–0.459) ai/ha with spray volume ranging from 187 to 364 L/ha (Leonard R.C., BASF 2002/5004272).

RCN (State or province)	Residues [mg/kg]			PHI
	Parent	500M07	Total	
GAP: 2 × 0.42 kg ai/ha with a PHI of 21 days				
Immature seed				
2002191 (GA)	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04,< 0.04	5
2002192 (VA)	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04,< 0.04	5
2002193 (AR)	0.24, 0.3	0.03, 0.03	0.27, 0.33	5
2002194 (AR)	< 0.02, 0.02	< 0.02, < 0.02	< 0.04, 0.04	5
2002195 (VW)	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04,< 0.04	5
2002196 (MN)	0.05, 0.08	< 0.02, < 0.02	0.07, 0.1	5
2002197 (IA)	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04,< 0.04	5
2002198 (IA)	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04,< 0.04	5
2002199 (NE)	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04,< 0.04	5
2002200 (NE)	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04,< 0.04	5
2002201 (ND)	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04,< 0.04	5
2002202 (ND)	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04,< 0.04	5
2002203 (ND)	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04,< 0.04	5
2002204 (SD)	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04,< 0.04	5
2002205 (IL)	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04,< 0.04	5
2002206(IL)	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04,< 0.04	5
2002216(Qb)	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04,< 0.04	5
Forage				
2002191 (GA)	2.48, 2.91	0.30, 0.34	2.78, 3.25	14
2002192 (VA)	2.70, 2.04	0.36, 0.24	3.06, 2.24	14
2002193 (AR)	3.19, 3.24	0.62, 0.63	3.81, 387	14
2002194 (AR)	1.39, 1.09	0.40, 0.31	1.79, 1.39	14
2002195 (VW)	0.75, 0.88	0.09, 0.08	0.84, 0.96	14
2002196 (MN)	2.42, 3.06	0.29, 0.31	2.71, 3.37	14
2002197 (IA)	1.08, 1.59	0.3, 0.3	0.38, 0.89	14
2002198 (IA)	0.65, 0.84	0.13, 0.18	0.78, 1.02	14
2002199 (NE)	1.11, 1.09	0.53, 0.45	1.64, 1.54	14
2002200 (NE)	2.16, 1.33	0.39, 0.23	2.55, 1.56	14
2002201 (ND)	1.58, 1.62	0.16, 0.17	1.74, 1.79	14
2002202 (ND)	2.40, 3.22	0.29, 0.28	2.69, 3.5	14
2002203 (ND)	1.34, 0.67	0.13, 0.07	1.47, 0.74	14
2002204 (SD)	1.96, 1.41	0.25, 0.18	2.21, 1.59	14
2002205 (IL)	1.35, 1.24	0.17, 0.16	1.52, 1.4	14
2002206(IL)	0.89, 0.90	0.2, 0.18	1.09, 1.08	14
2002216(Qb)	2.0, 1.7	0.22, 0.21	2.22, 1.91	14
2002191 (GA)	0.76, 0.99	0.11, 0.12	0.87, 1.11	28
2002192 (VA)	0.92, 072	0.25, 0.19	1.17, 1.91	28
2002193 (AR)	1.37, 1.38	0.76, 0.77	2.13, 2.15	28
2002194 (AR)	0.64, 0.66	0.37, 0.38	1.01, 1.04	28
2002195 (VW)	1.47, 0.03	0.29, < 0.02	1.76, 0.05	28

RCN (State or province)	Residues [mg/kg]			PHI
	Parent	500M07	Total	
2002196 (MN)	0.96, 1.01	0.27, 0.27	1.23, 1.28	28
2002197 (IA)	1.47, 1.47	0.63, 0.68	2.10, 2.15	28
2002198 (IA)	0.79, 0.65	0.25, 0.21	1.04, 0.86	28
2002199 (NE)	4.25, 4.10	1.88, 1.85	6.13, 5.95	28
2002200 (NE)	1.7, 2.64	0.64, 1.14	2.34,378	28
2002201 (ND)	2.18,2.0	0.6, 0.36	2.78, 2.36	28
2002202 (ND)	2.82, 2.3	0.65, 0.54	3.47, 2.48	28
2002203 (ND)	1.79, 1.8	0.6, 0.59	2.39, 2.39	28
2002204 (SD)	1.81, 1.92	0.69, 0.71	2.5, 2.63	28
2002205 (IL)	1.83, 2.0	0.45, 0.39	2.28, 2.39	28
2002206(IL)	2.16, 2.14	0.51, 0.49	2.67, 2.63	28
2002216(Qb)	1.96, 1.74	0.46, 0.45	2.42, 2.19	28

Table 26. Pyraclostrobin residues in soybeans resulting from supervised trials in Brazil and Argentina.

	Appl. per treatment					Residues [mg/kg]				Trials number
location	kg as/ha	Water L/ha	kg as/hL	No of tr.	Portion analysed (a)	Parent	500M07	Total	PHI days	Method
GAP: 2 × 0.075 kg ai/ha with a PHI of 14 days										
BR/ BR2[1] Nova Ramadas-RS 2000/365/	0.100	200	0.050	2	Grain	< 0.02	< 0.02	< 0.04	0	#2001/5002355 D9908
						< 0.02	< 0.02	< 0.04	7	
						≤ 0.02	< 0.02	< 0.04	14	
						< 0.02	< 0.02	< 0.04	21	
						< 0.02	< 0.02	< 0.04	28	
BR/ BRT[1] Londrina-PR 2000/366/	0.100	200	0.050	2	Grain	≤ 0.02	< 0.02	< 0.04	14	#2001/5002355 D9908
	0.200	200	0.100	2		< 0.02	< 0.02	< 0.04	14	
BR /BRT[1] Lapa-PR 2000/367/	0.100	200	0.050	2	Grain	≤ 0.02	< 0.02	< 0.04	14	#2001/5002355 D9908
	0.200	200	0.100	2		< 0.02	< 0.02	< 0.04	14	
BR/ BRV[1] Überlandia-2000/368/	0.100	200	0.050	2	Grain	≤ 0.02	< 0.02	< 0.04	14	#2001/5002355 D9908
	0.200	200	0.100	2		< 0.02	< 0.02	< 0.04	14	
CDR/F/2000/362/BRT	0.0997				Grain	≤ 0.02	0.02	< 0.04	14	#2001/5002354 D9908
	0.1995					< 0.02	0.02	< 0.04	14	
CDR/F/2000/361/BRV	0.0997				Grain	≤ 0.02	0.03	< 0.04	14	
	0.1995					< 0.02	0.02	< 0.04	14	
CDR/F/2000/364/BR2	0.0997				Grain	0.02	0.02	< 0.04	0	
						< 0.02	0.02	< 0.04	7	
						≤ 0.02	0.02	< 0.04	14	
						0.02	0.02	< 0.04	21	
						< 0.02	0.02	< 0.04	28	
AR Chaco[1]	0.075	200	0.038	2	pod with grain	0.29	< 0.02	0.31	1	#2001/1017043 445/0
					grain	0.03	< 0.02	0.05	20	
					grain	< 0.02	< 0.02	< 0.04	48	
	0.150	200	0.075	2	pod with grain	0.55	< 0.02	0.57	1	
					Grain	0.04	< 0.02	0.06	20	
					Grain	< 0.02	< 0.02	< 0.04	48	

1. Reported by the 2004 JMPR

Sunflower

In 2001, seven field trials were conducted in the US and Canada (Versoi P. L., Abdel-Baky S., BASF 2001/5002552) to investigate the residue behaviour of BAS 500 F in sunflowers. The test formulation, BAS 500 02 F, was applied twice at a rate of 0.224 kg ai/ha. There was a seven day target interval

between the two applications. Locally available adjuvants were added to each spray mixture. Seed samples were taken 21 days after the last application, which is the registered pre-harvest interval. In seeds, pyraclostrobin residue levels ranged from < 0.02 to 0.22 mg/kg.

At the Texas site one separate plot received 5× recommended rate, in order to produce material containing sufficient residues for processing[1]. Sunflowers were treated with two sequential foliar applications of BAS 510 F at 1.08 and 1.12 kg ai/ha, with a 6 day retreatment interval, totalling 2.2 kg ai/ha/season (5× of the proposed label rate for pyraclostrobin). Mature sunflower seeds were harvested 21 days after the last application. A locally available spray adjuvant was included with each application. All applications were made as foliar sprays using ground equipment.

In 2004, one further residue trial was performed in US (Leonard R.C., BASF 2005/5000022). Application rate and sampling was carried out as described above. The residue levels detected were below the limit of quantitation.

The results are summarized in Table 27.

Table 27. Residues of pyraclostrobin in sunflower seed derived from supervised field trials in USA and Canada (P. L. Versoi, S. Abdel-Baky, BASF 2001/5002552).

	Application				Residues [mg/kg]		
RCN (State or Province)	Single [kgai/ha]	Vol. L/ha	Total kgai/ha	PHI (days)	Parent	500M07	Total
GAP: 2 × 0.21-0.42 kg ai/ha with a PHI of 21 days							
2001284 (ND)	0.224	234	0.4448	21	< 0.02, 0.05	< 0.02, < 0.02	< 0.04, 0.07
2001285 (ND)	0.235,0.224	187	0.459	21	0.02; 0.04	< 0.02, < 0.02	0.04; 0.06
2001286 (ND)	0.235,0.224	187	0.459	21	0.02, < 0.02	< 0.02, < 0.02	0.04, < 0.04
2001287 (SD)	0.224	187	0.448	20	0.10,0.10	< 0.02, < 0.02	0.12,0.12
2001288 (SD)	0.213,0.224	178, 187	0.437	20	0.06, 0.05	< 0.02, < 0.02	0.08, 0.07
2001289 (TX)	0.213,0.224	458, 468	0.437	21	0.06, < 0.02	< 0.02, < 0.02	0.08, < 0.04
	1.08, 1.12		2.2	21	1.40, 0.63	0.35, 0.17	1.75, 0.80
2001 290 (MB)	0.224	112	0.448	21	0.11.0.22	< 0.02, 0.03	0.13,0.25
2004 152 (IL)[1]	0.224			21	< 0.02	< 0.02	< 0.04

1. Leonard R.C., BASF 2005/5000022

Coffee

Field trials were carried out in Brazil (Abdel-Baky S., BASF 2001/5002354 and 2000/5276, Regenstein H., BASF 2003/1013063) to complement the data submitted to the 2004 JMPR.

Coffee was treated with BAS 512 00F at target rates of 0.1 kg ai/ ha and 0.2 kg ai/ha.

The samples of coffee beans at full ripening stage (red coffee berry) were taken. The results of the trials are given in Table 28.

Table 28. Pyraclostrobin residues in coffee beans resulting from supervised trials in Brazil.

	Application per treatment				Residues [mg/kg]				Trials number
Location	kg ai/ha	Water L/ha	kg ai/hL	No of tr.	Parent	500M07	Total	PHI days	method
GAP: 2 × 0.2 kg ai/ha with a PHI of 45 days									
BR Santo Antonio	0.175	500	0.035	2	0.03	< 0.02	0.05	45	2001/5002355
De Posse-SP	0.350	500	0.070		< 0.02	< 0.02	< 0.04	45	D 9908
BRAraguariMG129	0.175	500	0.035	2	< 0.02	< 0.02	< 0.04	45	# 2001/5002355
BR	0.350	500	0.070		0.08	< 0.02	0.10	45	D 9908
BR Romaria-MG	0.175	500	0.035	2	0.12	< 0.02	0.14	0	# 2001/5002355
CDR/F/2000					< 0.02	< 0.02	< 0.04	15	D 9908
130 BRV					< 0.02	< 0.02	< 0.04	30	
					< 0.02	< 0.02	< 0.04	45	
					0.11	< 0.02	0.13	60	
BR	0.175	500	0.035	2	0.15	< 0.02	0.17	45	# 2001/5002355
Miraselva-P	0.350	500	0.070		0.12	0.02	0.14	45	D 9908

	Application per treatment				Residues [mg/kg]				Trials number
Location	kg ai/ha	Water L/ha	kg ai/hL	No of tr.	Parent	500M07	Total	PHI days	method
127 BRT									
CDR/F/2000/125BRV	0.183			1	< 0.02	< 0.02	< 0.04	0	2001/5002354/D 9908
					< 0.02	< 0.02	< 0.04	15	
					< 0.02	< 0.02	< 0.04	30	
					≤ 0.02	< 0.02	< 0.04	45	
CDR/F/2000/126BRV	0.183				< 0.02	< 0.02	< 0.04	45	2001/5002354/D 9908
	0.366				0.12	0.03	0.15		
CDR/F/2000/126BRV	0.183				≤ 0.02	< 0.02	< 0.04	45	2001/5002354/D 9908
	0.366				0.05	< 0.02	0.07	45	
CDR/F/2000/126BRV	0.183				≤ 0.02	< 0.02	< 0.04	45	2001/5002354/D 9908
	0.366				< 0.02	< 0.02	< 0.04	45	
CDR/F/2000/127BRT	0.15				0.15	0.02	0.17	45	2000/5276 /D9908
	0.30				0.12	0.02	0.14	45	
CDR/F/2000/128BRU	0.15				0.03	< 0.02	0.05	45	2000/5276/ D9908
	0.30				< 0.02	< 0.02	< 0.04	45	
CDR/F/2000/129BRV	0.15				≤ 0.02	< 0.02	< 0.04	45	2000/5276 D9908
	0.30				0.08	< 0.02	0.1	45	

Hops

During the 2000 and 2001 growing seasons, two studies (Schneider K.H., BASF 2001/1015050 and BASF 2001/1015052) with a total of eight field trials were conducted in the representative areas for hop cultivation in Germany. The BAS 516 01 F was tested in hops with three applications with 2.1 L/ha to 3.0 L/ha in a spray volume of 2300 to 3000 L/ha resulting in application rates of 0.21 to 0.30 kg ai/ha for pyraclostrobin. The applications were done about 6–7, 5 and 3 weeks before commercial harvest of the crop; the intended PHI is 21 days. For the analysis green hop cones were sampled immediately after the last application as well as about 14, 21 and 28 days later. During the last two samplings the collected green cones were divided into two portions. One was deep-frozen and the other part was dried for 6 hours at 60°C and deep-frozen on the following day.

During the 2003 growing season, one study (Schulz H., BASF 2003/1001292) with another four field trials was conducted in the representative areas for hop cultivation in Germany applying two formulated products. The BAS 516 01 F (100 g/L pyraclostrobin, 200 g/L boscalid, SE) and BAS 516 04 F (12.8% pyraclostrobin, 25.2% boscalid, WG) were compared, both with three applications at growth stages BBCH 61–63, 75 and 81. In both variants, the application rate at the first treatment was about 210 g/ha of pyraclostrobin. In the second and third treatments, about 250 g/ha of pyraclostrobin was used. The spray volumes per hectare were 2300 and 2700 L respectively. For the analysis, green hop cones were sampled immediately after the last application as well as 14, 21 and 28 days later.

In 2001, three field trials were conducted in the US (Jordan J.M., BASF 2001/5002574) to investigate the residue behaviour of BAS 500 F in dried hop cones. The test formulation, BAS 500 02 F, was applied three times at a use rate of approximately 0.25 kg ai/ha. There was a ten-day target interval between the applications. At each trial site one plot was treated with concentrated spray solution (187–935 L/ha) and another one was treated with diluted spray (935–3740 L/ha)

Hop cone samples were taken 0, 7 and 14 days after last application. They were dried on the field prior to shipment to the analytical laboratory.

The results are summarized in Table 29.

Table 29. Summary of residues in green and dry hops derived from supervised trials.

CROP	Application				Residues [mg/kg]					Ref. Report No
Country/ year trial code	Formulation	No.	kg ai/ha	kg ai/hL	Matrix	Day	Parent	500M07	Total residue	
GAP: 3 × 0.25 kg ai/ha with a PHI of 21 days										
Germany[1] 2000 (RF 0100)	BAS51601 F	3	0.250 to 0.300	0.009	cone, green	0	2.0	0.03	2.0	#2001/ 1015050
					cone, green	13	1.2	0.11	1.3	
					cone, green	20	1.3	0.09	1.4	
					cone, green	26	1.1	0.08	1.2	
					cone, dried	20	7.4	0.93	8.4	
					cone, dried	26	2.4	0.49	2.9	
Germany[1] 2000 (RF 0200)	BAS51601 F	3	0.250 to 0.300	0.009	cone, green	0	2.3	< 0.02	2.3	#2001/ 1015050
					cone, green	13	1.7	0.08	1.8	
					cone, green	20	0.95	0.07	1.0	
					cone, green	26	0.5	0.04	0.55	
					cone, dried	20	5.1	0.76	5.9	
					cone, dried	26	3.8	0.59	4.4	
Germany[1] 2000 (RF 0300)	BAS51601 F	3	0.250 to 0.300	0.009	cone, green	0	2.6	0.07	2.7	#2001/ 1015050
					cone, green	13	1.5	0.17	1.7	
					cone, green	20	0.97	0.13	1.1	
					cone, green	26	1.4	0.13	1.5	
					cone, dried	20	3.5	0.96	4.5	
					cone, dried	26	2.3	0.67	2.9	
Germany[1] 2000 (RF 0400)	BAS51601 F	3	0.250 to 0.300	0.009	cone, green	0	7.2	0.12	7.3	#2001/ 1015050
					cone, green	13	1.5	0.23	1.7	
					cone, green	20	1.6	0.50	2.1	
					cone, green	26	0.35	0.07	0.41	
					cone, dried	20	3.4	0.49	3.9	
					cone, dried	26	4.5	1.16	5.7	
Germany[1] 2001 (RF 0201)	BAS51601 F	3	0.210 to 0.250	0.009	cone, green	0	4.0	0.04	4.0	#2001/ 1015052
					cone, green	14	0.58	0.08	0.67	
					cone, green	21	0.91	0.10	1.0	
					cone, green	28	0.37	0.05	0.42	
					cone, dried	21	1.4	0.12	1.5	
					cone, dried	28	1.7	0.11	1.8	
Germany[1] 2001 (RF 0301)	BAS51601 F	3	0.210 to 0.250	0.009	cone, green	0	1.3	0.03	1.3	#2001/ 1015052
					cone, green	14	0.45	0.06	0.51	
					cone, green	21	0.41	0.10	0.52	
					cone, green	28	0.05	0.04	0.09	
					cone, dried	21	1.0	0.09	1.1	
					cone, dried	28	1.1	0.09	1.2	
Germany[1] 2001 (RF 0401)	BAS51601 F	3	0.210 to 0.250	0.009	cone, green	0	4.0	0.11	4.1	#2001/ 1015052
					cone, green	14	2.0	0.30	2.3	
					cone, green	21	1.0	0.21	1.2	
					cone, green	28	0.76	0.12	0.88	
					cone, dried	21	7.2	0.47	7.6	
					cone, dried	28	2.9	0.27	3.2	
Germany[1] 2001 (RF 0501)	BAS51601 F	3	0.210 to 0.250	0.009	cone, green	0	1.7	< 0.02	1.7	#2001/ 1015052
					cone, green	14	0.24	0.04	0.28	
					cone, green	21	0.44	0.04	0.48	
					cone, green	28	0.15	< 0.02	0.17	
					cone, dried	21	2.1	0.10	2.2	
					cone, dried	28	1.3	0.05	1.3	

CROP	Application				Residues [mg/kg]					Ref. Report No
Country/ year trial code	Formulation	No.	kg ai/ha	kg ai/hL	Matrix	Day	Parent	500M07	Total residue	
Germany[1]	BAS51601 F	3	1)	0.0091	cone, green	0	4.6	0.100	4.7	#2003/
2003			0.210	0.0093	cone, green	14	0.78	0.069	0.84	1001292
(AGR/16/03)			2+3)		cone, green	21	0.98	0.101	1.1	
			0.250		cone, green	28	1.5	0.152	1.7	
BAS 516 04 F		3	1)	0.0095	cone, green	0	2.1	0.036	2.1	
			0.218	0.0095	cone, green	14	1.5	0.048	1.6	
			2+3)		cone, green	21	1.8	0.050	1.9	
			0.250		cone, green	28	1.9	0.085	2.0	
Germany[1]	BAS51601 F	3	1)	0.0091	cone, green	0	5.81	0.13	5.9	#2003/
2003			0.210	0.0093	cone, green	14	2.17	0.32	2.5	1001292
(AGR/17/03)			2+3)		cone, green	21	2.2	0.25	2.4	
			0.250		cone, green	28	0.56	0.11	0.67	
BAS 516 04 F		3	1)	0.0095	cone, green	0	11	0.09	11	
			0.218	0.0095	cone, green	14	7.2	0.28	7.5	
			2+3)		cone, green	21	2.5	0.15	2.6	
			0.250		cone, green	28	3.5	0.16	3.7	
Germany[1]	BAS51601 F	3	1)	0.0091	cone, green	0	4.2	0.15	4.3	#2003/
2003			0.210	0.0093	cone, green	14	4.2	0.52	4.8	1001292
(AGR/18/03)			2+3)		cone, green	21	1.9	0.33	2.2	
			0.250		cone, green	28	0.98	0.17	1.2	
BAS 516 04 F		3	1)	0.0095	cone, green	0	6.5	0.13	6.6	
			0.218	0.0095	cone, green	14	4.2	0.21	4.4	
			2+3)		cone, green	21	6.8	0.33	7.1	
			0.250		cone, green	28	2.0	0.12	2.1	
Germany[1]	BAS51601 F	3	1)	0.0091	cone, green	0	5.6	0.25	5.8	#2003/
2003			0.210	0.0093	cone, green	14	2.0	0.29	2.2	1001292
(AGR/19/03)			2+3)		cone, green	21	4.7	0.67	5.4	
			0.250		cone, green	28	0.77	0.15	0.92	
BAS 516 04 F		3	1)	0.0095	cone, green	0	6.9	0.17	7.1	
			0.218	0.0095	cone, green	14	2.0	0.13	2.1	
			2+3)		cone, green	21	5.0	0.29	5.2	
			0.250		cone, green	28	3.7	0.21	4.0	
USA, WA	BAS50002 F	3	0.25	0.036	cone, dried	0	22	0.33	22	#001/
2001					cone, dried	7	9.1	0.38	9.4	5002574
					cone, dried	14	9.3	0.56	9.9	
USA, ID	BAS50002 F	3	0.25	0.053	cone, dried	0	19	0.32	19	
2001					cone, dried	7	16	0.44	16	
					cone, dried	14	11	0.46	12	
USA OR	BAS50002 F	3	0.25	0.033	cone, dried	0	16	0.29	16	
2001					cone, dried	7	4.9	0.30	5.2	
USA, WA	BAS50002 F	3	0.25	0.0133	cone, dried	0	19.	0.24	20	
2001					cone, dried	7	13	0.54	14	
					cone, dried	14	7.4	0.47	7.9	
USA, ID	BAS50002 F	3	0.25	0.018	cone, dried	0	18	0.42	18	
2001					cone, dried	7	12	0.48	12	
					cone, dried	14	7.8	0.42	8.2	
USA OR	BAS50002 F	3	0.25	0.018	cone, dried	0	5.6	0.18	5.7	
					cone, dried	7	7.6	0.45	8.0	

1. BAS 516 01 F was used for the treatments

PROCESSING

Hops

Reports of two processing studies were made available for the meeting (Schulz H., 2002, BASF 2001/1015048, BASF 2001/1015049). Hops were treated in the Netherlands and in Germany three times with 0.097-0.113 kg ai/ha. Samples were taken 20–22 days after last application (GAP 3 × 0.057–0.25 kg ai/ha and PHI of 21 days).

The green cone samples were dried and processed according to general industrial practice in a pilot plant in Germany.

The residues in dried cones and beer were:

Sample	Residue mg/kg	Processing factor
Dried cone	1.57	
Beer	< 0.04	< 0.023
Dried cone	4.75	
Beer	< 0.04	< 0.008

The estimated processing factor for beer is < 0.0156.

Soybean

A single field trial was conducted with 5 × maximum recommended rate in order to obtain detectable residues in soybean seed (Versoi P.L, Scott Malinsky D., BASF 5002529). The two treatments were performed with 1.12 kg ai/ha 7 days apart, and samples were taken 13 days after the second application.

The harvested seeds were processed at laboratory scale simulating the commercial practice. Analytical method D 9908 was used to determine the residues in RAC and processed fractions.

There were no detectable 500M07 residues in processed fractions. The detected residues in RAC and processed fractions are shown in Table 30.

Table 30. Residues of pyraclostrobin in soybean and processed fraction.

Sample	Pyraclostrobin [mg/kg]	Processing factor[1]	Average processing factor
Soybean seed	0.04, 0.03		
Hull	0.05, 0.05	1.25, 1.67	1.46
Meal	< 0.02, < 0.02	< 0.4, < 0.67	0.53
Refined oil	< 0.02, < 0.02	< 0.4, < 0.67	0.53

1. Calculated from the residues of parent pyraclostrobin

Sunflower

Sunflower seeds derived from crops treated with 5X recommended rate, were processed to meal and refined oil (Versoi P. L., Abdel-Baky S., BASF 2001/5002552). The scheme of processing is shown in Figure 1. The results are summarised in Table 31.

Table 31. Residues in sunflower seed, meal and refined oil.

Sunflower Matrix[1]	Residues (mg/kg)			Concentration Factor
	Parent	500M07	Total	
Seed, RAC	1.4 0,0.63	0.35,0.17	1.75,0.80(1.38)	-
Meal	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04, < 0.04 (< 0.04)	< 0.00985
Refined oil	< 0.02, < 0.02	< 0.02, < 0.02	< 0.04, < 0.04 (< 0.04)	< 0.00985

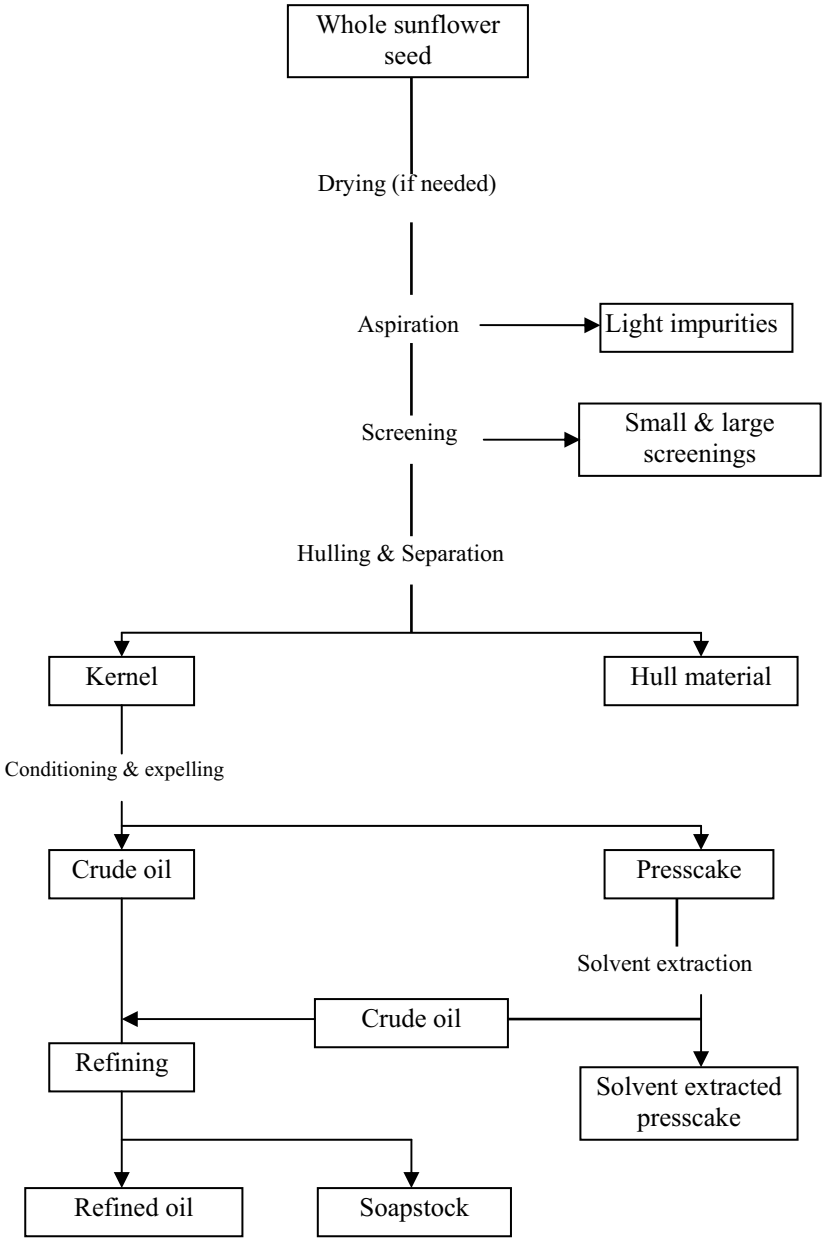

Figure 1. Flow diagram for processing sunflower seed.

RESIDUE AND ANALYTICAL ASPECTS

Pyraclostrobin was evaluated by the JMPR in 2003 and an ADI of 0-0.03 mg/kg bw per day and an ARfD of 0.05 mg/kg bw per day were established. The 2004 JMPR defined the residues as parent compound for compliance with MRLs and for dietary intake calculations, and estimated a number of maximum residue levels in various commodities.

Additional information on registered uses and results of supervised trials were submitted for evaluation. The Meeting evaluated the new data together with those included in the 2004 evaluation for those commodities only for which recommendations were not made by the 2004 JMPR.

The samples were analysed with analytical methods based on LC/MS/MS detection providing an LOQ of 0.02 mg/kg for pyraclostrobin and its major metabolite 500M07 (BF500-3, methyl-N-[[[1-(4-chlorophenyl)-pyrazol-3-yl]oxy]-o-tolyl]carbamate). The methods are described in detail in the

2004 Evaluations. The applicability of the methods was confirmed with concurrent recovery tests in each study. The average recoveries were typically between 80 and 99% for pyraclostrobin and 500M07. No interference of plant matrices was observed in most of the studies.

The storage intervals of samples from sampling to analysis were within the period covered by the storage stability tests reported by the 2004 JMPR.

Results of supervised trials on crops

Apple

A total of 25 field trials were conducted in different representative apple growing areas in Belgium, Germany, France, Italy and the Netherlands according to corresponding GAP, i.e., 3–4 applications at 0.067–0.1 kg ai/ha with a PHI of 6–8 days.

The residues determined were: 0.03, 0.034, 0.041, 0.051, 0.057, 0.058, 0.064, 0.07, 0.07, 0.081, 0.095, 0.101, 0.104, 0.118, 0.12, 0.131, 0.139, 0.142, 0.143, 0.163, 0.167, 0.184, 0.276, 0.289, 0.29 mg/kg.

The 2004 JMPR reported Brazilian trials conducted with 0.15 and 0.3 kg ai/ha which are 1.5× and 3× above the Brazilian GAP rate. The residues in apples ranged from < 0.02 to 0.38 mg/kg 14 days after the last of four treatments with 0.15 kg ai/ha.

Taking into account that early applications do not affect the residues and based on the residue data derived from trials performed in accordance with the European GAP, the Meeting estimated a maximum residue level of 0.5 mg/kg, HR of 0.29 mg/kg and STMR level of 0.104 mg/kg.

Raspberry

Nine trials were carried out in accordance with US GAP (four applications at 0.196 kg ai/ha with a 0 day PHI). The residues in mature fruit were: 0.5, 0.51, 0.63, 0.78, 0.78, 0.89, 0.94, 1.03, 1.28 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg, HR of 1.28 mg/kg and STMR value of 0.78 mg/kg for raspberry.

Stone fruits

The 2004 JMPR evaluated numerous trials carried out in USA and estimated maximum residue levels for peaches (0.5 mg/kg), cherries (1 mg/kg) and plums (0.3 mg/kg). The pyraclostrobin residues from European trials performed according to Hungarian and Italian GAP were also reported. They ranged between < 0.02–0.21 mg/kg for cherry (n = 16), < 0.02–0.1 mg/kg for plum (n = 13) and < 0.02–0.13 for peach (n = 14). These residues are covered by the maximum residue levels estimated by the JMPR based on US residue trial data.

No residue trials on apricot was reported but as apricot is now included on the label in Canada and USA (5 applications at 0.134 kg ai/ha, with 10 and 0 day PHI respectively) and Hungary (2–3 applications at 0.067 kg ai/ha, with a 7 day PHI), the Meeting concluded that maximum residue levels for stone fruit can be estimated taking into account the cherry residues reported by the 2004 JMPR. Pyraclostrobin residues in cherries from 12 US trials were 0.25 (2), 0.27, 0.34, 0.38, 0.42, 0.43, 0.48, 0.50 (2), 0.51, 0.63 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg, HR of 0.63 mg/kg and a STMR of 0.43 mg/kg for stone fruits, and withdraws its previous recommendations made for cherry, peach and plum (including prunes).

Leek

Eleven supervised field trials were performed on leeks according to GAP in Belgium and The Netherlands (maximum of 3 applications at 0.1 kg ai/ha with a PHI of 14 days). The corresponding residues were 0.05, 0.12, 0.15, 0.16, 0.19, 0.22(2), 0.24, 0.25, 0.29, 0.42 mg/kg.

The meeting estimated a maximum residue of 0.7 mg/kg, HR of 0.42 and an STMR of 0.22 mg/kg.

Brassica vegetables

Broccoli and cauliflower

Thirteen field trials were conducted in different representative cauliflower and broccoli growing areas in Europe consisting of three applications at a rate of 0.067 kg ai/ha of pyraclostrobin. The applications took place at about 28, 21 and 14 days prior to harvest.

The residues in cauliflower at about 14 days after the last application (corresponding to GAP in several EU countries) were: < 0.02 (6), 0.04 mg/kg.

The residues in broccoli at about 14 days after last application were: < 0.02 (5), 0.03 mg/kg.

The medians of residue populations in broccoli and cauliflower are not significantly different and the residue data can be combined.

The combined residues are: < 0.02 (11), 0.03, 0.04 mg/kg.

Trials were also performed with 0.1 kg ai/ha (1.5× recommended rate) and resulted in somewhat higher residues: < 0.02 (8), 0.04, 0.04, and 0.18 mg/kg.

Based on the GAP of 0.067 kg ai/ha dose, the Meeting estimated a maximum residue level of 0.1 mg/kg, HR of 0.04 mg/kg and STMR of 0.02 mg/kg for flowerhead brassicas.

Brussels sprouts

Nine field trials were conducted in Brussels sprouts in the United Kingdom, The Netherlands, Denmark, Germany, Sweden and France. Three applications were made according to GAP at rates of 0.067 kg ai/ha for pyraclostrobin. The samples taken at around 14 days contained residues of: < 0.02 (3), 0.03 (2), 0.06, 0.08, 0.1, and 0.14 mg/kg.

Trials were also carried out at a rate of 0.1 kg/ha (1.5× GAP). Samples taken at around 14 days after last application contained residues of: 0.04, 0.06, 0.06, 0.07, 0.11, 0.12, 0.13, and 0.21 mg/kg.

The Meeting took into account the residues derived from applications performed according to GAP and estimated a maximum residue level of 0.3 mg/kg, HR of 0.14 mg/kg and an STMR of 0.03 mg/kg.

Cabbage

Fifteen field trials were conducted in different representative head cabbage growing areas in the EU. The cabbages were treated with pyraclostrobin in accordance with GAP, i.e., three applications at a rate of 0.067 kg ai/ha.

A further 12 field trials were conducted with about four applications at a rate of 0.1 kg ai/ha, 1.5× the GAP rate. The residues ranged between non-detected (LOQ = 0.02 mg/kg) and 0.08 mg/kg.

The samples taken at around the PHI of 14 days from fields treated according to GAP contained residues of: < 0.02 (11), 0.04, 0.05, 0.09, and 0.09 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg, HR of 0.09 mg/kg and STMR of 0.02 mg/kg.

Fruiting vegetables

Cucumber

Supervised field trials were conducted at eight sites in the USA applying six sequential applications (7 ± 1 day apart) at a rate of 0.224 kg ai/ha and a total seasonal rate of 1.34 kg ai/ha. The US GAP allows four applications at a rate of 0.224 kg ai/ha.

The samples collected at 0 day PHI contained residues of: 0.03, 0.05, 0.06, 0.07, 0.09, 0.12, 0.14, and 0.41 mg/kg.

The Brazilian GAP specifies 4 sequential applications at rate of 0.1 kg ai/ha and a PHI of 7 days. Four trials carried out according to GAP resulted in residues below the LOQ (0.02 mg/kg) in all samples.

The medians of the two residue populations are different and were not combined.

The Meeting noted that cucumber is a rapidly growing crop and the early applications are made when the fruits are not present on the plants, therefore the residue pattern is not affected by the early treatments. Consequently, the Meeting considered that residue values derived from six sequential applications could be used, and estimated a maximum residue level of 0.5 mg/kg, HR of 0.41 mg/kg and an STMR of 0.08 mg/kg for cucumber.

Cantaloupe

In eight US trials pyraclostrobin was applied six times at a rate of 0.224 kg ai/ha, corresponding to US GAP. The residues in found directly after last application (day 0) were: 0.05, 0.08, 0.09, 0.1, 0.11, 0.12, 0.12, and 0.13 mg/kg.

The Meeting estimated for cantaloupe a maximum residue level of 0.2 mg/kg, HR of 0.13 mg/kg and STMR of 0.105 mg/kg.

Peppers

Seven field and six greenhouse trials were conducted on peppers with three applications at a rate of 0.1 kg ai/ha in Europe according to Italian GAP. The residues of pyraclostrobin in fruit samples collected 2–3 days after the final application ranged between < 0.02 and 0.25 mg/kg. There was no significant difference between the residue populations of field grown or greenhouse grown peppers.

The combined residues were: 0.03(4), 0.06, 0.07, 0.08, 0.09, 0.13(4) and 0.30 mg/kg.

The residues of pyraclostrobin from European trials were lower than the residues reported from trials conducted according to US GAP (six applications at 0.224 kg ai/ha with a 0 day PHI): 0.14, 0.22, 0.82 mg/kg. The two residue populations have different median values and cannot be combined.

The Meeting concluded that the residue data base reflecting the higher residue population derived from US GAP was not sufficient for estimating maximum residue level for bell peppers or chilli peppers, and used the results of trials performed according to maximum GAP in Europe. The Meeting estimated a maximum residue of 0.5 mg/kg, HR of 0.30 mg/kg and STMR of 0.08 mg/kg.

Eggplant

The 2004 JMPR estimated a maximum residue level for tomatoes of 0.3 mg/kg, an HR of 0.21 mg/kg and an STMR of 0.12 mg/kg for outdoor application based on the US GAP.

Twenty six field and greenhouse trials performed according to the GAP in Poland (three applications at a rate of 0.067–0.1 kg ai/ha with a PHI of 3 days) resulted in residues 2–3 days after the final application in the ranges of < 0.02 to 0.13 mg/kg. There was no significant difference between the residue populations of field and greenhouse tomatoes.

The residue levels estimated, based on the critical US GAP, covers the residues obtained in European trials.

Since the evaluation in 2004, US and Canadian labels authorising the use of the compound on eggplant became available (six applications at 0.224 kg ai/ha with a 0 day PHI) which is the same as that for tomato. Furthermore, the Meeting noted that there was no difference between residues derived from outdoor and protected growing conditions of tomato.

The Meeting concluded that the residue levels estimated for tomato can be applied for eggplant as well, and estimated a maximum residue level of 0.3 mg/kg, an HR of 0.21 mg/kg and a STMR of 0.12 mg/kg.

Kale

In the United Kingdom pyraclostrobin is registered for use as three applications at a rate of 0.067 kg ai/ha and a PHI of 14 days. Six field trials were conducted in curly kale in Denmark, the UK, the Netherlands and Sweden with four applications at 0.1 kg ai/ha. The applications were done about 5, 4, 3 and 2 weeks prior to commercial harvest. Samples were taken from 0 to 20–21 days after final application.

The residues in samples taken at 14 days were: 0.02, 0.06, 0.09, 0.26, 0.31, and 0.61 mg/kg.

The meeting considered that the early application does not affect the residues at harvest, and estimated a maximum residue level of 1 mg/kg, HR of 0.61 mg/kg and STMR of 0.175 mg/kg for kale.

Lettuce, head

In USA, pyraclostrobin has approval in lettuce for four applications at 0.117–0.23 kg ai/ha with a 0 day PHI. Supervised field trials performed on head lettuce with four applications at 0.224 kg ai/ha rate resulted in residues of: 1.95, 3.69, 4.96, 13.7, 14.9, and 19.7 mg/kg.

Seventeen field trials were carried out in typical growing regions of Europe according to GAP (two applications at 0.1 kg ai/ha and PHI of 14 days). Samples collected at around 14 days contained residues of: < 0.02 (6), 0.03, 0.04(4), 0.06, 0.08(3), 0.28, and 0.38 mg/kg.

Eight trials on lettuce were performed in greenhouse according to European GAP. The residues detected in lettuce head 14 days after the last application were: 0.03, 0.04, 0.13, 0.23, 0.29, 0.33, 0.75, and 0.81 mg/kg.

The US GAP would lead to an estimated maximum residue level of 40 mg/kg, an HR value of 19.7 mg/kg and a median residue of 9.33 mg/kg for lettuce head. This residue level would result in an estimated intake of 390% of the ARfD.

Consequently, in accordance with the principles of alternative GAP as described in Section 2.2, the Meeting considered the next lowest GAP and used the residues in greenhouse lettuce treated according to the European GAP for the estimation of maximum residue level of 2 mg/kg, HR of 0.81 mg/kg and an STMR of 0.26 mg/kg for lettuce head.

Snap beans

The 2004 JMPR was not able to recommend a maximum residue level for snap beans as there was no GAP at that time. The present meeting was provided with the US Label (GAP: two applications at 0.087–0.13 kg ai/ha dose with a 7 day PHI).

The nine field trials reported to the 2004 JMPR were performed with a rate of 0.224 kg ai/ha for individual treatments and total seasonal applied amount of 0.448 kg ai/ha.

The residues of pyraclostrobin in ranked order were: < 0.02, 0.04, 0.08, 0.1, 0.1, 0.11, 0.12, 0.13, 0.16 mg/kg.

As all trials were performed with a dosage corresponding to 1.7 × maximum rate, the Meeting agreed that maximum residue level for snap beans could not be estimated.

Peas

A total of 21 field trials were conducted in vining peas in France, the United Kingdom, Germany, Denmark and Sweden applying pyraclostrobin twice with a target rate of 0.067 and 0.1 kg ai/ha. The samples were taken at earliest commercial harvest (corresponding to approximately 8–14 days after the last application). In all green pea samples the residues were below the LOQ of 0.02 mg/kg. As the PHI in France is 35 days, no residues can be expected in green peas.

The Meeting estimated for green peas a maximum residue level, HR and STMR values of 0.02* and 0.02, 0.02 mg/kg, respectively.

Soybean

The US GAP consists of two applications at a rate of 0.1–0.2 kg ai/ha with a PHI of 21 days and a seasonal maximum of 0.41 kg ai/ha. In 17 field trials the rate of pyraclostrobin applied was double that of the GAP with a total application rate of 0.448 kg ai/ha/season. The immature seeds were harvested at five days contained residues in the range of < 0.02 and 0.3 mg/kg.

The mature seeds collected from the 17 trials, sampled 28 days after the second application, were found to not contain any detectable residues (LOQ of 0.02 mg/kg).

In eight trials performed approximating Brazilian GAP (two applications at 0.075 kg ai/ha with a 14 day PHI) the residues were: < 0.02 (7) and 0.03 mg/kg.

As the majority of samples (84%) contained non-detectable residues at day 5 post-application, and no residue was detectable in mature seeds, using this supportive information the Meeting concluded that the Brazilian data and GAP enable the estimation of maximum residue limits of 0.05 mg/kg, and STMR values 0.02 mg/kg for soybeans.

Spelt

No special residue trials were performed for spelt. However, as spelt is a registered crop in Belgium and Luxembourg with the same GAP as wheat, the Meeting concluded that the residue levels estimated by the 2004 JMPR for wheat grains are applicable to spelt as well.

Sunflower seed

The US GAP allows two applications with 0.1–0.2 kg ai/ha applied at 7–14 days intervals with a 21 day PHI. Field trials were performed by applying 0.224 kg ai/ha twice and collecting samples 21 days after the final application. The residues in ranked order were: < 0.02, 0.02, 0.04, 0.05, 0.06, 0.06, 0.1, and 0.22 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.055 mg/kg for sunflower seed.

Coffee

To complement the data submitted to the 2004 JMPR, additional field trials were carried out in Brazil with target rates of 0.1 kg ai/ha and 0.2 kg ai/ha (GAP is 0.2 kg ai/ha). The coffee bean samples, collected at full ripening stage (red coffee berry), were taken 45 days after the last application and contained residues of: < 0.02 (4), < 0.02, 0.03, 0.03, 0.11, 0.15, and 0.15 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg, and an STMR of 0.025 mg/kg for coffee beans.

Hops

A total of 12 field trials were conducted in the representative areas for hop cultivation in Germany with application rates of 0.21 to 0.30 kg ai/ha. Green hop cones were sampled immediately after the last application and at about 14, 21 and 28 days later.

During the last two samplings the collected green cones were divided into two parts. One part was deep-frozen and the other part was dried for 6 hours at 60°C and was then deep-frozen on the following day.

The residues in dried cones were: 1.1, 1.7, 2.1, 3.5, 4.5, 5.1, 7.2, and 7.4 mg/kg

The formulations and the spray volumes used did not have any observable effect on the magnitude of residues.

Six field trials were conducted in the US, where there is no GAP, with three applications at approximately 0.25 kg ai/ha applied in concentrate (low-volume) and dilute (high-volume) spray solutions. Hop cone samples were taken 0, 7 and 14 days after the last application. These were dried on the field prior to shipment for analysis. The residues in dried cones taken at day 14 were: 7.4, 7.6, 7.8, 9.3, 11 and 12 mg/kg.

The Meeting considered the residues determined in dried cones in German trials, and estimated a maximum residue of 15 mg/kg, and an STMR of 4.0 mg/kg for dried hop cone.

Animal feed commodities

Soybean forage

Seventeen field trials were performed in USA according to GAP. The label specifies a minimum of 14 day interval between last application and feeding forage to animals. The residues in soybean forage at day 14 after the last application were: 0.75, 0.82, 0.90, 1.01, 1.10, 1.24, 1.30, 1.34, 1.60, 1.69, 1.75, 1.85, 2.37, 2.70, 2.74, 2.81, and 3.22 mg/kg.

The Meeting estimated highest residue of 3.22 mg/kg and an STMR of 1.6 mg/kg for soybean forage.

Fate of residue during processing

Hops

Hops were treated three times at a rate of 0.097–0.113 kg ai/ha in the Netherlands and Germany. Samples were taken 20–22 days after the final application (GAP 3 applications at 0.057–0.25 kg ai/ha with a PHI of 21 days). The green cone samples were dried and processed in a pilot plant in Germany.

The beer obtained from dried hops containing 1.57–4.75 mg/kg pyraclostrobin did not contain any detectable residues (< 0.04 mg/kg). The Meeting estimated an average processing factor of < 0.0156. Based on the STMR for hops (3.5 mg/kg), the estimated STMR-P for beer is 0.055 mg/kg.

Soybean seed

A single field trial was conducted applying pyraclostrobin at five times the maximum recommended rate (two applications of 1.12 kg ai/ha, 7 days apart) in order to obtain detectable residues in soybean seed. The harvested seeds underwent laboratory scale processing that simulated commercial practice. The average processing factors for hull was 1.46. The meal and refined oil did not contain any detectable residues. The calculated processing factor was 0.58 for both commodities. Based on the STMR for soybean (0.02 mg/kg), the estimated STMR-P for refined soybean oil is 0.012 mg/kg.

Sunflower seed

Sunflower seeds derived from crops treated twice with pyraclostrobin at five times the maximum recommended rate, were processed to meal and refined oil. The meal and refined oil did not contain any detectable residues resulting in a processing factor of < 0.014. Based on the STMR for sunflower (0.055 mg/kg), the estimated STMR-P for refined oil is 0.00077 mg/kg.

Farm animal dietary burden

The animal dietary burden estimated by the 2004 JMPR is based on peanut hay (7.28–14.4 mg/kg) and the cereal fodder (5.4–10.8 mg/kg) and is substantially larger than what would be expected from peanut forage (0.97 mg/kg) or feeding leafy vegetables. The farm animal dietary burden estimated by the 2004 JMPR is therefore not affected by the potential use of treated soybean, kale and other vegetables as animal feed.

RECOMMENDATION

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

The definition of the residue for compliance with MRL and for dietary intake estimation is; *pyraclostrobin.*

Summary of recommendations for MRLs, STMRs and HRs for

CCN	Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR/P mg/kg
		New	Previous		
FP0226	Apple	0.5		0.104	0.29
	Beer			0.055	
VB0402	Brussels sprouts	0.3		0.03	0.14
VB0041	Cabbage, head	0.2		0.02	0.09
VC 4199	Cantaloupe	0.2		0.105	0.13
FS0013	Cherry	W	1		
SB0716	Coffee beans	0.3		0.025	
VC0424	Cucumber	0.5		0.08	0.41
VO 0440	Eggplant	0.3		0.12	0.21
VB 0042	Flowerhead brassica	0.1		0.02	0.04
DH1100	Hops, dry	15		4	
VL0480	Kale	1		0.175	0.61
VA0384	Leek	0.7		0.22	0.42
VL0482	Lettuce, head	2 [a]		0.26	0.81
FS0247	Peach	W	0.5		
VP0064	Peas (immature succulent seeds)	0.02*		0.02	0.02
VO0051	Peppers	0.5		0.08	0.25
FS0014	Plum	W	0.3		
FB0272	Raspberry	2		0.78	1.28
VD0541	Soya bean (dry)	0.05		0.02	
	Soya bean oil, refined			0.012	
GC4673	Spelt	0.2		0.02	0.09
FS0012	Stone fruits	1		0.43	0.63
SO0702	Sunflower seed	0.3		0.055	

CCN	Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR/P mg/kg
		New	Previous		
OR0702	Sunflower seed oil, (refined)			0.00077	

a Estimated figures are based on a lower alternative GAP.

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes of pyraclostrobin, based on the STMRs estimated for 59 commodities included those which were evaluated by the 2004 JMPR for the 13 GEMS/Food regional diets were in the range of 0 to 7% of the maximum ADI (0.03 mg/kg bw per day). The Meeting concluded that the long-term intake of residues of pyraclostrobin resulting from its uses that have been considered by JMPR is unlikely to present a public health concern.

Short-term intake

The IESTI of pyraclostrobin calculated on the basis of the recommendations made by the JMPR represented 0–80% of the ARfD (0.05 mg/kg bw) for children and 0–30% for the general population.

The Meeting concluded that the short-term intake of residues of pyraclostrobin resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

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QUINOXYFEN (222)

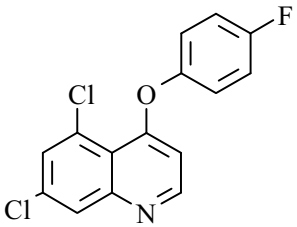
First draft prepared by Stephen Funk, Health Effects Division, US Environmental Protection Agency, Washington, DC, USA

EXPLANATION

Quinoxyfen is a fungicide used for protection against powdery mildew diseases on a variety of crops. At the 36th session of the CCPR (ALINORM 04/27/24), quinoxyfen was listed as a candidate for evaluation of a new compound by the 2006 JMPR. It was furthermore identified as a candidate for work sharing.

The sponsor has submitted study reports on the metabolism in lactating goats, wheat, sugar beet, grapes, cucumber, and tomato, on the environmental fate (aerobic soil metabolism, hydrolytic degradation, photochemical degradation, and confined rotational crops), on analytical methods for the determination of quinoxyfen in various matrices, and on the storage stability of quinoxyfen in stored frozen analytical samples. Crop field trials were supplied for cherries, grapes, strawberries, currants, melons, peppers, lettuce, wheat, barley, hops, sugar beets and sugar beet tops. GAP information (labels) was also supplied. Processing studies were submitted for wheat, barley, and grapes, and dairy cow and laying hen feeding studies were provided. National use information was supplied by Australia, the Netherlands, and the USA. Germany provided field trial data for currants.

IDENTITY

ISO common name:	Quinoxyfen
IUPAC name:	5,7-dichloro-4- quinolyl 4 fluorophenyl ether
Chemical Abstract name:	5,7-dichloro-4-(4-fluorophenoxy)quinoline
CAS No.:	124495-18-7
CIPAC No.:	566
Synonyms:	XR-795; XDE-795; DE-795
Molecular Formula:	$C_{15}H_8Cl_2FNO$
Structural Formula:	
Molecular Weight:	308.14
Minimum purity	97%

PHYSICAL AND CHEMICAL PROPERTIES

Pure Active Ingredient:

Chemical/physical property	Results		Reference
Appearance	Off-white flocculent solid		21129, Cowlyn and Boothroyd, 1994
Melting point	106 – 107.5ºC		
Relative density	1.56		
Solvent solubility, 20ºC	Hexane Methanol Toluene	9.64 g/L 21.5 g/L 272 g/L	

Chemical/physical property	Results		Reference
	Dichloromethane 589 g/L Acetone 116 g/L Ethyl acetate 179 g/L		
Water solubility, 20ºC	pH	µg/L	8605, Cowlyn and Boothroyd, 1994
	6.45 5 7 9	116 ± 5.1 128 47 36	
Vapour pressure	1.2 x 10^{-5} Pa @ 20ºC		
	2.0 x 10^{-5} Pa @ 25ºC		
Volatility, Henry's Law Constant @ 20ºC	Calculation: 3.19 x 10^{-2} Pa.m^3. $mole^{-1}$		
Partition coefficient, 20ºC and pH 6.6	Log P_{OW} = 4.66 ± 0.002		

Technical Material

Chemical/physical property	Results	Reference
Melting point	100 - 106ºC	27304, Cowlyn, 1994
Relative density	1.49	
Solvent solubility, 20ºC	n-heptane 10.2 g/L Methanol 24.6 g/L Xylene 200 g/L Dichloromethane 236 g/L Acetone 111 g/L Ethyl acetate 138 g/L Acetonitrile 22.8 g/L n-Octanol 37.9 g/L	
Dissociation constant	pKa of protonated DE-795 = 3.63; equivalent Ka = 2.37 × 10^{-4}	

Formulations

Quinoxyfen is a protectant fungicide. In order to provide both protective and curative control, some formulations have been developed consisting of Quinoxyfen with a curative fungicide. Quinoxyfen is available in the following formulations.

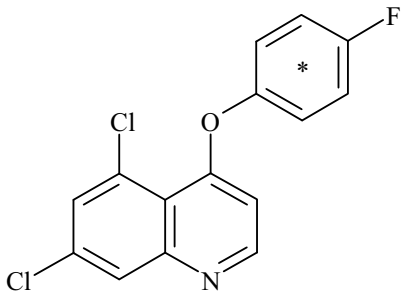
Formulation	Active ingredient content
Suspension concentrate (SC), containing only Quinoxyfen as the active ingredient	250 g/L Quinoxyfen (EF-1295)
	500 g/L Quinoxyfen (EF-1186)
Suspension concentrate (SC), containing a mixture of Quinoxyfen and Cyproconazole	200 g/L Quinoxyfen + 60 g/L Fenarimol (EF-1303)
	75 g/L Quinoxyfen + 80 g/L Cyproconazole (EF-1406)
Emulsion, oil in water (EW), containing a mixture of Quinoxyfen and Fenpropimorph	66 g/L Quinoxyfen + 250 g/L Fenpropimorph. (EF-1288)

The supervised trials reported to the Meeting used either the EF-1295, EF1186, or EF-1303 formulations.

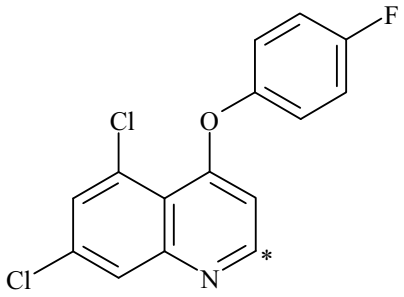
METABOLISM AND ENVIRONMENTAL FATE

The various metabolism studies were conducted using quinoxyfen radio-labelled with the ^{14}C label in the phenyl ring (phenoxy label) or on the second carbon of the quinoline ring (quinoline label).

Phenyl-Ring Labelled [^{14}C]Quinoxyfen

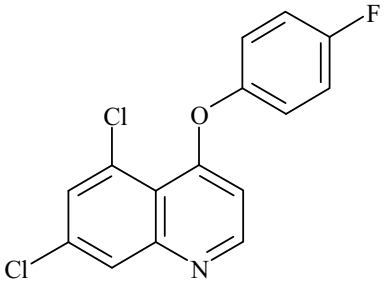


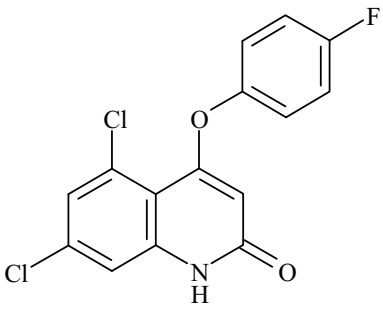
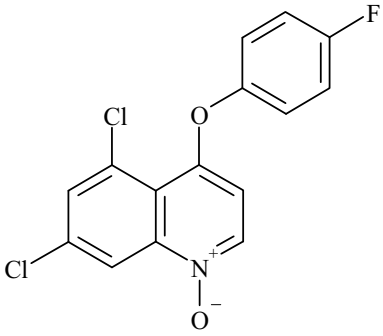
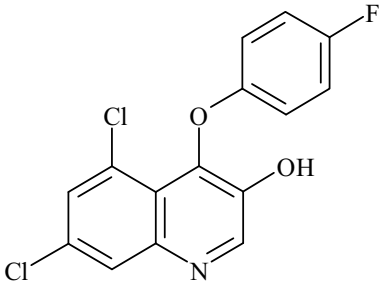
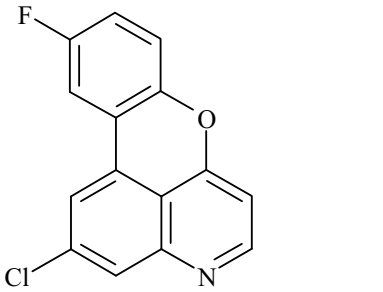
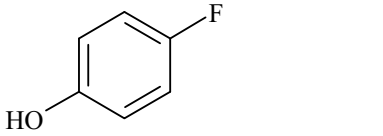
Quinoline-Ring Labelled [^{14}C]Quinoxyfen [labelled on the second carbon of the quinoline ring]

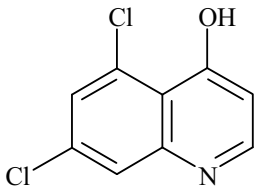


The following table summarizes the metabolites identified in the various metabolism and environmental fate studies.

Table 1: Summary of metabolites and degradates.

Common Name/Code	Chemical Name	Structure
Quinoxyfen DE-795	5,7-dichloro-4-(4-fluorophenoxy)quinoline	

Common Name/Code	Chemical Name	Structure
2-Oxo DE-795[1] 2-Oxo Quinoxyfen	5,7-dichloro-4-(4-fluorophenoxy)-2-oxo-quinoline	
DE-795 n-oxide Quinoxyfen n-oxide	5,7-dichloro-4-(4-fluorophenoxy)quinoline-N-oxide	
3-OH DE-795[1] 3-hydroxy Quinoxyfen	5,7-dichloro-4-(4-fluorophenoxy)-3-hydroxy-quinoline	
CFBPQ	2-chloro-10-fluoro(1)benzopyrano (2,3,4-de)quinoline	
4-Fluorophenol.	4-Fluorophenol	

Common Name/Code	Chemical Name	Structure
DCHQ	5,7-dichloro-4-hydroxyquinoline	

[1] For all radiolabelled studies (plant, livestock, rat and environmental fate) the metabolite identified as 3-hydroxy quinoxyfen is in fact 2-oxo quinoxyfen.

General for all radiolabelled quinoxyfen studies

The manufacturer has reported that in all studies utilizing radio-labelled quinoxyfen, a metabolite has been misidentified (18219, N. R. Pearson and G. L Reeves, 2005). The metabolite identified as 3-hydroxy quinoxyfen was subsequently positively identified as 2-oxoquinoxyfen by X-ray diffraction. Text and figures have been corrected to reflect this change of structure assignment.

Animal metabolism

Metabolism in lactating goats- adapted in part from the evaluation of Australia

The Meeting received a report on the metabolism, distribution, and elimination of ^{14}C-quinoxyfen, labelled either in the phenoxy ring or the quinoline ring, in lactating dairy goats (n=5; 51-60 kg bw), (29257, Dunsire and Paul, 1995). Two goats were orally dosed with phenoxy ^{14}C-quinoxyfen (purity > 98%), twice daily for 5 consecutive days, at a rate of 10.7 mg quinoxyfen/kg feed (equivalent to 0.20 mg ai/kg bw, 91.16 mg total over 5 days). Similarly, two goats were treated with quinoline ^{14}C-quinoxyfen, twice daily for 5 consecutive days, at a rate of 11.7 mg quinoxyfen/kg feed (equivalent to 0.21 mg ai/kg bw, 101.2 mg total over 5 days). The remaining goat was used as the untreated control animal.

Urine and faeces were collected at intervals of 24 hours until sacrifice. The metabolism cages were washed with water at the time of excreta collection and the cage washings were retained for analysis of the TRRs. Milk samples were collected prior to the first treatment with quinoxyfen, and then twice daily throughout the study period (at about 8:00 am and 4:00 pm) until animals were sacrificed. The weights of urine, faeces, cage wash and milk samples were recorded and total radioactivity were measured using liquid scintillation counting (LSC). Faecal samples were homogenised with water and subjected to combustion analysis before analysis by LSC.

Goats were sacrificed 16 hours after the final dose, and samples of the following fluids/tissues were collected for TRR analysis: whole blood, plasma, liver, kidney, skeletal muscle, subcutaneous fat, omental fat, perirenal fat, GI tract and contents, and carcass. Tissue samples were analysed for their TRR content using combustion analysis and LSC.

The distribution of quinoxyfen TRRs recovered from goats after 10 twice daily oral doses of radio-labelled quinoxyfen (approximately10 mg ai/kg feed/day) are tabulated below. Results are expressed as a percentage of administered dose, and as µg equiv/kg (where applicable).

Table 2. Distribution of the radiolabelled residue in the body fluids and tissues of goats (29257).

Sampling time/period (h)	% Administered (µg equiv./kg)				
	Control	Phenoxy ^{14}C-quinoxyfen		Quinoline ^{14}C-quinoxyfen	
	Animal 1	Animal 2	Animal 3	Animal 4	Animal 5
Urine					
0-24	0.0	5.8	1.2	1.6	1.20
24-48	0.0	8.3	14.	2.5	2.4
48-72	0.0	7.9	9.1	2.8	0.05
72-96	0.0	9.1	9.8	3.0	5.7
96-120	0.0	9.2	10.	2.8	2.9
Subtotal	***NA***	***40.***	***44.***	***13.***	***12.***
Faeces					
0-24	0.0	3.2	0.86	4.5	1.4
24-48	0.02	8.5	6.1	13.	10.
48-72	0.01	7.1	15.	7.0	12.
72-96	0.01	10.	6.8	16.	21.
96-120	0.15	9.5	8.6	16.	20.
Subtotal	***NA***	***39.***	***38.***	***56.***	***65.***
Cage wash					
0-24	0.0	0.07	0.03	0.03	0.02
24-48	0.0	0.29	0.20	0.04	0.06
48-72	0.0	0.13	0.29	0.04	0.03
72-96	0.0	0.26	0.28	0.06	0.57
96-120	0.0	0.44	0.61	0.16	0.10
Subtotal	***NA***	***1.2***	***1.4***	***0.33***	***0.78***
Milk					
0-8	0.0	0.03 (28.)	0.02 (29.)	0.02 (20.1)	0.01 (26.)
8-24	0.0	0.09 (53.)	0.08 (71.)	0.06 (36.5)	0.06 (56.)
24-32	0.0	0.06 (66.)	0.07 (107.6)	0.04 (46.4)	0.03 (77.)
32-48	0.0	0.11 (68.)	0.11 (96.)	0.06 (44.6)	0.07 (72.)
48-56	0.0	0.06 (76.)	0.07 (120.)	0.04 (50.2)	0.04 (88.)
56-72	0.0	0.10 (74.)	0.12 (109.)	0.06 (48.0)	0.08 (76.)
72-80	0.0	0.10 (90.)	0.09 (140.)	0.05 (59.0)	0.05 (101.)
80-96	0.0	0.12 (75.)	0.13 (110)	0.08 (54.4)	0.07 (74.)
96-104	0.0	0.06 (81.)	0.07 (130.)	0.05 (62.5)	0.06 (120.)
104-120	0.0	0.10 (74.)	0.13 (110.)	0.06 (48.8)	0.06 (64.)
Subtotal	NA	0.83	0.89	0.52	0.53
Tissues					
Liver	0.0	0.92	0.92	0.69	1.3
Kidney	0.0	0.04	0.05	0.03	0.02
GI tract wall	0.0	3.1	3.3	6.9	2.5
GI tract contents	0.0	8.3	11.	11.76	13.
Carcass	0.01	2.1	3.4	1.7	2.3
Subtotal	***NA***	***14.***	***19.***	***21.***	***19.***
Total	**NA**	**95.**	**103**	**91**	**98**

Concentrations of total radioactivity in tissues for the phenoxy label were liver, 1.03 and 0.93 mg/kg; kidney, 0.29 and 0.34 mg/kg; muscle, 0.022 and 0.032 mg/kg; perirenal fat, 0.18 and 0.19 mg/kg; omental fat, 0.20 and 0.17 mg/kg; and subcutaneous fat, 0.12 and 0.12 mg/kg. Concentrations of total radioactivity in tissues for the quinoline label were liver, 0.94 and 1.5 mg/kg; kidney, 0.22 and 0.17 mg/kg; muscle, 0.015 and 0.032 mg/kg; perirenal fat, 0.13 and 0.32 mg/kg; omental fat, 0.12 and 0.26 mg/kg; and subcutaneous fat, 0.073 and 0.19 mg/kg. TRR levels in milk at 16 hours after the final dose were 0.074 and 0.11 mg/kg for the phenoxy label and 0.049 and 0.064 mg/kg for the quinoline label.

Samples were extracted with methanol. Liver was also subjected to protease digestion (pepsin, 0.1 M HCl, 37°C, overnight), enzyme deconjugation (β-glucuronidase with sulphatase activity) of the methanol extract, mild base hydrolysis, and/or strong base (6 N NaOH) or strong acid (6 N HCl) hydrolysis (reflux, 6 hour). Kidney was subjected to protease digestion. Some extraction efficiencies are summarized in Table 3.

Table 3. Extraction efficiencies of pooled tissue and milk samples (29257).

Matrix	Treatment Step	Extraction Efficiency (% TRR)	
		Phenoxy label	Quinoline label
Milk	Methanol extraction	85	87
Kidney	Methanol extraction	71	45
Kidney	Protease digestion	100	100
Liver	Methanol extraction	42	25
Liver	Protease digestion	100	100
Liver	1M NaOH hydrolysis at 40° C	94	92
Muscle	Methanol extraction	84	76
Omental fat	Methanol extraction	100	100
Subcutaneous fat	Methanol extraction	93	91
Perirenal fat	Methanol extraction	100	100

The TRRs in excreta (urine, faeces), milk and tissues (kidney, liver, fat) from goats were characterized and/or identified. Extracts were analyzed by TLC and HPLC, and some identifications (from urine and faeces only) were confirmed by GC/MS. Available standards (not labelled) included dichlorohydroxyquinoline (DCHQ), N-oxide quinoxyfen, 4-fluorophenol, 2-oxo quinoxyfen, and 6-hydroxy quinoxyfen. Values are expressed as a percentage of the TRR (except as noted) and as mg equiv. /kg (in parenthesis) in Table 4.

Table 4. Characterization/Identification of the radiolabelled residue in excreta, milk, and tissues (29257).

Sample matrix	Extraction method	Percentage TRR (mg equiv./kg)[1]						
		Label	Quinoxyfen	Isomeric hydroxy quinoxyfens[2]	4-FP	DCHQ	2-oxo	Not identified[4]
Urine	Direct (none)	Phenoxy	ND[3]	ND	3-6	ND	ND	89-92
		Quinoline	ND	ND	ND-21	11-12	ND	67-82
	Acid hydrolysed + methanol	Phenoxy	ND-3.5	ND-5[7]	35-58	ND	ND	25-55
		Quinoline	ND-3.5	ND-22[7]	ND-19	38-56	ND	15-38
Faeces	Methanol	Phenoxy	27-31	49-55[6]	ND-1.1	ND	ND	10-13
		Quinoline	24-26	59-62[6]	ND	2.5	ND	6-10
Milk	Methanol	Phenoxy	30-42 (0.027-0.038)	ND-0.94 (ND-0.0008)	ND (ND)	ND (ND)	ND-1.4 (ND-0.001)	36-42 (0.032-0.038)
		Quinoline	37-42 (0.021-0.023)	ND (ND)	ND (ND)	ND-3.0 (ND-0.002)	ND (ND)	38-40 (0.021-0.022)
Kidney	Methanol	Phenoxy	1.6-2.3 (0.005-0.008)	ND (ND)	ND (ND)	ND (ND)	ND (ND)	63-66[5] (0.21-0.22)
		Quinoline	2.4-3.5 (0.005-0.07)	ND (ND)	ND (ND)	ND-1.6 (ND-0.003)	ND (ND)	31-39 [5] (0.59-0.75)
Liver	Methanol	Phenoxy	ND-1.2 (ND-0.012)	ND-2.1 (ND-0.022)	ND-6.7 (ND-0.068)	ND (ND)	ND (ND)	30-34 (0.30-0.35)
		Quinoline	1.8-3.8 (0.022-0.047)	ND (ND)	ND (ND)	ND-3.2 (ND-0.041)	ND (ND)	3.8-20 (0.050-0.25)
	Enzyme deconjugate (of MeOH extract)	Phenoxy	10.5 (0.051)	3.0 (0.015)	13.2 (0.064)	ND (ND)	ND (ND)	60.4 (0.29)
		Quinoline	18.3 (0.054)	10.4 (0.031)	ND (ND)	19.7 (0.058)	ND (ND)	29.0 (0.085)
	Protease digest	Phenoxy	6-7 (0.059-0.070)	ND (ND)	ND-8.3 (ND-0.085)	ND (ND)	ND (ND)	70-74 (0.64-0.71)
		Quinoline	9-15	ND	ND	ND	ND	50-56

Sample matrix	Extraction method	Percentage TRR (mg equiv./kg)[1]						
		Label	Quinoxyfen	Isomeric hydroxy quinoxyfens[2]	4-FP	DCHQ	2-oxo	Not identified[4]
			(0.12-0.20)	(ND)	(ND)	(ND)	(ND)	(0.62-0.72)
	Protease digest + acid hydrolysis	Phenoxy	5-11 (0.043-0.099)	ND-1.2 (ND-0.011)	ND-22.4 (ND-0.201)	ND (ND)	ND-1.3 (ND-0.012)	59-68 (0.52-0.61)
		Quinoline	2-13.5 (0.019-0.128)	ND (ND)	ND (ND)	ND-15.3 (ND-0.144)	ND (ND)	48-71 (0.45-0.67)
	Protease digest + enzyme deconjugate[6]	Phenoxy	ND-74	ND-8.5	ND	ND	ND-90	ND-22.
		Quinoline	ND-84	ND-4.8	ND	5.5-7.0	ND-81	5.2-8.1
Omental fat	MeOH extract	Phenoxy	51-98 (0.099-0.19)	ND (ND)	ND (ND)	ND (ND)	ND (ND)	1.8-26. (0.004-0.050)
		Quinoline	53-96 (0.100-0.18)	ND (ND)	ND (ND)	ND (ND)	ND (ND)	ND-22 (ND-0.041)
Perirenal fat	MeOH extract	Phenoxy	70-96 (0.13-0.18)	ND (ND)	ND (ND)	ND (ND)	ND (ND)	1.2-14.4 (0.002-0.028)
		Quinoline	90-97 (0.19-0.20)	ND (ND)	ND (ND)	ND (ND)	ND (ND)	ND-3.4 (ND-0.007)
Subcutaneous fat	MeOH extract	Phenoxy	76-83 (0.096-0.10)	ND-1.5 (ND-0.002)	ND (ND)	ND (ND)	ND (ND)	3.7-5.4 (0.005-0.007)
		Quinoline	75-94 (0.061-0.076)	ND (ND)	ND (ND)	ND (ND)	ND (ND)	1.9-6.5 (0.002-0.005)

[1] 4-FP = 4-fluorophenol; DCHQ = 5,7-dichloro-4-hydroxyquinoline; 2-oxo = the 2-oxo derivative of quinoxyfen. Calculated on a %TRR basis by the JMPR reviewer using percent of radioactivity in the extract and extraction efficiency.

[2] Radioactive material that co-chromatographed (TLC, HPLC) with the N-Oxide quinoxyfen standard in faeces extracts was shown by GC-MS to be comprised of several isomeric hydroxy-quinoxyfen metabolites. Other extracts were not subjected to GC/MS/ Thus, identifications of N-oxide in various tissue extracts are not confirmed.

[3] not detected.

[4] Generally characterized as “polar material” by TLC and HPLC.

[5] Deconjugation with mixed glucuronidase/sulphatase treatment did not change the profile.

[6] Percentage of total radioactivity in the final extract.

[7] N-oxide quinoxyfen was identified by HPLC in acid-hydrolysed urine but was not confirmed by TLC.

The major component identified in liver was a conjugated form of quinoxyfen (approximately 10–15% TRR). The only components identified in kidney were quinoxyfen (approximately 3% TRR) and DCHQ (about 2% TRR), with no apparent quinoxyfen conjugates present. In both kidney and liver, major portions of the TRR were characterized as polar based on TLC and HPLC behaviour. The major component present in fat was quinoxyfen (approximately 90% TRR), while milk contained quinoxyfen (about 40% TRR) and some very polar material. Small amounts of radioactivity corresponding to 4-fluorophenol, DCHQ, and several hydroxy-quinoxyfen metabolites were also present in the liver. Small amount of radioactivity corresponding to 2-oxo quinoxyfen, DCHQ, and isomeric hydroxy quinoxyfens were found in the milk. For both labels, hydroxy metabolites and parent compound were the major components present in faeces, while urine contained mainly a polar component which was easily hydrolysed to 4-fluorophenol or DCHQ. A proposed metabolic pathway is presented in Figure 1.

The metabolism in goat and rat (7474, Schumann, 1995) are qualitatively similar. Cleavage of the ether linkage to form 4-fluorophenol and DCHQ is seen in both animals. Isomers of fluorophenyl-ring hydroxylated quinoxyfen were found in the rat (bile, faeces), whereas isomers of quinoline-ring hydroxylated quinoxyfen (2-oxo) were found in the goat metabolism study. The latter were at very low levels (< 0.1% of the administered dose for the 2-oxo quinoxyfen) in the rat.

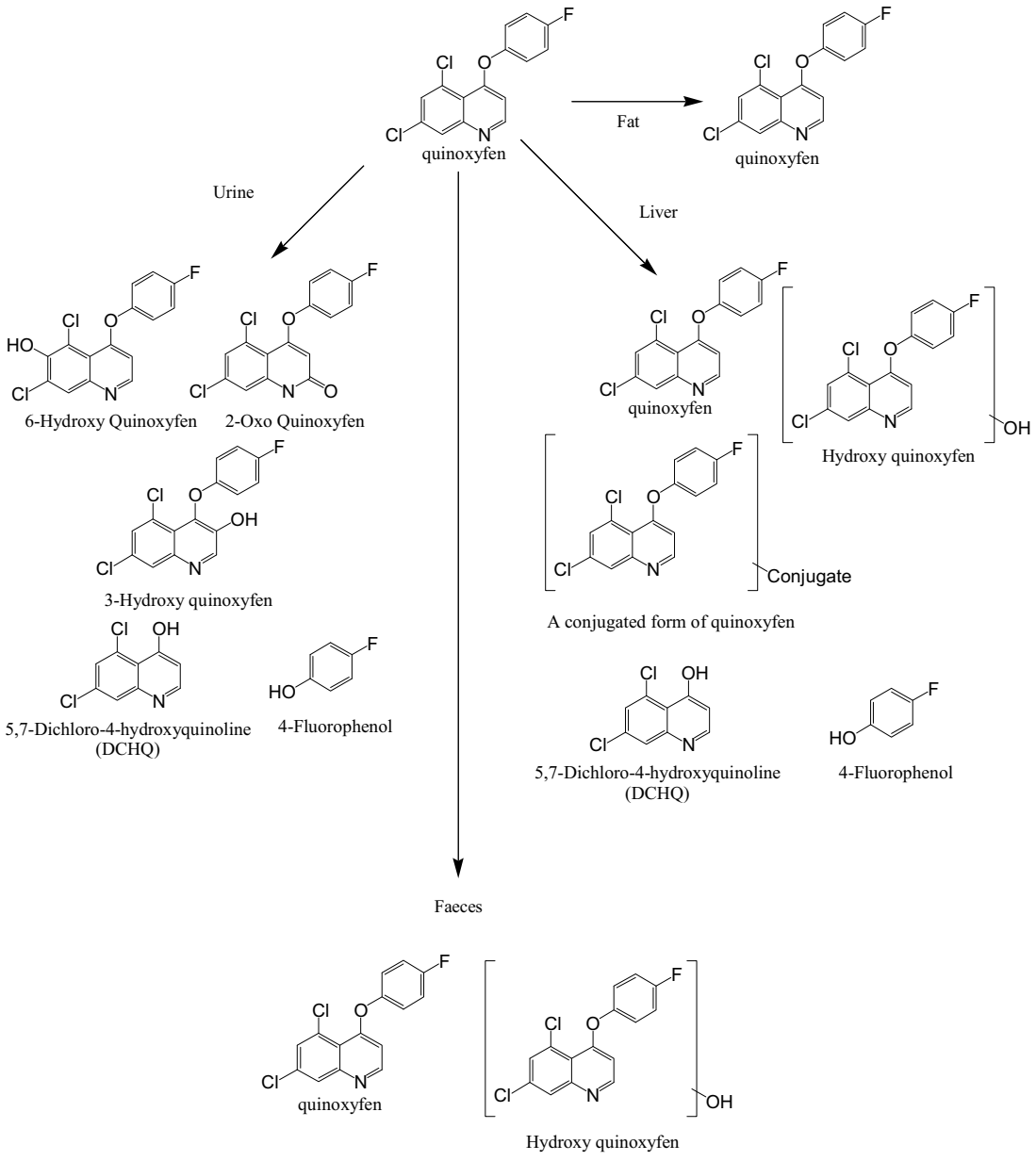


Figure 1: Proposed Metabolic Pathway for Quinoxyfen in the Lactating Goat

Metabolism in poultry

The Meeting did not receive a poultry metabolism study. Likewise, the evaluations of Australia, the EC, and the USA do not include a poultry metabolism study. However, see the poultry feedings study below, where radiolabelled quinoxyfen was utilized.

Plant metabolism

Wheat – adapted from the Evaluation of the EC (UK)

The Meeting received two study reports on the metabolism of quinoxyfen in wheat (31757, Haq and Brown, 1995a; 31659; Haq, MacDonald, and Brown, 1995). In the first study, which was a probe study, ^{14}C-quinoxyfen was applied to winter wheat plants at growth stage BBCH 32. Another application was made to previously untreated plants at growth stage BBCH 57 (70% of inflorescence emerged). Samples were collected at various time intervals and assayed for total radioactivity.

In the second study, UK trials were carried out in 1993, with [4-flurophenoxy-U-^{14}C]- or [2-quinoline-^{14}C]-quinoxyfen (radiochemical purities > 98%) formulated as emulsifiable concentrates and applied to winter wheat grown in outdoor pots in single applications of 250 g ai/ha at BBCH 32(stem elongation phase – 2nd node) or in one application of 250 g ai/ha at BBCH 49 (late booting stage – first awns visible). To aid characterisation of metabolites, applications at 1000 g ai/ha were also made at BBCH 32. Plants receiving an application at BBCH 32 were sampled at days 0, 14, 29 and 105 (harvest) after treatment. Plants receiving an application at BBCH 49 were sampled at days 0 and 78 (harvest) after treatment.

Roots were separated from aerial parts and, for harvest samples, grain separated from straw. A series of solvent washes were used to remove residues from the surface: methanol: water; dichloromethane; methanol. Remaining material was extracted with acidified acetonitrile. Samples were combusted both before and after extraction to determine total radioactivity. Radioactivity remaining in the post-extraction residual material (non-extractable) was determined by combustion/LSC. Radioactive fractions were further characterised by TLC, HPLC, and mass spectrometry. Grain samples from harvest were further separated into grain and chaff. The sequence of washes and extraction procedures employed for grain were similar to those used for straw. Only grain from plants receiving the late season application was used for studying the distribution and characterisation of radioactivity.

The distribution of radioactive residues is presented in Table 5. Total radioactivity in straw at harvest was 3–5 mg/kg for the higher application rate. Total radioactivity in grain was 0.03–0.05 mg/kg.

Table 5. The distribution of radioactivity in winter wheat straw and grain following treatment with phenoxy- and quinoline-labelled [^{14}C] quinoxyfen (% TRR and mg/kg) (31757; 31659).

Sample[1]	Aqueous wash		dichloromethane wash		methanol wash		Total wash		Acetonitrile extract		Non extractable		Total Activity
	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	mg/kg
STRAW: P-label, , BBCH 32													
Day0 1N	15.7	0.672	25.6	1.10	39.4	1.70	80.7	3.50	15.4	0.66	3.86	0.166	4.3
4N	14.9	3.03	26.8	5.40	35.8	7.30	77.5	15.7	17.0	3.40	5.50	1.12	20.4
Day 14 1N	10.4	0.212	20.8	0.42	23.4	0.48	54.6	1.11	21.3	0.43	24.1	0.49	2.04
4N	10.8	1.16	23.2	2.50	24.8	2.05	58.9	6.28	20.7	2.20	20.4	2.18	10.7
Day 29 1N	8.14	0.070	8.72	0.07	20.7	0.17	37.6	0.30	30.8	0.25	31.5	0.26	0.8
4N	7.93	0.370	11.3	0.54	22.2	1.05	41.5	1.90	25.9	1.22	32.6	1.60	4.7
Harvest1N	8.97	0.090	5.19	0.052	14.4	0.14	28.6	0.28	10.8	0.11	60.6	0.60	1.0
4N	11.7	0.6	10.8	0.52	24.4	1.16	46.9	2.24	11.4	0.55	42.0	2.00	4.8
STRAW: P-label, , BBCH 49													
Day 0 1N	5.10	0.08	23.0	0.35	38.2	0.58	66.4	1.01	30.8	0.47	2.81	0.04	1.53
Harvest1N	7.53	0.16	5.47	0.12	12.8	0.28	25.8	0.56	10.7	0.23	63.5	1.37	2.16
STRAW: Q-label, BBCH 32													
Day 0 1N	15.2	0.804	24.6	1.30	45.7	2.41	85.5	4.52	9.18	0.485	5.33	0.281	5.28
4N	14.4	5.11	34.0	12.0	42.2	15.0	90.6	32.1	6.49	2.30	2.88	1.02	35.4
Day 14 1N	6.61	0.113	16.5	0.283	22.9	0.393	46.0	0.789	17.1	0.294	36.9	0.633	1.72
4N	6.29	0.563	28.2	2.53	24.8	2.22	59.3	5.31	14.8	1.32	26.0	2.33	9.00
Day 29 1N	5.64	0.065	10.0	0.116	19.3	0.223	35.0	0.404	21.1	0.243	43.9	0.507	1.15
4N	7.16	0.323	14.3	0.643	20.4	0.922	41.9	1.89	23.8	1.07	34.3	1.55	4.51
Harvest1N	12.2	0.262	6.43	0.138	14.5	0.311	33.1	0.712	8.77	0.189	58.2	1.25	2.15
4N	10.6	0.555	8.75	0.460	16.5	0.867	35.8	1.88	9.91	0.520	54.2	2.85	5.25

Sample[1]	Aqueous wash		dichloromethane wash		methanol wash		Total wash		Acetonitrile extract		Non extractable		Total Activity
	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	mg/kg
STRAW: Q-label, , BBCH 49													
Day 0 1N	8.22	0.290	24.8	0.88	29.5	1.04	62.5	2.21	34.3	1.21	3.18	0.112	3.53
Harvest1N	9.65	0.330	4.54	0.155	10.3	0.353	24.5	0.838	12.0	0.410	63.5	2.17	3.42
GRAIN: P-label, BBCH 49													
Harvest1N	2.48	0.0009	0.92	0.0003	0.82	0.0003	4.22	0.0015	7.91	0.0028	87.9	0.0307	0.0349
GRAIN: Q-label, , BBCH 49													
Harvest1N	2.64	0.0014	0.20	0.0001	0.54	0.0003	3.38	0.0018	7.12	0.0038	89.5	0.048	0.0535

[1] 1 N = 250 g ai/ha; 4N = 1000 g ai/ha. Harvest = 105 days after treatment for early application, 78 days after treatment for late application. There were many additional whole plant sample analyses reported, but without sufficient information to calculate TRRs in mg equivalents/kg.

Table 6. Distribution and characterisation of total residues obtained from aqueous and solvent washes and acetonitrile extraction following treatment with [^{14}C] quinoxyfen. (31757; 31659).

Sample	quinoxyfen		Metabolite A[2]		Metabolite B		Metabolite C		Metabolite E		Metabolite F		Metabolite G	
	% TRR	amount mg/kg	% TRR	amount mg/kg	% TRR	amount mg/kg	% TRR	amount mg/kg	% TRR	amount mg/kg	% TRR	amount mg/kg	% TRR	amount mg/kg
STRAW: P-label, BBCH 32														
Day 0 1N[1]	69.	3.0			0.88	0.038			1.2	0.052			1.1	0.047
4N	75.	15.			0.81	0.16			1.5	0.31			0.75	0.15
Day 14 1N	34.	0.68	16.	0.33	15.	0.31	4.5	0.091	2.9	0.060	1.4	0.029	1.9	0.038
4N	39.	4.2	17.	1.8	11.	1.2	6.2	0.66	1.2	0.13	1.2	0.12	1.4	0.15
Day 29 1N	16.	0.13	23.	0.18	4.0	0.033	15.	0.12	4.7	0.039	1.7	0.014	1.7	0.014
4N	16.	0.77	18.	0.83	7.8	0.37	14.	0.67	2.6	0.12	0.91	0.043	7.3	0.35
Harvest 1N	8.3	0.083	23.	0.23			1.9	0.019	4.5	0.045			1.4	0.015
4N	27.	1.3	22.	1.0	0.70	0.034	1.8	0.087	5.5	0.26			1.4	0.067
STRAW: P-label, BBCH 49														
Harvest 1N	11.	0.23	17.	0.36	0.45	0.01	2.0	0.044	4.3	0.093			1.5	0.032
STRAW: Q-label, BBCH 32														
Day 0 1N	75.6	4.0			0.35	0.018			1.5	0.081			1.4	0.074
4N	85.	30.	0.08	0.028					0.96	0.34			0.95	0.34
Day 14 1N	30.	0.52	24.	0.40	1.5	0.026	2.2	0.038	1.7	0.029	2.1	0.037	1.3	0.023
4N	47.	4.2	20.	1.8					1.1	0.101	1.6	0.14	0.85	0.076
Day 29 1N	17.	0.20	27.	0.32	4.4	0.051			4.0	0.046	0.50	0.006	2.1	0.024
4N	33.	1.5	29.	1.3					2.5	0.11	0.57	0.026	1.1	0.051
Harvest 1N	7.9	0.17	27.	0.58	1.8	0.038	1.1	0.025	2.9	0.062			1.2	0.025
4N	20.	1.00	22.	1.2			1.1	0.057	3.3	0.17				
STRAW: Q-label, BBCH 49														
Harvest 1N	5.0	0.17	24.	0.83			1.4	0.049	3.8	0.13			1.8	0.060

Sample	quinoxyfen		Metabolite A[2]		Metabolite B		Metabolite C		Metabolite E		Metabolite F		Metabolite G	
	% TRR	amount mg/kg	% TRR	amount mg/kg	% TRR	amount mg/kg	% TRR	amount mg/kg	% TRR	amount mg/kg	% TRR	amount mg/kg	% TRR	amount mg/kg
GRAIN: P-label, BBCH 49														
Harvest 1N	0.11	.00004	8.6	0.003			2.5	.00088	0.68	.00021			0.24	.00007
GRAIN: Q-label, BBCH 49														
Harvest 1N	0.03	.00002	9.0	0.0048			1.23	.00069	0.10	.00006			0.03	.00002

[1] 1N = 0.250 g ai/ha, 4N = 100 g ai/ha

[2] A is 6 or more components, possibly organic acids.

Levels of the quinoxyfen in straw declined 4–8 fold from time 0 to harvest. At harvest, 58–63% of total radioactive residue (TRR) was non-extractable, approximately 0.1–0.2 mg/kg quinoxyfen were found in day 105 samples following application at BBCH 32 and about 0.2 mg/kg quinoxyfen were found in day 78 samples following application at BBCH 49. In grain at harvest, total radioactivity was approximately 0.04–0.05 mg/kg, of which > 90% was unextractable. Less than 0.001 mg/kg quinoxyfen was found.

No qualitative differences were found between the metabolite profiles of the phenyl- and quinoline-labelled forms.

The distribution and characterisation of metabolite residues in straw and grain are summarised in Table 7. The extractable fraction (solvent wash and acidified acetonitrile extract) was found to contain 4–6 metabolites (designated A, B, C, E, F, and G) accounting for approximately (A) 0.2–0.8 mg/kg (several components), (C) 0.02–0.1 mg/kg, (E) 0.03-0.1 mg/kg and (G) 0.02–0.06 mg/kg. It was suggested that E and G were formed from quinoxyfen by hydroxylation and loss of Cl, respectively, however no structure was given for E. Metabolite G was tentatively identified as a photodegradation product since it exhibited similar chromatographic characteristics to the product of aqueous photolysis of quinoxyfen, CFBPQ (see Figure 2). In grain, qualitatively similar metabolites were found to those found in straw, although levels were lower and were < 0.01 mg/kg.

Further experiments to increase the amount of extractable residue were carried out on samples from plants that had received a treatment at BBCH 49. Extraction of the 'non-extractable' straw fraction remaining after acetonitrile extraction involved extraction in 0.1M sodium hydroxide, followed by Soxhlet extraction in 0.2M sodium hydroxide in methanol. Grain was extracted with 0.1M sodium hydroxide. Straw was also subjected to enzyme hydrolysis using either pancreatin (a mixture that includes amylase, trypsin, lipase, ribonuclease, and protease), glucosidase, glucuronidase or cellulase.

The resulting extracts of base hydrolysis were analysed by TLC, LSC and combustion/LSC. Extracts from enzyme hydrolyses were partitioned with methyl t-butyl ether under both acidic and basic conditions. The distribution of the radioactivity between the organic and residual aqueous phases was determined by LSC. The ratio of radioactivity in the organic and aqueous partitions was compared to aliquots of material that had not been incubated in the presence of enzymes.

Alkaline hydrolysis of the straw resulted in up to ca. 82% of the previously unextracted radioactive residue being released (approximately 41% TRR in straw). TLC profiling of the hydrolysed extract indicated that the resulting radiolabelled compounds were polar in nature, as all the radioactivity remained at the origin of the chromatogram.

Alkaline hydrolysis of the grain resulted in up to 38% of the previously unextracted radioactive residue being released (approximately. 33% TRR in grain). Due to the gelatinous nature of the extracts the applicant stated that it was not possible to carry out profiling of these extracts.

Enzyme hydrolysis of straw resulted in comparable levels of radioactivity in the organic portion of the resulting extracts compared to samples following the same partitioning process that had not been incubated with enzymes. This indicated that the previously unextracted radioactivity that the

hydrolysis systems released (approximately 70% pancreatin, 94% glucosidase, 89% glucuronidase and 93% cellulase) were polar species.

Potassium permanganate was used as an oxidising agent on straw samples which had been previously extracted with acetonitrile. The agent was buffered with iron nitrate and silver nitrate. The remaining fibre was rinsed with a solution of oxalic acid in ethanol. The radioactivity in the washes and remaining fibre were determined by LSC and combustion/LSC. This system will oxidise lignin but not cellulose and therefore provided an estimate of the partition of 'unextracted' radioactivity between cellulose (direct measurement) and lignin (remainder by mass balance). Of the 64 and 73% of TRR remaining in the acetonitrile extracted tissue, approximately 37 and 40% (about 24 and 29% TRR) have been shown be associated with cellulose. The balance is considered to be associated with lignin (about 31 and 45% TRR). All values quoted are for phenyl- and quinoline labels, respectively.

Direct measurement of lignin associated radioactivity was made on straw samples which had been previously extracted with acetonitrile. The straw was soaked in dioxane: water and shaken for 4 days at room temperature, then filtered. The process was repeated for a further 2 days at 60°C, it was then centrifuged. The centrifuge pellet was extracted again as before but for 5 days at 50°C. The radioactivity of the supernatants was determined by LSC. This method of estimating lignin associated radioactivity resulted in an estimate of lignin associated radioactivity of 16 and 21% TRR for the phenyl- and quinoline-labels, respectively.

Measurement of the starch related residue of the wheat grain was carried out by finely milling the grain which was then extracted using dimethyl sulphoxide, centrifuged and the starch containing supernatant was decanted. The starch was precipitated from the supernatant with anhydrous ethanol and separated by centrifugation. The radioactivity of the different fractions was determined by combustion/LSC or LSC. These data showed that around 13 and 53% of the TRR in grain was precipitated from the system by the addition of ethanol and is therefore probably incorporated into starch. Approximately 59 and 38% of the residue remained in the supernatant (dimethyl: sulphoxide:ethanol:water) from which the starch was precipitated. All values quoted are for phenoxy and quinoline labels, respectively.

Further characterisation of the polar, origin associated 'metabolite A' in the normal phase TLC system was carried out. Hydrochloric acid was used to hydrolyse the aqueous wash samples at 85°C from the BBCH 49 application. TLC analysis demonstrated that no quinoxyfen or related material was released, by the hydrolysis. Partial hydrolysis of the quinoxyfen was observed but ca. 70% of the parent compound remained unhydrolysed. To better characterise 'metabolite A', derivatization of the aqueous washes of samples from the BBCH 49 application was carried out. Acetylation was used, dried samples were suspended in pyridine: acetic acid at 95°C, to facilitate the partitioning of the 'metabolite' into toluene. This acetylation enabled 42 and 26% of the radioactivity in the washes to be partitioned into toluene for the phenyl- and quinoline-labelled treatments, respectively. The resulting solutions were evaporated to dryness and re-suspended in acetonitrile. Reverse and normal phase TLC of the acetylated extract indicated that 'metabolite A' was (after undergoing an acetylation procedure) made up of several (at least six components).

Reverse phase HPLC analysis of washes and extracts of samples from the BBCH 49 application resulted in a broad peak within 1 minute of the solvent front. When the mobile phase was acidified (2% acetic acid) a broad peak resolved from the solvent front was observed. The applicant suggests this is evidence that 'metabolite A' and the other uncharacterised components are acidic anions. They propose that the acidification of the mobile phase results in the retention of the component on the reverse phase column, but that the multiple components of the metabolite are not resolved by this system. It was noted that in all the chromatograms the UV profile closely mirrored the radio-chromatogram, indicating that the metabolites were very similar to/may be the same as the matrix material. The use of a reverse phase ion exchange column in the chromatographic system resulted in several broad poorly resolved peaks being observed.

Tricapryl methyl ammonium chloride, (a lipophilic quaternary ammonium salt) was used as an ion pair reagent to try to extract the postulated organic acids that are proposed as constituting 'metabolite A' into dichloromethane. This system was used on the aqueous washes of the mature

samples from all the treatment regimes. At pH 2, 30% of the radioactivity was extracted into the dichloromethane. At pH 10, 82% of the radioactivity is extracted into the dichloromethane. This provides further evidence for the acidic anionic nature of the metabolites.

LC-MS studies (electrospray interface) on the ion pair extracts described above identified only one poorly resolved peak when negative ion mode was employed. The retention time of the peak matched a peak in the radioactive trace. A reasonable mass spectrum was produced with a postulated molecular ion at m/z 501, with a strong base peak at m/z305. However a corresponding peak in the UV trace at the same retention time was proposed by the applicant as indicating that 'metabolites' such as the 'metabolite A' complex and wheat natural products were in fact the same and resulted from natural incorporation.

Aqueous photolysis studies were conducted on aliquots of the parent material and resulted in the production of polar material, demonstrating that polar components were generated on photo-degradation of quinoxyfen. Chromatographic analysis of the ion paired product of this polar material showed that it was not the same as the polar material that comprised Metabolite A. This demonstrates that the metabolites produced are not simply photo-degradation products of quinoxyfen.

To support the proposal that polar metabolites represented by the 'metabolite A' complex may have arisen by the incorporation of small carbon units (derived from quinoxyfen) into natural products, an investigation into the nature of the matrix natural products extracted with surface wash solvents used was carried out. The study demonstrated that the surface washes were relatively inefficient at extracting the dislodgeable matrix material from the plant, (third or fourth extractions still removed material). 13C-NMR analysis of the aqueous and methanol wash material demonstrated that it comprised largely sugar moieties (either as mono, oligo or polysaccharides). The same analysis of the dichloromethane wash indicated that long chain hydrocarbons were present (waxes).

Metabolism in sugarbeets- adapted in part from the Evaluation of the USA

The metabolism of ^{14}C-quinoxyfen in sugarbeet roots and leaves following maximum seasonal application (for European use) of 300 g ai/ha applied in two applications, as a suspension concentrate formulation was reported to the Meeting (81925, Graper and Balcer, 2001). The last application was delivered 60 days after the first application and 26 days before the mature harvest. Immature samples were taken between the first and second applications.

Additional higher rate plots (600 g ai/ha) were included to facilitate the characterization and identification of radioactive residues. One application was made and only immature crops were harvested. Half of the plots at either rate were treated with ^{14}C-quinoxyfen labelled in the phenoxy ring and the other half labelled at the quinoline ring.

Sampling intervals for immature leaves and roots were 0, 7, 14, and 28 days after treatment, except that no root and leaf samples were taken from the 600 g ai/ha plot at 0 DAT. The radioactive residues in the samples were isolated and characterized by liquid extraction, partition, and chromatography.

Half of the immature top samples were sequentially surface washed with a dilute surfactant solution (0.1% aqueous solution of Aerosol OT) and dichloromethane. The remaining half of immature tops along with mature tops, and immature and mature root samples were not surface washed. Radioactivity was determined by liquid scintillation counting (LSC) for liquid samples (surface washings) and by combustion/LSC for solid samples (roots and tops). The limit of detection (LOD) and limit of quantitation (LOQ) were reported at 0.002 and 0.007 mg/kg, respectively. TRRs were calculated by summing the radioactivity determined for the surface washings and the respective tissue sample. The TRRs are presented in Table 7.

Table 7. TRRs in /on sugarbeet root and top samples following applications of phenyl- and quinoline-labelled [^{14}C]Quinoxyfen. (81925).

Timing and Method of application	Matrix	PHI (days)	TRR (mg/kg) [1]	
			Phenyl-label	Quinoline-label
Two foliar applications for a total application of 0.35-0.36 kg ai/ha	Whole plant	0 after 1st application	6.56 (8.60)	9.00 (10.0)
	Immature sugar beet root	7 after 1st application	0.123	0.077
		14 after 1st application	0.067	0.067
		28 after 1st application	0.014	0.025
	Mature sugar beet root	26 after 2nd application	0.078	0.049
	Mature "split" sugar beet root	26 after 2nd application	0.059	--
	Immature tops	7 after 1st application	3.28 (2.74)	3.08 (4.68)
		14 after 1st application	0.952 (1.15)	1.12 (2.44)
		28 after 1st application	0.503 (0.300)	1.08 (0.345)
	Mature tops	26 after 2nd application	1.89	2.20
Single foliar application at 0.59-0.65 kg ai/ha	Immature sugar beet root	7 after 1st application	0.287	0.124
		14 after 1st application	0.087	0.172
		28 after 1st application	0.063	0.081
	Immature tops	7 after 1st application	18.3 (19.8)	19.0 (16.0)
		14 after 1st application	9.72 (9.13)	7.30 (12.7)
		28 after 1st application	2.75 (3.47)	6.09 (2.60)

[1] TRR of surface-washed top samples are given in parentheses and are sum of the radioactivity determined in surface washings and residual tissue.

For both labels, it was found that the majority of the radioactivity was extractable in 80:20 acetonitrile/water (68–77% TRR root; 55% [quinoline label] –74% TRR leaf).

Chromatographic analyses of the acetonitrile: water extract showed the nature of radioactivity to be similar between the phenyl and quinoline labels. The parent quinoxyfen was identified as the principal residue component accounting for: 26% of TRR in phenyl-labelled roots, 25% of TRR in quinoline-labelled roots, 30% of TRR in phenyl-labelled tops, and 19% of TRR in quinoline-labelled tops. The remainder of radioactivity was characterized as polar residues consisting of multiple metabolites

The organic extracts, partitioning phases, and hydrolysates of sugar beet roots and tops were initially analyzed by HPLC conducted on a YMS ODS-AQ C18 column with a gradient mobile phase of ACN and water each with 0.1% acetic acid, using in-line radioactivity detection and UV detection (225 or 290 nm). Radioactive residues were identified by co-chromatography with the following non-labelled reference standards: quinoxyfen, 2-oxo quinoxyfen, 6-hydroxy quinoxyfen, DCHQ, and 4-fluorophenol.

Thin-layer chromatography (TLC) analysis of the initial aqueous ACN extract was performed to confirm the identity of quinoxyfen. TLC analyses were conducted using silica gel F254 plates and a toluene: acetone (75:25, v:v) solvent system. Reference standards were observed under UV light (254 nm) and radioactive residues were quantitated using phosphor imaging.

Using TLC, the minor peak which co-chromatographed with the 2-oxo metabolite by HPLC analysis, chromatographed near the 6-OH metabolite. This metabolite was not further identified, but is thought to possibly be the CFBPQ [2-chloro-10-fluoro(1)benzopyrano(2,3,4-de)quinoline] metabolite. The CFBPQ metabolite co-chromatographed with the 2-Oxo standard using HPLC analysis in a separate tomato metabolism study (see below). Because CFBPQ was the main metabolite observed in

an aqueous photolysis study (see below), this metabolite is likely formed via photolysis on the surface of the leaves and may have been absorbed and possibly further metabolized.

The aqueous phase of the quinoline-labelled extract was also analyzed by an additional HPLC system with a gradient phase over a longer period and an even more multicomponent profile was observed. The parent and DCHQ metabolite eluted near the standards, but a definitive identification could *not* be made.

Polar metabolites released by acid hydrolysis of the phenyl-labelled aqueous phase were further isolated by C18 SPE. The fraction containing the highest radioactivity was analyzed by HPLC and LC/MS. HPLC analysis was similar to the aqueous phase with 4-fluorophenol identified as the major residue, while LC/MS demonstrated that none of the radioactivity co-eluted with 4-fluorophenol, 2-Oxo, or quinoxyfen. It was proposed that the residues were metabolites which had lost the quinoline portion of the quinoxyfen molecule and which contain the chlorine atoms, such as conjugates of 4-fluorophenol. The isolated SPE fraction was also acid hydrolysed and partitioned with dichloromethane. HPLC and LC/MS analysis confirmed that the majority of radioactivity was 4-fluorophenol. Because the 4-fluorophenol metabolite was identified by co-chromatography prior to acid hydrolysis in the aqueous phase and was determined from the organic phase following acid hydrolysis, the petitioner stated that the 4-fluorophenol metabolite is present as a conjugate in the SPE fraction. The 4-fluorophenol conjugate(s) was confirmed by LC/MS.

The nonextractable residues, after initial extraction of samples with acetonitrile: water, were 23.2–32.0% of TRR for roots and 17.8–35.9% of TRR for tops. No further attempts were made to characterize bound residues in roots since the TRR was ≤ 0.02 mg/kg. To characterize bound residues in tops, sub-samples were subjected to acid detergent fibre, cellulose, and lignin isolation procedures. The results of these procedures showed that most of the radioactivity was associated with lignin.

Table 8. Summary of residue characterization/Identification in mature sugarbeet roots and tops following two foliar applications of phenyl-or quinoline-Label [^{14}C]Quinoxyfen (81925).

Metabolite/Fraction	Root		Tops	
	% TRR [a]	mg/kg[a]	% TRR	mg/kg
Phenyl-Label Quinoxyfen	TRR = 0.078 mg/kg		TRR = 1.9 mg/kg	
Quinoxyfen	26	0.020	30	0.56
4-Fluorophenol	--	--	17	0.32
CFBPQ [b]	--	--	5.0	0.094
Multiple Unknowns, Rt = 6 minutes	37	0.029	--	--
Multiple Unknowns, Rt = 25 minutes	--	--	19	0.36
Other unknowns	7.4	0.008	8.5	0.16
Total Extractable	77	0.060	74	1.4
Total Identified	26	0.020	52	0.98
Total Characterized [c]	44	0.037	28	0.52
Total Unresolved	Not reported (NR)	NR	NR	NR
Total Unextractable [d]	23	0.018	18	0.34
TOTAL	100	0.078	92	1.7
Quinoline-Label Quinoxyfen	TRR = 0.049 mg/kg		TRR = 2.2 mg/kg	
Quinoxyfen	25	0.012	19	0.43
DCHQ	--	--	6.9	0.15
CFBPQ [b]	--	--	3.0	0.065
Unknown Rt = 6 minutes	18	0.009	--	--
Other unknowns	16	0.007	25.	0.56

Metabolite/Fraction	Root		Tops	
	% TRR [a]	mg/kg[a]	% TRR	mg/kg
Total Extractable	68	0.033	55	1.2
Total Identified	25	0.012	29	0.64
Total Characterized [c]	34	0.016	25	0.56
Total Unresolved	NR	NR	NR	NR
Total Unextractable [d]	32	0.016	36	0.79
TOTAL	100	0.049	91	2.0

[a] Values were normalized in the report to compensate for non-homogeneity problems in roots.

[b] Co-eluted with 2-oxo standard using HPLC, but eluted near the 6-OH standard with TLC; this metabolite was not further identified, but is thought to possibly be the CFBPQ metabolite, (2-chloro-1)-fluoro[1]benzopyrano[2,3,4-de]quinoline.

[c] Total Characterized = sum of all unidentified/characterized metabolites.

[d] Total Unextractable = TRR - Total Extractable, as stated in report. Much of the unextractable residue in tops was characterized as lignin.

Metabolism in grapes- adapted from the Evaluation of the USA

The Meeting received a study report on the metabolism of quinoxyfen in/or grape vines (42894, Caley and Kingsley, 1995). ^{14}C-quinoxyfen, labelled in either the phenoxy ring or the quinoline ring, was formulated as a suspension concentrate and applied either at the rate of 375 mg/L or 750 mg/L active ingredient. Both radiolabelled compounds had a radiopurity > 97% (by TLC) and specific activities of 91.45 µCi/mg for the phenoxy label and 82.50 µCi/mg for the quinoline label, as determined by liquid scintillation counting. Applications were made to grape vines grown in a glasshouse, either as a direct spray to berries at approximately 18 days after the end of flowering (1st application) *or* 5 weeks later when fruits were about 70% of mature size (2nd application). In addition, at each application time point, part of a whole vine was treated with 375 mg ai/L suspension to investigate translocation into untreated parts of the vine. Fruits treated at the 'early stage' were collected at pre-harvest intervals (PHIs) of 0, 30, and 45 days; fruits treated at the 'late stage' were collected at PHIs of 0 and 10 days.

The harvested fruits were surface washed sequentially with water, dichloromethane, and methanol. Treated vines samples from the translocation studies were not surface washed. Plant tissue (vines and grapes) samples were then frozen in dry ice and homogenized using a blender. Radioactivity was determined by liquid scintillation counting (LSC) for liquid samples (surface washings) and by combustion/LSC for solid samples (vines and grapes). The TRRs were calculated by summing the radioactivity determined for the surface washings and the respective tissue sample.

Table 9. TRRs in /on the fruits and vines of grapes following a single direct spray application of phenyl- and quinoline-labelled [^{14}C]Quinoxyfen. (42894).

Timing and Method of Application	Grape Matrix	PHI (days)	TRR (mg/kg)[1]	
			Phenyl-label	Quinoline-label
Early application at 375 mg ai/L	Fruit	0	13.3	9.12
		30	2.95	2.21
		45	2.51	1.98
Late application at 375 mg ai/L	Fruit	0	4.86	4.95
		10	2.91	4.24
Early application at an exaggerated rate of 750 mg ai/L	Fruit	45	6.76	5.27
Translocation experiment: Early application to the vine at 375 mg ai/L	Treated fruit	45	1.16	1.50
	Untreated fruit	45	0.008	0.007
	Treated vines (stems/leaves)	45	15.9	23.4
	Untreated vines (stems/leaves)	45	nd [2]	nd

Timing and Method of Application	Grape Matrix	PHI (days)	TRR (mg/kg)[1]	
			Phenyl-label	Quinoline-label
Translocation experiment: Late application to the vine at 375 mg ai/L	Treated grape fruit	10	2.85	1.88
	Untreated grape fruit	10	0.006	0.006
	Treated vines (stems/leaves)	10	18.0	17.4
	Untreated vines (stems/leaves)	10	nd	nd

[1] TRR reported for grapes is the total of radioactivity determined in surface washings and tissue.

[2] Not detected.

The fruit's TRR decreased from the 0-day sampling interval to subsequent sampling intervals. The majority of radioactivity was removed from the grapes by surface washing; > 98% of the TRR was released at 0 day and > 81% TRR was released at maturity. TRR in untreated grapes were 0.002 mg/kg. The TRRs of grapes from the exaggerated rate study were basically proportional to the increase in the application rate.

In the translocation study, low levels (< 0.01 mg/kg) of radioactivity were observed in untreated grapes, and non-detectable residues were observed in the untreated vines (stems/leaves). No translocation of radioactivity from the treated vine and grapes to the untreated vines (stems/leaves) or grapes appeared to have occurred.

Surface washings, extracts, and hydrolysates (0.1 N NaOH) of grape fruit and vines were analyzed by normal-phase TLC and reversed-phase HPLC. HPLC analyses were conducted on Spherisorb ODS 2 (guard and analytical) columns with a gradient mobile phase of ACN and water, using in-flow radio-detection and UV detection (295 nm). TLC analyses were conducted on $60F_{254}$ silica plates using a mobile phase of toluene:isopropyl alcohol:acetic acid (8:2:1, v:v:v). Radioactive residues were identified by co-chromatography with the following non-labelled reference standards: quinoxyfen, quinoxyfen n-oxide, 4-fluorophenol, and DCHQ (dichloro-hydroxy quinoline). Reference standards were observed under UV light (254 nm), and radioactive residues were quantitated using phosphor imaging.

Table 10. Summary of characterization/identification of residues in mature grapes following a single direct spray application of phenoxy- and quinoline-Labelled [^{14}C]Quinoxyfen at 375 mg ai/L. (42894).

Metabolite/Fraction	Early application 45-day PHI		Late application 10-day PHI	
	% TRR	mg/kg	% TRR	mg/kg
Phenoxy-label Quinoxyfen	TRR = 2.51 mg/kg		TRR = 2.91mg/kg	
Quinoxyfen	93.	2.3	97.	2.8
Unknown Peak 1	3.3	0.083	3.0	0.088
Unknown Peak 3	0.7	0.018	--	--
NaOH hydrolysate	2.0	0.049	--	--
Total Extractable	99	2.5	99	4.2
Total Identified	93	2.3	97	2.8
Total Characterized[1]	6.0	0.15	3.0	0.088
Total Unresolved	Not reported (NR)	NR	NR	NR
Total Unextractable[2]	4.2	0.10	1.4	0.041
TOTAL	103.	2.6	101.	2.9

Metabolite/Fraction	Early application 45-day PHI		Late application 10-day PHI	
	% TRR	mg/kg	% TRR	mg/kg
Quinoline-label Quinoxyfen	TRR = 1.985 mg/kg		TRR = 4.235 mg/kg	
Quinoxyfen	94	1.8	98.	4.1
Unknown Peak 1	2.1	0.042	0.3	0.011
Aqueous	1.5	0.030	0.3	0.011
NaOH hydrolysate	1.2	0.024	--	--
Total Extractable	98	1.9	99.	4.2
Total Identified	94	1.8	98.	4.2
Total Characterized [a]	4.8	0.096	0.6	0.022
Total Unresolved	NR	NR	NR	NR
Total Unextractable [b]	4.6	0.091	1.2	0.051
TOTAL	103	2.04	100.	4.2

[1] Total Characterized = sum of all unidentified/characterized metabolites

2 Total Unextractable = TRR - Total Extractable; actual value presented in report.

Metabolism in cucumber – adapted from the Evaluation of the USA

The Meeting received a study report on the metabolism of ^{14}C-quinoxyfen in cucumbers grown in a glasshouse (45725, Chapleo and Caley, 1996). ^{14}C-quinoxyfen, labelled in either the phenoxy ring or the quinoline ring, was formulated as a suspension concentrate containing 75 mg ai/L and was applied with a compressed air sprayer to the fruit and foliage of plants as a spray at the commencement of fruit ripening, 592 mg/plant. Further applications were made to the same plants 10 days and 23 days after the initial treatment. Both radio-labelled forms were applied to separate groups of plants at each application point. Immature fruits and foliage were harvested from a single plant on the day of the first application, and prior to the second and third applications. Mature fruits and foliage were harvested from the remaining two plants 7 days following the third application. Efforts where focused on the characterization of the nature of the residue in mature samples collected from the 3 treatment regime.

In a separate experiment, ^{14}C-quinoxyfen, labelled in either the phenoxy ring or the quinoline ring and formulated as the suspension concentrate, was applied to the foliage only of two plants. Foliage and fruits were harvested 21 days after the single application.

Mature fruits were washed sequentially with water, dichloromethane and methanol and the levels of radioactivity were determined in the washes and fruit. . The TRR for each matrix was calculated by summing the radioactivity determined for the surface washings and the respective foliage or fruit tissue sample. Greater than 88% of the total radioactive residue (TRR) was removed by surface washes from samples taken immediately after the initial treatment. Much less was removed at subsequent harvests, and at the final harvest 7 days after last application, 57% TRR (phenyl label) and 36% TRR (quinoline label) were removed by surface washing. The TRR levels at various harvest intervals are summarized in Table 11.

Table11. TRRs in /on cucumber fruit and foliage following application(s) of phenoxy- and quinoline-Labelled [^{14}C]Quinoxyfen (45725).

Timing and Method of application	Cucumber Matrix	PHI	TRRs (mg/kg) [1]	
			phenoxy-label	quinoline-label
One to three spray treatments were made to the fruits and foliage of cucumbers beginning at the commencement of fruit ripening at an average rate of 75 mg ai/L per application	Fruit	Just after 1st application	0.12	0.14
		Prior to 2nd application	0.025	0.050
		Prior to 3rd application	0.017	0.017
		7 days (after final application)	0.079	0.076
	Foliage	Just after 1st application	2.7	2.0
		Prior to 2nd application	2.1	1.4
		Prior to 3rd application	3.5	2.9
		7 days (after final application)	4.2	3.4
For the translocation experiment, one spray application was made	Fruit	23 days (coincided with 3rd application above)	0.005	0.014
	Foliage	23 days (coincided with 3rd application above)	0.97	1.1

[1] TRR reported is the total of radioactivity determined in surface washings and tissue.

A sub-sample of treated fruit, that had been surface washed for TRR determination, was extracted (2×) with acetonitrile (ACN): water:1 M HCl (94:5:1, v:v:v) and then centrifuged. The extracts were combined and concentrated for thin-layer chromatography (TLC) analysis. The concentrated extract was re-dissolved in methanol and a precipitate formed. The precipitate was removed by centrifugation and re-dissolved in water.

To investigate the nature of non-extractable ('bound') residues, a sub-sample of fruit which had undergone initial extraction as described above was subjected to acid hydrolysis (refluxed with 1 M HCl for 18 hours). The hydrolysate was partitioned (3×) with hexane, and the hexane fractions were combined. Low levels of radioactivity were partitioned into the hexane phase; therefore, TLC analysis was not performed.

A sub-sample of treated foliage, that had been surface washed for TRR determination, was extracted (2×) with ACN:water:1 M HCl (94:5:1, v:v:v) and then centrifuged. The extracts were combined and concentrated for TLC analysis. Separate sub-samples of the methanol surface washings and extracts were subjected to enzyme hydrolysis to further investigate the nature of polar radioactive components. Residues were re-dissolved in 0.1 M sodium acetate buffer, pH 5. Then β-glucuronidase in sodium acetate buffer was added, and the mixture was incubated at 37°C for 18 hours.

To investigate the nature of non-extractable residues, a sub-sample of non-extractable residues following the initial extraction of residues was subjected to acid hydrolysis (refluxed with 1 M HCl for 18 hours). The hydrolysate was partitioned (3×) with hexane, and the hexane fractions were combined and concentrated for TLC analysis. The aqueous fraction was subjected to solid-phase extraction (SPE) with a phenyl Bond-Elute cartridge; residues were eluted with methanol. Radioactivity was determined in the water fraction (flow-through), methanol fraction, and solid-phase gel. The water fraction was concentrated by freeze-drying, and the methanol fraction was concentrated by rotary evaporation for TLC analysis.

Surface washings, extracts, and hydrolysates of cucumber fruit and foliage were initially analyzed by TLC conducted on $60F_{254}$ silica plates using a mobile phase of toluene:isopropyl alcohol:acetic acid (8:2:1, v:v:v). Radioactive residues were identified by co-chromatography with the following non-labelled reference standards: quinoxyfen, 2-oxo quinoxyfen, quinoxyfen n-oxide and DCHQ (dichloro-hydroxy quinoline). Reference standards were observed under UV light (254 nm) and radioactive residues were quantitated using phosphor imaging.

To confirm results, the surface washings and extracts of phenyl-and quinoline-labelled fruit and foliage samples were also analyzed by high-performance liquid chromatography (HPLC). HPLC analyses were conducted on a Spherisorb ODS 2 (guard and analytical) columns with a gradient mobile phase of ACN and water, using in-flow radio-detection and UV detection (295 nm). Identification of quinoxyfen residues was also confirmed by liquid chromatography/mass spectroscopy (LC/MS) analysis. A summary of residue characterization and identification is presented below in Table 12.

Table 12. Summary of residue characterization/identification in cucumber fruit and foliage harvested 7 days following the last of three spray treatments of phenoxy-or quinoline-labelled [^{14}C]Quinoxyfen at an average rate of 75 mg ai/L per application.(45725).

Metabolite/Fraction	Cucumber Fruit		Cucumber Foliage	
	% TRR	mg/kg	% TRR	mg/kg
Phenoxy-labelled Quinoxyfen	TRR = 0.079 mg/kg		TRR = 4.2 mg/kg	
Quinoxyfen	74	0.058	74	3.1
Quinoxyfen n-oxide	2.8	0.002	1.4	0.057
2-Oxo quinoxyfen	--	--	3.7	0.16
Unknown A	4.1	0.003	0.7	0.028
Unknown F	--	--	0.1	0.002
Origin	7.8	0.005	15[3]	0.64
Water surface wash	3.7	0.003	--	--
Precipitate	1.0	0.001	--	--
SPE gel	--	--	< 0.1	0.002
Total Extractable	93	0.074	< 95	4.0
Total Identified	77	0.060	79	3.3
Total Characterized [1]	17	0.012	< 16	0.67
Total Unresolved	Not reported (NR)	NR	NR	NR
Total Unextractable [2]	13	0.010	13	0.56
TOTAL	107	0.082	108	4.6
Quinoline-Label Quinoxyfen	TRR = 0.076 mg/kg		TRR = 3.4 mg/kg	
Quinoxyfen	64	0.049	56	1.9
Quinoxyfen n-oxide	2.5	0.001	3.3	0.11
Unknown A	1.5	0.001	0.6	0.02
Unknown B	--	--	0.3	0.011
Unknown F	2.1	0.002	0.2	0.006
Origin	10	0.008	27[3]	0.91
Water surface wash	2.6	0.002	--	--
Precipitate	3.1	0.002	--	--
Acid hydrolysate; hexane phase	0.1	< 0.001	0.1	0.002
Acid hydrolysate; aqueous phase	8.1	0.006	--	--
SPE gel	--	--	0.3	0.010
Total Extractable	95	< 0.072	88	3.0
Total Identified	67	0.050	60	2.0
Total Characterized [1]	28	< 0.022	28	0.96
Total Unresolved	NR	NR	NR	NR
Total Unextractable [2]	17	0.013	11	0.39
TOTAL	111	0.084	99	3.4

[1] Total Characterized = sum of all unidentified/characterized metabolites

[2] Total Unextractable = TRR - Total Extractable.

[3] A polar mixture associated with the baseline of TLC plates. Found in the TLC analysis of the aqueous and methanol surface washes, initial tissue extracts, and various extracts from the acid hydrolysates of the tissue remaining following solvent extractions. Shown not to be glucose conjugates.

Metabolism in tomato – adapted from the US Evaluation

The Meeting received a study report on the application of ^{14}C-quinoxyfen labelled either in the phenoxy or quinoline ring to tomatoes at the US maximum seasonal rate of 600 g ai/ha (five weekly applications of 120 g ai/ha), (78962, Byrne *et al.,* 2000). Tomato plants were grown to maturity outdoors in separate plots treated either with ^{14}C-phenoxy and ^{14}C-quinoline quinoxyfen. Immature fruit was collected at 0, 7, 14, and 28 days after the first application. Mature fruit was collected 14 days after the 5th and final application (42 days after the first application). Vines were collected and analyzed 0, 7, and 42 days after the first application.

Treated tomato samples from all sampling intervals were surface washed sequentially with a dilute soap (0.01%) solution and methylene chloride. Treated vines samples, collected prior to the third application and were not analyzed. Surface-washed plant tissue (fruit and foliage) samples were then frozen in dry ice and homogenized using a mill. Radioactivity was determined by liquid scintillation counting (LSC) for liquid samples (surface washings) and by combustion/LSC for solid samples (homogenized fruit and foliage tissue). The limit of detection (LOD) and limit of quantitation (LOQ) were reported as 0.0029 and 0.011 mg/kg, respectively. The TRRs presented in Table 13 below were calculated by summing the radioactivity determined for the surface washings and the respective tissue sample.

Table 13. TRRs in /on tomato fruits and foliage following application(s) of phenoxy- and quinoline-labelled [^{14}C] Quinoxyfen (78962).

Timing and Method of application	Matrix	PHI (days)	TRR [1]	
			Phenoxy-label	Quinoline-label
One to five post-emergence foliar applications were made at a nominal rate of 0.12 kg ai/ha per application.	Immature fruit	0 after 1st application	0.057	0.092
		Prior to 2nd application	0.042	0.063
	Immature fruit	Prior to 3rd application	0.083	0.093
		Prior to 5th application	0.13	0.19
	Mature fruit	14 after 5th application	0.19	0.243
	Immature foliage	0 after 1st application	5.4	6.6
		Prior to 2nd application	4.2	3.6
	Mature foliage	14 after 5th application	11	14

The soap (aqueous) and DCM surface-washings of mature fruits contained sufficient radioactivity for chromatographic analysis (HPLC and thin-layer chromatography (TLC)); the DCM surface-washings were concentrated, and residues were re-dissolved in ACN and water prior to analysis.

Sub-samples of post-rinsed samples were extracted (3x) with ACN: water (80:20, v:v) and then centrifuged. The extracts were combined, and an aliquot was concentrated in the aqueous phase and partitioned with DCM: ACN (80:20, v:v). The organic phase was concentrated and diluted with ACN and water for HPLC analysis; the aqueous phase was directly analyzed by HPLC.

To investigate the nature of non-extractable ('bound') residues, a sub-sample of non-extractable residues following the initial extraction was subjected to acid hydrolysis (1 N HCl refluxed for 4 hours) and then vacuum filtered. The acidified pellet was rinsed with water, centrifuged, and filtered. The rinsate was combined with the acid hydrolysate. The nonextractable residues remaining following acid hydrolysis were then extracted with ACN to remove acid-labile, non-water soluble residues.

A separate but larger sub-sample of milled tomato fruit was subjected to a series of fractionation procedures in order to elucidate the nature of bound residues. These sub-samples were extracted and acid hydrolysed as described above, and the nonextractable residues were subjected to ADF isolation, or lignin or cellulose determinations. Briefly, nonextractable residues were refluxed for 1 hour in acid detergent solution (hexadecyltrimethylammonium bromide in 2 N sulfuric acid).

The solids were collected by vacuum filtration and dried in an oven (80° C) overnight. The dried solid acid detergent fibre (ADF) fraction and liquid ADF rinsate were analyzed by LSC or combustion/lSC. To isolate lignin, chilled sulphuric acid was added to nonextractable residues, and the sample was refrigerated overnight. The sample was diluted with water, refluxed for 2 hours, cooled, and vacuum filtered. The solids were dried in an oven (60° C) overnight, and the solid (lignin) fraction was determined by LSC; the liquid filtrate is *assumed* to contain primarily dissolved cellulose. To isolate cellulose, buffered saturated potassium permanganate was added to nonextractable residues, and the sample was filtered. Additional potassium permanganate was added and filtered to ensure complete oxidation/solubilisation of lignin. Excess potassium permanganate was removed from the oxidized solid by the addition of oxalic and hydrochloric acids in ethanol, after which the solids were sequentially washed with ethanol and acetone. The solids were dried in an oven (60°C) overnight, and the solid (cellulose) fraction was determined by LSC; the liquid filtrate is *assumed* to contain primarily oxidized lignin.

Surface washings of phenyl- and quinoline-labelled mature tomatoes contained 57% and 62% TRR, respectively, with the largest amount of radioactivity recovered in the organic surface-washing. The remainder of radioactivity (20–27% TRR) was largely extracted using ACN:water (80:20, v:v). Approximately half of the extract was partitioned into organic solvent. Phenyl- and quinoline-labelled bound residues of mature tomatoes were subjected to sequential acid hydrolysis (1 N HCl at reflux) and ACN extraction, which released an additional 2.4–4% TRR. Bound residues remaining following simple extraction and hydrolysis accounted for 4.0% and 3.9% of TRRs in phenyl- and quinoline-labelled tomatoes, respectively. The levels of radioactivity in the surface washings, extracts, and bound fractions were similar for phenyl- and quinoline-labelled tomato fruit.

Individual surface washings, and the organic and aqueous extracts were subjected to HPLC analysis for characterization/identification of residues. Most of the radioactivity in all of the surface washings was identified as quinoxyfen (51–54% TRR). Quinoxyfen was also detected as the major residue present at approximately 12% TRR in the organic and aqueous extracts. An unknown peak eluting at approximately 42 minutes and present at < 2% TRR was observed in the organic extract (both labels). This peak had a similar retention time as the 2-oxo metabolite. The remainder of the peaks detected in the aqueous and organic extracts were minor polar unknowns, each present at < 5% TRR. The residue profiles for the phenyl- and quinoline-labelled fruit were similar. An additional 2.4–4% TRR was released from non-extractable residues with acid hydrolysis and subsequent ACN extraction.

A larger sample of surface-washed fruit tissue was extracted and subjected to acid hydrolysis and ACN extraction for further characterization of non-extractable residues. Bound residues were characterized to be associated with ADF (10–12% TRR, 0.020-0.028 mg/kg) containing lignin, cellulose, and hemicellulose.

Tomato foliage samples harvested 14 days following five applications were subjected to extensive investigation. The soap (aqueous) and methylene chloride (DCM) surface-washings contained sufficient radioactivity for chromatographic analysis (HPLC and TLC); the DCM surface-washings were concentrated, and residues were re-dissolved in ACN and water prior to analysis.

A sub-sample of post-rinsed foliage was extracted (3x) with ACN: water (80:20, v:v) and then centrifuged. The extracts were combined, and an aliquot was concentrated in the aqueous phase and partitioned with DCM:ACN (80:20, v:v). The organic phase was concentrated and diluted with ACN and water for HPLC analysis; the aqueous phase was directly analyzed by HPLC.

To investigate the nature of nonextractable residues, a sub-sample of nonextractable residues from the initial extraction was subjected to acid hydrolysis (1 N HCL refluxed for 4 hours) and vacuum filtered. The acidified pellet was rinsed with water, centrifuged, and filtered. The rinsate was combined with the acid hydrolysate. The nonextractable residues remaining following acid hydrolysis were then extracted with ACN to remove acid-labile, non-water soluble residues. The acid hydrolysate was partitioned with DCM: ACN (80:20, v:v). The DCM phase was concentrated, and the organic and aqueous phases were analyzed by HPLC.

A separate but larger sub-sample of milled tomato foliage was subjected to extraction, acid hydrolysis, and ACN extraction. The nonextractable residues following acid hydrolysis and ACN extraction were subjected to ADF isolation, or lignin or cellulose determinations. The similar procedures described for fruits were employed for this purpose.

Non-extractable residues were subjected to sequential acid hydrolysis (1 N HCl at reflux) and ACN extraction, which released an additional 8.4–9.7% TRR. Bound residues remaining following simple extraction and hydrolysis accounted for 5.9% and 6.7% TRR in phenyl- and quinoline-labelled foliage, respectively. Levels of radioactivity in the surface washings, extracts, and bound fractions were similar for phenyl- and quinoline-labelled tomato foliage.

Individual surface washings and the organic and aqueous extracts were subjected to HPLC analysis for characterization/identification of residues. Most of the radioactivity in all of the surface washings was identified as quinoxyfen (31.6–34.5% TRR). Quinoxyfen was also detected as the major residue present at ≤ 9–12% TRR in the organic extract. 4-fluorophenol was identified as a minor residue (0.9% TRR) in the phenyl-labelled organic extract only. An unknown peak eluting at approximately 42 minutes and present at ≤ 3.2% TRR was observed in the organic extract (both labels). This peak had a similar retention time as the 2-oxo metabolite standard. An additional unknown peak (Rt = 35 minutes) was detected at ≤ 7% TRR, and the remainder of the peaks detected in the organic extracts were minor polar unknowns each present at < 1% TRR. The aqueous extracts of both phenyl- and quinoline-labelled foliage were comprised of more polar unknown peaks each present at ≤ 3.3% TRR. The residue profiles for the phenyl- and quinoline-labelled foliage were similar.

A larger sample of surface-washed foliage tissue was extracted and subjected to acid hydrolysis and ACN extraction. The majority of the bound residues were associated with ADF (3.7–4.6% TRR) containing lignin, cellulose, and hemicellulose.

The surface washings, extracts, and hydrolysates of tomato fruits and foliage were analyzed by HPLC. HPLC analyses were conducted on a YMC ODS-AQ column with a gradient mobile phase of ACN and water each containing 0.1% acetic acid, using in-flow radiodetector and UV detector (295 nm). Radioactive residues were identified by co-chromatography with the following non-labelled reference standards: quinoxyfen, 4-fluorophenol, 2-Oxo (2-oxo quinoxyfen), 6-OH (6-hydroxy quinoxyfen), and DCHQ (dichloro-hydroxy quinoline).

Residues of quinoxyfen were confirmed in the surface-washings and organic phase of the initial extraction by TLC analysis. TLC analyses were conducted on $60F_{254}$ silica plates using a mobile phase of toluene: acetone (75:25, v:v). Reference standards were observed under UV light (254 nm) and radioactive residues were quantitated using a linear detector.

Minor peaks observed by HPLC analysis of the DCM surface-wash of foliage samples eluted with potential photolysis products in the fruit, such as 2-oxo metabolite. However, these peaks did not co-chromatograph with the 2-oxo standard using TLC. These degradates were, therefore, further isolated using an open column system. The phenyl-labelled DCM surface-wash was applied to a medium bore silica column and one-minute fractions were collected for HPLC and LC/MS analysis. In all but the first fraction collected, some radioactivity eluted similar to the 2-oxo metabolite using gradient HPLC; however, multiple components were observed using isocratic HPLC (water: ACN each with 0.1% acetic acid; 4:6, v:v). It is reasonable to conclude that the residue consists of a range of components instead of a single metabolite accounting for > 10% of the radioactivity present.

The major residue identified by HPLC in fractions 1 and 2 was quinoxyfen, and was confirmed by LC/MS. In fraction 3, the major component was analyzed by LC/MS which did not demonstrate fragmentation across the ether linkage; the degradate was proposed to be CFBPQ (2-chloro-10-fluoro[1]benzopyrano[2,3,4-de]quinoline) based on the accurate mass. The spectrum of the degradate, by ^{1}H NMR analysis, was consistent with CFBPQ. The CFBPQ degradate was the main degradate identified in an aqueous photolysis study; therefore, CFBPQ may be formed via photolysis on the surface of the tomatoes or leaves.

In fraction 4, residues similar to the parent, but more polar and with different fragmentation patterns by LC/MS analysis, were thought to be rearrangement isomers of quinoxyfen. The chemical structures could not be conclusively determined with NMR. Analysis of fraction 5 by LC/MS indicated possible structures of a *p*-hydroxyphenoxy degradate and 2-oxo metabolite.

Table 14 lists the summary of residues identified and characterized from this study.

Table 14. Summary of characterization/identification of ^{14}C-residues in mature tomato fruits and foliage harvested 14 days following the last of five foliar applications of phenyl- or quinoline-Label [^{14}C]Quinoxyfen for a total rate of 600 g ai/A. (78962).

Metabolite/Fraction	Tomato Fruit 14-day PHI		Tomato Foliage 14-day PHI	
	% TRR	mg/kg	% TRR	mg/kg
Phenoxy-labelled Quinoxyfen	TRR = 0.191 mg/kg		TRR = 10.716 mg/kg	
Quinoxyfen	63	0.12	43	4.6
4-Fluorophenol	--	--	0.9	0.096
Unknown Rt = 35 minutes	--	--	7.0	0.75
Unknown Rt = 42 minutes	1.9	0.003	3.2	0.34
"Other" unknowns	each ≤ 5.0	each ≤ 0.008	each ≤ 3.3	each ≤ 0.36
Acid hydrolysate	3.6	0.007	7.6	0.81
ACN extract	0.4	0.001	2.1	0.22
Total Extractable	88	0.17	87	9.3
Total Identified	63.	0.12	44	4.7
Total Characterized [1]	> 11 [2]	> 0.019 [2]	> 23.	> 2.5
Total Unresolved	Not reported (NR)	NR	NR	NR
Total Unextractable [3]	4.0	0.008	5.9	0.64
TOTAL [4]	92	0.18	93	10
Quinoline-label Quinoxyfen	TRR = 0.24 mg/kg		TRR = 14. mg/kg	
Quinoxyfen	65	0.16	43	6.1
Unknown Rt = 35 minutes	--	--	6.5	0.92
Unknown Rt = 42 minutes	1.8	0.004	2.2	0.304
"Other" unknowns	each ≤ 1.6	each ≤ 0.002	each ≤ 2.1	≤ 0.30
Acid hydrolysate	2.1	0.005	6.6	0.93
ACN extract	0.3	0.001	1.8	0.26
Total	84	0.20	85	12
Total Identified	65	0.16	43	6.1
Total Characterized [1]	> 5.8 [2]	> 0.012 [2]	> 19. [2]	> 2.7
Total Unresolved	NR	NR	NR	NR
Total Unextractable [3]	3.9	0.009	6.7	0.9 5
TOTAL [4]	88	0.21	92	13

1 Total Characterized = sum of all unidentified/characterized metabolites

2 Because only the maximum single unknown level was reported; the total identified does not include unknowns below the maximum value.

3 Total Unextractable = TRR - Total Extractable; actual value presented in the report.

4 Total as presented in the report.

In summary, quinoxyfen was metabolized in plants with portions of the molecule becoming associated with natural plant constituents. The main residue identified in the roots, leaves, and fruits at harvest, was the parent compound, quinoxyfen. Hydroxylation of the quinoline or phenoxy rings was

observed. Cleavage of the ether bond was a minor pathway (sugar beet). CFBPQ was formed, perhaps via surface photolysis. There was no evidence of significant translocation from treated foliage to other parts of the plant. The metabolic pathways are indicated in Figure 2.

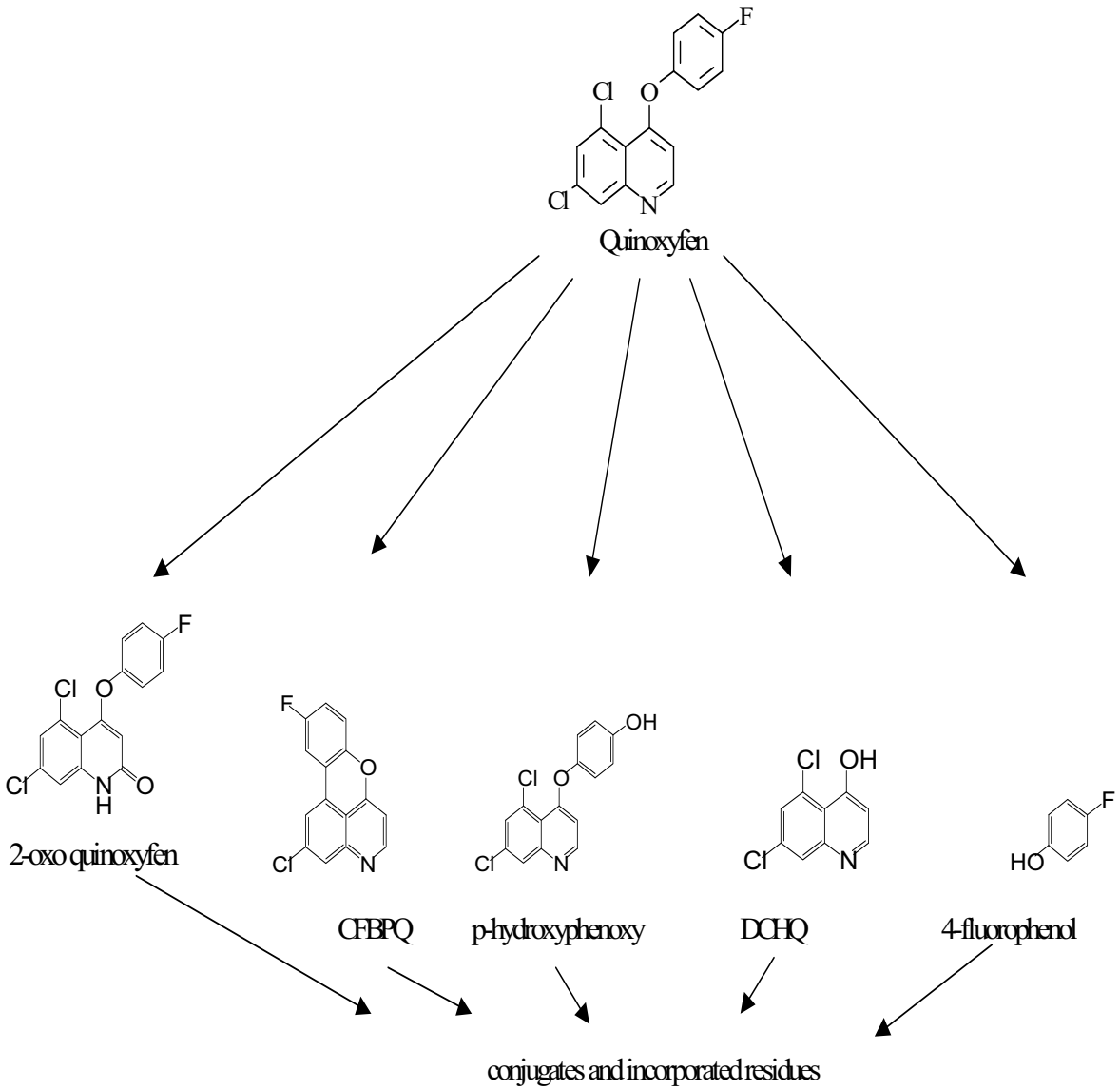


Figure 2. Proposed quinoxyfen metabolic pathway in plants.

Environmental fate

Aerobic soil metabolism

The Meeting received a study report on the aerobic soil metabolism of quinoxyfen (6357, Reeves, 1993; 8411, Ghosh and Portwood, 1994; and 29386, Cracknell *et al.,* 1995). In a laboratory study, one standard (Speyer 2.2) and three UK agricultural soils (Castle Rising, Marcham, and Wereham) were treated with quinoline-labelled [^{14}C]-quinoxyfen at a rate equivalent to 250 g ai/ha and incubated at 40% moisture content and 20ºC in the dark for up to 200 days. Under these conditions parent compound slowly degraded to give 5,7-dichloro-4-(4-fluorophenoxy)-2-oxo-quinoline 2-oxo-DE-795). In the three agricultural soils this did not exceed *ca* 8% of applied radioactivity (AR) at any time, although it did reach 27% AR at 200 days in non-agricultural Speyer 2.2 soil. The pattern of 2-oxo-DE-795 formation was such that no significant plateau and decline could be detected in any soil during the test period. A second metabolite identified as 5,7-dichloro-4-hydroxyquinoline (DCHQ) was also formed as a minor component in Speyer 2.2 soil, and as the only degradation product in acidic Wereham soil (pH4.2), where it reached 6% AR at 100 days. Non-extractable residue (NER; up to 25% AR) and small amounts of CO_2 (< 2% AR) were also seen. The distribution of radioactivity in the four soils is summarized in Table 15.

Table 15. Summary of aerobic soil degradation in various soil types (6357; 8411; 29386).

	Days After Treatment (% Applied Radioactivity)								
Matrix	0	4	8	16	36	64	100	150	200
DE-795	96 - 101	94 - 100	90 - 99	88 - 97	86 - 96	80 - 95	74 - 85	58 - 81	53 - 81
2-OH-quinoxyfen[1]	0	0	0	0 - 3	0 - 6	0 - 11	0 - 15	0 - 22	0 - 27
DCHQ	0	0	0	0	0	0	0 - 6	0 - 6	0 - 5
CO_2	0	trace	trace	trace	< 1	< 1	< 2	< 2	< 2
NER	1 - 5	2 - 6	3 - 10	3 - 11	5 - 14	7 - 16	10 - 18	14 - 25	15 - 25
Total	101 - 103	98 - 103	100 - 102	99 - 101	99 - 102	99 - 102	99 - 102	100 - 102	100 - 102

[1] Misidentified as 3-OH-quinoxyfen (18219, N. R. Pearson and G. L. Reeves, 2005)

At 150 days, the NER was further extracted by sonication with methanol at 55°C, and the extracts shown to contain between 34–46% of the NER as DE-795, 2-Oxo-DE-795 and DCHQ (in proportions similar to their content in the original soil extracts) that had become bound to the soil with time.

The proposed route of aerobic soil degradation is shown in Figure 3.

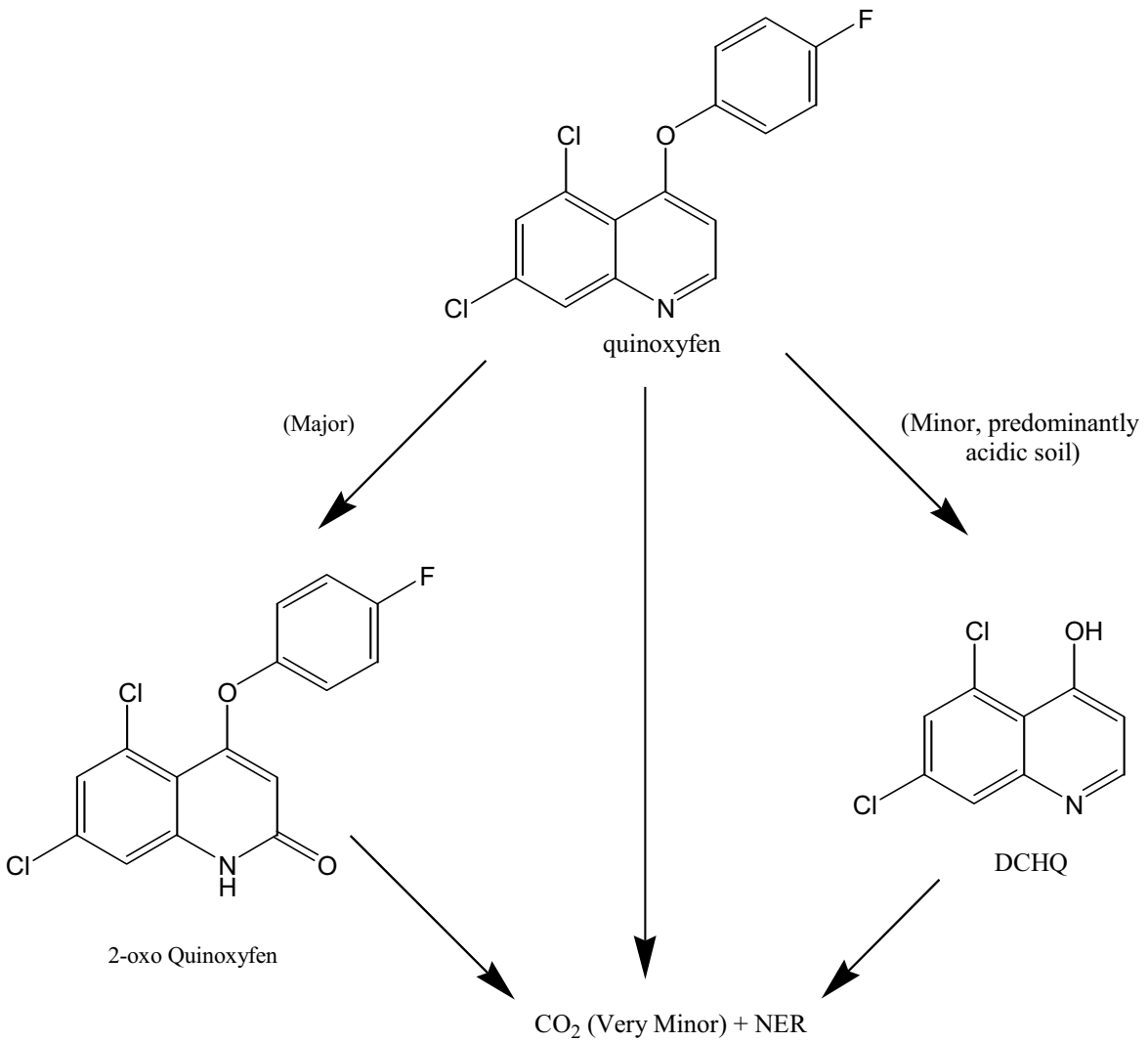


Figure 3. Pathway for the aerobic soil degradation of quinoxyfen.

DT_{50} and DT_{90} values were calculated for the soils from the above study as well as in a second standard soil (Speyer 2.1) as part of the aged residue column leaching study (24033, Reeves, 1994). The application rate was equivalent to 250 g ai/ha (1× rate) throughout. Comparative work was also done to investigate the effect of soil moisture content (Castle Rising at 60%moisture content), temperature (Speyer 2.2 at 10ºC and 30ºC) and application rate (Speyer 2.2 at 4× and ¼× rate) upon the degradation kinetics (K01). Table 16 shows the kinetic data (calculated assuming first-order kinetics) obtained under the various test regimes.

Table 16. Quinoxyfen laboratory aerobic soil degradation half-lives (1× = 250 g ai/ha) (24033).

Soil	Moisture content	Temp.	Application Rate	DT_{50} (days)	DT_{90} (days)
Speyer 2.2	40%	20°C	1×	220	ca 730
Castle Rising	40%	20°C	1×	510	ca 1700
Marcham	40%	20°C	1×	300	ca 1000
Wereham	40%	20°C	1×	470	ca 1500
Speyer 2.2	40%	20°C	¼×, 4×	220	ca 730-760

Soil	Moisture content	Temp.	Application Rate	DT_{50} (days)	DT_{90} (days)
Speyer 2.2	40%	10°C	1×	870	-
Speyer 2.2	40%	30°C	1×	87	ca 270
Castle Rising	60%	20°C	1×	450	ca 1500
Speyer 2.1	40%	20°C	1×	300	-

The results showed that quinoxyfen is *slowly* degraded in soil under dark aerobic conditions. There was an apparent decline in the degradation rate in all soils after 100 days, particularly in Marcham soil, suggesting biphasic or non-linear degradation. Therefore, the DT_{50} and DT_{90} values presented were calculated using only data to 100 days. The rate decline could not be attributed to any significant decrease in microbial biomass.

Hydrolytic degradation

The hydrolysis of Quinoxyfen was investigated in sterile buffer solutions (pH 4, 7 and 9) at a nominal concentration of 0.5 µg ai/mL (8557, Reeves, and Ghosh, 1994). Acetone was used as a 1% co-solvent to facilitate solubility. The degradation was studied at 50°C and all pH values, and at pH4 at 25°C and 40°C using [^{14}C]-quinoline labelled quinoxyfen. The rates of hydrolytic degradation ($t_{½}$) under the various test regimes are summarized in Table 17.

Table 17. Hydrolytic degradation of quinoxyfen (8557).

Temperature	Half-life ($t_{½}$)		
	pH4	pH7	pH9
50°C	7 days	Stable	Stable
40°C	16 days	ND	ND
25°C	75 days	ND	ND

Stable = no degradation after 5 days and <1% degradation after 21 days

ND = not determined due to stability seen at 50°C

At a temperature closer to environmental conditions (25°C), quinoxyfen was stable at pH 7 and pH 9 but degraded at pH 4 with a half-life of 75 days. At 25°C, DCHQ reached approximately 41% AR at 46 days (*ca* 33% AR at 30 days). The extent of hydrolysis confirmed that seen in early-stage environmental fate probe studies. The single hydrolysis product (which was seen at all temperatures) was identified as DCHQ by chromatographic analysis and mass spectrometry.

The effect of buffer (phthalate and citrate), quinoxyfen concentration (0.5 and 0.06 µg/mL) and co-solvent (acetone and THF) were further investigated in a hydrolysis study at pH 4 and 50ºC (6328, Baloch, R, *et al.,* 1997). The results confirmed the findings of the previous study by Reeves and Ghosh in showing that quinoxyfen was degraded at pH 4 (half-life of approximately 5 days; compared to 7 days from the Reeves study to give DCHQ). This was irrespective of buffer type, quinoxyfen concentration, or co-solvent.

Photochemical degradation

The Meeting received several study reports on the photochemical degradation of quinoxyfen. The photolysis quantum yield of quinoxyfen was determined in water/acetonitrile (8:2 v/v) at concentrations of between 4.6–17.9 mg ai/L using artificial light at the absorption maximum (298 nm) at 20°C (4429, Rüdel, 1995). The acetonitrile was shown to have no effect upon light absorption. Quinoxyfen degraded rapidly under these conditions with a quantum yield of 1.2 (± 0.2) × 10^{-2}. This value was then used to calculate photolysis half-lives in dilute aqueous solution at latitude 52°N using the ABIWAS program which is based on the model of Frank and Klopffer (1988). This gave half-lives of 1.7 hours in June and 22.8 hours in December, assuming average light intensities and weather

conditions. The maximal or expected 'worst case' half-lives for March (earliest application) and June (latest application) were 16.4 hours and 6.8 hours, respectively.

Quinoxyfen degraded to give two main photoproducts. The main degradate (up to 30% at 8 hours), with a chromatographic polarity between quinoxyfen and 5,7-dichloro-4-hydroxy quinoline (DCHQ), was identified by mass spectrometry as 2-chloro-10-fluoro[1]benzopyrano[2,3,4-de]quinoline (CFBPQ) (Portwood, D., 1996; Report GHE-P 4721). A second degradation product (up to 11%) matched DCHQ by HPLC. At 6 hours, a quantitative mass balance was seen in the irradiated solution compared to a dark control, indicating no volatile loss of radioactivity.

In order to investigate the photo degradation of quinoxyfen in aqueous solution under sunlight conditions, and to determine the rate and extent of formation of CFBPQ and its subsequent degradation, a non-guideline outdoor photolysis study was carried out (48846, MacDonald., 1997) using quinoxyfen radiolabelled [^{14}C] on the C-2 of the quinoline ring. This was performed at Letcombe Regis, UK (latitude around 50°N) using natural lake water, pH 7 buffer, and natural water/sediment, with exposure during August-September, 1996. Favourable conditions existed for photolysis with non-turbid, shallow water being used. The application rate was 0.5 µg/mL. Further samples without sunlight exposure were used as dark controls.

After 1 day in the light exposed samples, no significant levels of quinoxyfen were detected in either the natural water system or the water/sediment system. The water contained only CFBPQ and a very polar, multi-component material. Subsequent analysis of water samples to 28 days after treatment showed that CFBPQ rapidly degraded with a half-life of about 1–2 days in the natural water/buffer test systems, and approximately 4 days in the natural water with sediment present. The degradation of CFBPQ was followed by increasing amounts of very polar, multi-component material (7–27 components). On day 1, the water test system contained 54% of the applied radioactivity as CFBPQ, 27% as polar metabolite(s), and 0% as quinoxyfen. On day 14, 0.47% of applied radioactivity was CFBPQ, 73% polar material. The buffered water test system yielded similar results. The dark natural water controls consisted largely of quinoxyfen (108% applied radioactivity day 1, 74% day 14, 51% on day 28, with no degradates identified). The dark buffered water controls showed no degradation of quinoxyfen over 14 days and about 20% degradation from day 14 to day 28.

The photochemical degradation of quinoxyfen on soil was also studied (31076, Reeves, 1995). Speyer 2.2 soil (loamy sand) was surfaced treated with [^{14}C]-quinoxyfen labelled at the 2 position of the quinoline ring at a rate of 250 g ai/ha. The soil was maintained in a stainless steel incubation chamber covered with an airtight quartz glass plate. Irradiation was performed in 12 hour cycles of light and dark, using sun simulation lamps. Temperatures during the light and dark cycles were about 25°C and 18°C, respectively. Control samples were maintained under the same conditions, but with no light. At 1, 3, 7, 14, 21, and 30 days after treatment the headspace was purged and analyzed for carbon dioxide and volatiles. Soil samples were taken at the same intervals and immediately after application.

Quinoxyfen degraded under the artificial light conditions with a half-life of about 200 days, estimated to be equivalent to > 1 year in natural sunlight in the spring in England. The main degradation product, up to 6.5% of applied radioactivity, remained unidentified. DCHQ was found (2.5% maximum of applied radioactivity). Very little organic volatiles or carbon dioxide was found (< 1% applied radioactivity). Total recovery of radioactivity ranged from 96% to 103%. Greater than 70% of the applied radioactivity was identified as quinoxyfen after 30 days. The non-extractable residue in soil increased to a maximum of 11% of applied radioactivity on day 30. No degradation occurred with the dark controls, the non-extractable residue did not exceed 4% of applied radioactivity, and about 94% of applied radioactivity was identified as quinoxyfen.

Residues in succeeding crops (Confined rotational crop)

Uptake of quinoxyfen from soil into three succeeding crops was investigated (75502, Haq and Brown, 1995). Two radiolabelled forms of quinoxyfen (phenoxy-labelled and quinoline-labelled) were prepared as an emulsifiable concentrate spray solution and applied separately to Mendip loam soil to give an even distribution on the soil surface using a DeVilbiss spray gun. The emulsifiable

concentrate was diluted with water to give a final concentration of 400 g ai/hectare. To assess if the treated tubs received the required amount of quinoxyfen, the soil was sampled within 24 hours after application.

The three succeeding crops studied were, turnips (root crop), sunflowers (oilseed crop) and cabbage (leafy crop). The crops were sown into the soil, at the appropriate depth and density for the crop, 30 days after the application. The crops were sampled at harvest (118 days after treatment for cabbage and turnip, 150 days after treatment for sunflower) and separated into above-ground and below-ground portions. The latter were water washed to remove adhering soil. Crop matrix samples were homogenized and the total radioactivity determined by combustion.

Soil samples were extracted sequentially with acetonitrile and acetone. Extraction released about 88% of the radioactivity from day zero soils and about 80% of the radioactivity from harvest day soils. Extracts were analyzed by normal phase TLC and reverse phase HPLC (UV detection 295 nm and radioactivity monitor). Analysis of the soil extracts of Day 0 samples showed only quinoxyfen to be present. Analysis of the soil extracts of harvest samples indicated only quinoxyfen (> 95% of soil radioactivity) and very low levels of metabolites (< 5%), more polar than the parent. Thus, metabolism of quinoxyfen does not occur extensively over the time course of the study (150 days). It is possible that the more polar metabolites were available for uptake into the plants.

The raw agricultural commodity (RAC, i.e. turnip root, cabbage leaves and sunflower head) were homogenized and the total radioactive content of the homogenized fractions determined by combustion. Results are given in Table 18.

Table 18. Total radioactive residue (TRR) content of rotational crops (75502).

Commodity	[^{14}C] Label	µg/kg
Cabbages (leaves)	Phenoxy	0.43
Cabbages (leaves)	Quinoline	0.49
Cabbages (leaves)	Control	-
Sunflowers (Head)	Phenoxy	1.2
Sunflowers (Head)	Quinoline	0.28
Sunflowers (Head)	Control	-
Turnips (Root)	Phenoxy	3.5
Turnips (Root)	Quinoline	3.4
Turnips (Root)	Control	-

The levels of radioactivity taken up from soil treated with ^{14}C-quinoxyfen into the RAC of the three succeeding crops studied (turnip, cabbage and sunflower) were below 4 µg/kg quinoxyfen equivalents. The residue levels observed were too low to allow for chromatographic analysis.

RESIDUE ANALYSIS

Analytical Methods

Analytical methods for determination of residues of quinoxyfen have been developed for a wide range of substrates. The methods have been extensively validated with numerous recoveries on a wide range of substrates. The analytical methods for determination of residues of quinoxyfen in plant and animal matrices follow similar partitioning, clean-up and quantification procedures. Generally, quinoxyfen residues are extracted from plants and animal tissues samples with acidic acetonitrile. After addition of sodium bicarbonate solution to an aliquot of the extract, quinoxyfen is partitioned into hexane, which is then evaporated to dryness. The residue is reconstituted in hexane prior to an aminopropyl solid phase extraction using 1% acetone in hexane to elute quinoxyfen residues. The eluate is evaporated to dryness and reconstituted in 0.1% corn oil in tri-methyl pentane (TMP). Quinoxyfen is

quantified either by gas chromatography with mass selective detection (GC-MSD) or by HPLC with UV absorbance. This basic method has been modified and validated for use in various matrices. Validation study results were corrected for control values which were typically < 1% of the lowest fortification level. Matrix-specific methods are summarized below.

The Meeting received several methods for the determinate of quinoxyfen in grapes and processed grape commodities Method ERC 94.29 – Determination of grapes by reverse phase HPLC using UV absorbance detection at 235 nm (105043, Khoshab, 1995); Independent laboratory validation (41039, Khoshab, 1995) was used in all supervised trials on grapes conducted in various European countries and was used for some supervised trials on grapes conducted in Australia. The method involves extraction of quinoxyfen from the crop by macerating with acidic acetone. After addition of sodium bicarbonate solution to an aliquot of the extract, the residue was partitioned into hexane which is then evaporated to dryness. The residue is reconstituted in hexane prior to an aminopropyl solid phase extraction using 1% acetone in hexane to elute the quinoxyfen. The eluate is evaporated to dryness and reconstituted in acetonitrile/water. Quinoxyfen is quantified by reverse phase HPLC using UV absorbance detection at 235 nm. Confirmation is by GC/MSD (237 and 272 amu ions).

The method was validated by analysis of fortified samples for residues of quinoxyfen in grapes over the range of 0.01 – 1.0 mg/kg. The method was independently validated by an external laboratory, where samples were fortified with quinoxyfen over the range of 0.01–10 mg/kg. Table 19 summarizes the recovery data.

Table 19. Recovery of quinoxyfen from fortified grapes (Method ERC94.29) (105043; 41039).

Fortification levels	% Recovery[1]	
	Original method	Independent validation
Mean control values	0.0000 (n=6)	0.0002 (n=6)
0.01	94, 100[2]	82, 83[2]
0.01	98, 86	84, 95
0.01	95, 100	89, 88
0.20	99, 100	100, 93
1.0	98, 99	110, 103
10.0	Not fortified at this level	101, 100
Mean (overall)	97 ± 4.4	94 ± 9
% RSD over validated range (0.01-10.0 mg/kg)	4.5 (n=10)	9.5 (n=12)
% RSD at lowest validated level (0.01 mg/kg)	5.5 (n=6)	5.6 (n=6)
Mean at lowest validated level (0.01 mg/kg)	96 ± 5.3	87 ± 4.9

[1] Corrected for control value and moisture content

[2] Duplicate samples.

Method ERC 95.26 – Determination of quinoxyfen in grapes, must, wine, and pomace by gas chromatography with mass selective detection (GC-MSD) (105334, Khoshab and Roberts, 1996; 83731, Thompson, 2002) was used in processing studies for grapes carried out in European countries and in field trials and processing studies conducted in the US. In this method, quinoxyfen residues were extracted from grapes, pomace, and raisins and analyzed as in ERD 94.29. The final residue was reconstituted in 0.1% corn oil in tri-methyl pentane (TMP).

Quinoxyfen residues in grape must, grape juice, and wine were extracted by addition of sodium bicarbonate solution followed by partitioning into hexane which was then evaporated to dryness and reconstituted in 0.1% corn oil in TMP.

Quinoxyfen was quantified by gas chromatography with mass selective detection (GC-MSD). The m/z 237.0 was used for confirmation, and the m/z 306.9 ion for quantification. The method was validated by fortifying control samples with quinoxyfen at levels of 0.01 mg/kg to 2.0 mg/kg. Recovery experiments were performed in 4 batches by two analysts. Table 20 summarizes the recovery data, including the relative standard deviation at the lowest validated level as well as over the validated range.

Table 20. Recovery of quinoxyfen from fortified grape wine, must, pomace (Method ERC95.26) (105334).

Matrix	Fortification level (mg/L)	% Recovery[1]	Average % recovery	% RSD
Wine	0.01	88, 90[2]	87 ± 4	4.6 (n=8)
	0.01	85, 85		
	0.01	93, 88		
	0.01	84, 80		
	0.05	102, 99		
	0.05	107, 111		
	0.05	111, 117		
	0.05	105, 103		
	0.20	108, 107		
	0.20	110, 104		
	0.01 – 0.20	80 - 117	99 ± 11.2	11.3 (n=20)
Must	0.01	87, 76	88 ± 12.5	14.2 (n=6)
	0.01	102, 101		
	0.01	92, 72		
	0.05	99, 100		
	0.05	118, 115		
	0.05	105, 101		
	0.2	99, 112		
	0.5	100, 99		
	0.01 – 0.5	72 - 118	99 ± 12.4	12.5 (n=16)
Pomace	0.05	77, 80	78 ± 2.7	3.4 (n=6)
	0.05	80, 73		
	0.05	79, 79		
	0.20	69, 72		
	0.50	85, 98		
	1.0	75, 75		
	2.0	76, 77		
	0.05 – 2.0	69 - 98	78 ± 6.9	8.9 (n=14)

[1] Corrected for control Mean control values: wine = 0.0000 (n=8); must= 0.0000 (n=6); pomace= 0.0000 (n=6)

[2] Duplicate samples.

Table 21. Recovery of quinoxyfen from grapes, juice, and raisins (Method ERC95.26) (83731).

Matrix	Fortification level (mg/L)	% Recovery	Average % recovery	% RSD
Grapes	0.01	100		
	0.01	97	99 ± 1	1.5 (n=3)
	0.01	99		
	0.10	89		
	0.10	98	92 ± 5	5.6 (n=3)
	0.10	89		
	1.0	78		
	1.0	95	89 ± 10	11 (n=3)
	1.0	95		
	0.01 – 1.0	78 – 100	93 ± 7	7.5 (n=9)
Juice	0.01	89		
	0.01	87	88 ± 2	1.6 (n=3)
	0.01	86		
	0.10	76		
	0.10	76	76 ± 0	0 (n=3)
	0.10	76		
	1.0	73		
	1.0	70	71 ± 2	3 (n=3)
	1.0	69		
	0.01 – 1.0	69 - 89	78 ± 7.5	9.6 (=9)
Raisins	0.01	78		
	0.01	77	78 ± 1	1(n=3)
	0.01	78		
	0.10	96		
	0.10	96	96 ± 1	0 (n=3)
	0.10	97		
	1.0	89		
	1.0	90	88 ± 3	0.8 (n=3)
	1.0	85		
	0.01 – 1.0	77 - 97	87 ± 8	9.4 (n=9)

Method GRM 99.02 – Determination of quinoxyfen in grapes and dried grapes by gas chromatography with mass selective detection (GC-MSD) (74780, Teasdale, 2000) Independent laboratory validation (102718, Dobbs, 2001) was used for determination of quinoxyfen in supervised trials on grapes in Australia. Sample preparation is similar to that of ERC95.26. Quinoxyfen was quantified by gas chromatography with mass selective detection (GC-MSD). The quantitation ion is 237 amu, and the confirmation ion is 272 amu.

At least two samples (in duplicate) were fortified at quinoxyfen concentration levels of 0.01 to 0.5 mg/kg. A summary of the recovery data are summarized in Table 22.

The independent laboratory validation (Dobbs, 2001) conducted for the determination of residues of quinoxyfen on raisins using Method GRM 99.02 showed average recoveries of 94 ± 8% over the concentration range of 0.01 mg/kg to 0.1 mg/kg. Individual recovery values were within the acceptable range of 70–120%. The results are included in Table 22.

Table 22. Recovery of quinoxyfen from fortified grapes and dried grapes (Method GRM99.02) (74780, 102718).

Fortification level (mg/kg)	n	Recovery range (%)	Mean (%)	Standard deviation	% RSD
GRAPES					
0.01	8	98 - 105	101	2.6	2.6
0.05	3	78 - 104	102	20.5	20.1
0.2	4	91- 115	103	11.1	10.8
0.5	4	94 - 98	96	1.7	1.8
0.01 - 0.5	19	78 - 115	100	8.8	8.8
DRIED GRAPES					
0.01	4	68 - 95	82	11.9	14.5
0.05	4	84 - 99	92	7.4	8.0
0.2	4	77 - 115	93	15.8	17.0
0.5	4	70 - 95	79	10.8	13.7
0.01 - 0.5	20	68 -115	86	12.4	14.4
DRIED GRAPES (ILV)					
0.01	5	84 - 86	85	0.84	9.8
0.02	3	98 - 103	101	2.6	2.6
0.10	5	95 - 106	100	4.3	4.3
0.01 – 0.1	13	84 - 106	94	8	8.5

Mean control values: Grapes = 0.0002 mg/kg (n=8); Dried grapes = 0.003 mg/kg (n=8).

Method ERC 95.26 – Validation of method for determination of quinoxyfen in cherries by GC-MSD (102722, Chen, 2002) was validated for use in the supervised trials on cherries conducted in the US. Untreated samples of cherries were fortified at concentration levels of 0.01mg/kg to 1.0 mg/kg. The recovery results are summarized in Table 23.

Table 23. Recovery of quinoxyfen from fortified cherries (102722).

Fortification level (mg/L)	% Recovery[1]	Average % recovery	% RSD
0.01	92		
0.01	96	94 ± 2	2.13%
0.01	94		
0.1	94		
0.1	97	96 ± 2	2.16%
0.1	98		
1.0	79		
1.0	98	92 ± 11	11.9%
1.0	98		
0.01 – 1.0	79 - 98	94 ± 6	6..4% (n=9)

Method ERC 95.26 – Validation of method for the determination of quinoxyfen residues in lettuce by GC-MSD (208698, Barney, 2005)- was validated for use on lettuce samples collected from trials in the US after minor modifications of ERC95.26 such as changes in the filtration procedure or volume of solvent used, which did not affect the performance of the method. Method validation was carried out at fortification levels of 0.01, 0.1, and 1.0 mg/kg of quinoxyfen. The results are summarized in Table 24.

Table 24: Recovery of quinoxyfen from fortified leaf lettuce samples (Method ERC95.26) (208698).

Fortification level (mg/L)	% Recovery[1]	Average % recovery	% RSD
0.01	87	88 ± 2.2	2.5
0.01	87		
0.01	89		
0.01	91		
0.01	90		
0.01	85		
0.10	86	88 ±2.1	2.4
0.10	87		
0.10	90		
1.0	84	89 ± 4.5	5.0
1.0	89		
1.0	93		
0.01 – 1.0	84 - 93	88 ± 2.6	2.9 (n=12)

Method ERC 96.16 – Independent laboratory validation for the determination of quinoxyfen residues in melon peel and pulp by GC-MSD (45724, Khoshab, and Rawle, 1996) was used in the supervised trials on melons conducted in European countries and involved extraction of quinoxyfen with acidic acetone and partitioning into hexane after addition of sodium bicarbonate solution. The extract was evaporated to dryness and the residue was reconstituted in hexane prior to an aminopropyl solid phase extraction using 1% acetone in hexane to elute the quinoxyfen. The eluate was then evaporated to dryness and reconstituted in tri-methyl pentane containing 1% corn oil and 1,4-dibromonaphthalene as internal standard. Quinoxyfen was measured by gas chromatography with mass selective detection using m/z 237 ion. The m/z 286 ion was used for quantification of the internal standard.

Method 96.16 was independently validated by an external laboratory and the results were consistent with recovery data and chromatography generated on the original method (Table 25).

Table 25. Recovery of quinoxyfen from fortified melon peel and pulp (Method ERC 96.16) (45724).

Fortification level (mg/kg)	% Recovery	
	Original method	Independent Validation
MELON PEEL		
0.01	82, 82	103, 87
0.01	88, 89	87, 86
0.01	101, 100	92, 95
0.01	93, 92	77, 86
0.05	92, 92	96, 94
0.20	94, 104	84, 93
0.50	100, 97	97, 105
1.0	101, 98	101, 100
Mean Overall	94 ± 6.6	92 ± 7.7
% RSD (0.01-1.0 mg/kg)	7.0 (n=16)	8.4 (n=16)
Mean (0.01 mg/kg)	91 ± 7.2	89 ± 7.7
% RSD (0.01 mg/kg)	7.9 (n=6)	8.6 (n=6)
MELON PULP		
0.01	91, 94	102, 83
0.01	92, 95	89, 89
0.01	92, 100	98, 84
0.01	91, 96	86, 98

Fortification level (mg/kg)	% Recovery	
	Original method	Independent Validation
0.05 0.20 0.50 1.0	92, 98 103, 105 102, 101 99, 99	89, 98 93, 89 102, 101 100, 104
Mean Overall	97 ± 4.6	94 ± 7.0
Mean (0.01 mg/kg)	94 ± 3.1	91 ± 7.2
% RSD (0.01-1.0 mg/kg)	4.7 (n=16)	7.5 (n=16)
% RSD (0.01 mg/kg)	3.3 (n=6)	7.9 (n=6)

Method ERC 96.08 – Validation of method for the determination of quinoxyfen residues in peppers (76968, Khoshab and MacMillan, 1996; 08006, Chen, 2003) uses an extraction and clean-up scheme and analysis method similar to ERC 96.16, for use in trials on peppers in the US.

Table 26. Recovery of quinoxyfen from fortified samples of peppers (Method ERC 96.08) (76968; 08006).

Fortification level (mg/L)	Range% Recovery	Average % recovery	% RSD
0.01	78 - 120	87.7 ± 10	11.3 (n=18)
0.05	92 - 102	99.1 ± 4.1	4.2 (n=14)
2.0	101 - 107	103.5 ± 2.6	2.6 (n=4)
0.01 – 2.0	78 - 120	93.9 ± 9.8	10.4 (n=36)

Method GRM 99.04 – Determination of residues of quinoxyfen in sugar beet roots and tops by GC-MSD (74779, Teasdale, Lyons, Rhodes, and Patel, 2000) was used in trials in European countries. The method uses the extraction, clean-up, and analysis schemes of ERC 96.16. The validation was performed in 4 batches by 2 analysts. The results are summarized in Table 27.

Table 27. Recovery of quinoxyfen from fortified sugar beet roots and tops (74779).

Fortification level (mg/kg)	% Recovery[1]	Average % recovery	% RSD
SUGAR BEET ROOTS			
0.01 0.01 0.01 0.01	103, 100 92, 98 97, 87 103, 118	100 ± 9.2	9.2 (n=8)
0.05 0.05	98, 96 100, 102	99 ± 2.6	2.6 (n=4)
0.2 0.2	90, 103 101, 102	99 ± 61	6.2 (n=4)
0.5 0.5	89, 91 89, 86	89 ± 2.1	2.4 (n=4)
0.01 – 0.5	86 - 118	97 ± 7.6	7.8 (n=20)
SUGAR BEET TOPS			
0.1 0.1 0.1 0.1	104, 104 101, 105 110, 87 109, 105	103 ± 7.1	6.9
0.5 0.5	99, 100 100, 97	99 ± 1.4	1.4
2.0 2.0	97, 96 92, 101	96 ± 3.7	3.8

Fortification level (mg/kg)	% Recovery[1]	Average % recovery	% RSD
5.0 5.0	97, 96 98, 96	97 ± 0.6	0.6
0.1 – 5.0	87 - 110	100 ± 5.5	5.5

[1] Corrected for control value and moisture content.

Mean control values: Sugar beet roots = 0.0001 mg/kg (n=8); tops = 0.0002 mg/kg (n=8)

Method ERC 94.5 – Determination of quinoxyfen residues in wheat and barley straw and grain by GC-MSD (63697, Gambie and Nicholson, 1994); Independent laboratory validation (31621, Gambie, Rawle, and Shaw, 1995) was used in supervised trials conducted on wheat and barley in various European countries. After extraction of the residues in acidic acetone, sodium bicarbonate was added and the extract was partitioned into hexane which was evaporated to dryness. The residue was next reconstituted in hexane and the resulting solution cleaned up in an aminopropyl solid phase extractor using 1% acetone in hexane. The eluate was evaporated to dryness and reconstituted in 0.1% corn oil in tri-methyl pentane containing 1,4-dibromonaphthalene as internal standard. Quinoxyfen was quantified by gas chromatography with mass selective detection. Ions monitored were 237 and 272 m/z for quinoxyfen and 286 m/z for the internal standard. This method is very similar to method ERC 96.16.

The method was validated by fortification of samples of wheat and barley grain at quinoxyfen concentration levels of 0.01–1.0 mg/kg and samples of straw at levels of 0.05–10 mg/kg. Two independent external laboratories also performed recovery experiments at the same levels of fortification. The results are summarized in Table 28.

Table 28. Summary of recoveries of quinoxyfen from fortified wheat and barley grain and straw samples (63697; 31621).

Fortification level (mg/kg)	% Recovery[1]		
	Original method, ERC 94.5	Independent validation lab 1	Independent validation lab 2
WHEAT AND BARLEY GRAIN			
0.01	80, 91[2]	92, 81[2]	100, 93
0.01	95, 86	107, 107	98, 93
0.01	92, 86	86, 80	88, 107
0.05	93, 96	119, 119	-
0.10	84, 94	73, 80	97, 110
0.5	-	-	104, 105
1.0	103, 144	-	
Mean	92 ± 7.2	94 ± 17.2	99.5 ± 7
Mean control values	0.0003 mg/kg (n=6)	0.0002 (n=6)	0.000 (n=3)
% RSD over the validated range	7.9 (n=12)	18.2 (n=10)	7 (n=10)
% RSD at the lowest validated value (0.01 mg/kg)	6.1 (n=6)	13.3 (n=6)	6.9 (n=6)
WHEAT AND BARLEY STRAW			
0.05	87, 88	89, 92	87, 91
0.05	76, 80, 76, 78	88, 83	89, 85
0.05	90, 92, 93	79, 71	94, 87
0.05	82, 86	-	-
0.20	82, 88	-	97, 94
0.20	80, 88	-	-

Fortification level (mg/kg)	% Recovery[1]		
	Original method, ERC 94.5	Independent validation lab 1	Independent validation lab 2
0.50	95, 89	77, 85	103, 95
1.0	90, 96,98	85, 89	-
5.0	76, 83	-	-
10.0	76, 82	72, 85	88, 86
Mean Overall	85 ± 6.8	82.9 ± 6.8	91.3 ± 5.4
Mean (0.05 mg/kg)			
Mean control values	0.0029 (n=12)	0.0003 (n=6)	0.0096 (n=3)
% RSD over the validated range	7.9 (n=24)	8.2 (n=12)	5.9 (n=12)
% RSDat the lowest validated value (0.05 mg/kg)	7.4 (n=11)	9,2 9 (n=6)	3.7 (n=6)

1 Corrected for control values.

2 Replicate samples.

Method ERC 95.16 – Determination of quinoxyfen residues in flour, bran and bread by GC-MSD (31600, Gambie and Press, 1995) was used in the processing study on wheat and involved extraction and clean-up of quinoxyfen residues by the procedure of Method ERC 94.5. Quinoxyfen was quantified by gas chromatography with mass selective detection. Ions monitored are 286 amu for the internal standard and 237 amu (quantitation ion) and 272 amu (confirmation) for quinoxyfen. Ion 307 is also considered if the ratio of 237/272 is not within ± 10% of the value of the bracketing standard.

The method was validated for each analyte (flour, bran and bread) by fortification with quinoxyfen at concentration levels of 0.01 mg/kg to 0.20 mg/kg. The results are summarized in Table 29.

Table 29: Recovery of quinoxyfen from fortified wheat flour, bran, and bread (31600).

Fortification level (mg/kg)	% Recovery[1]		
	FLOUR	BRAN	BREAD
0.01	77, 88	75, 73	86, 96
0.01	107, 107	101, 101	98, 98
0.01	90, 87	80, 86	91.90
0.05	86, 77	73, 77	84, 90
0.20	87, 94	86, 89	104, 98
Mean Overall	90 ± 10.4	84 ± 10.4	94 ± 6.3
Mean control values	0.0002 mg/kg (n=6)	0.0004 mg/kg (n=6)	0.000 (n=6)
% RSD over the validated range (0.01-0.20 mg/kg)	11.6 (n=10)	12.4 (n=10)	6.7 (n=10)
% RSD at the lowest validated value (0.01 mg/kg)	12.9 (n=6)	14.3 (n=6)	5.3 (n=6)

[1]Corrected for appropriate control value

Method ERC 95.10 – Determination of quinoxyfen residues in beer by GC-MSD (63705, Teasdale and Press, 1995)-was used in the study involving processing of barley into beer. Quinoxyfen residues were extracted from beer samples with methyl-tertiary-butyl ether after addition of sodium bicarbonate solution and acetone. The extract was evaporated to dryness and the residue was reconstituted in 0.1% corn oil in tri-methyl pentane. Quinoxyfen was quantified by gas chromatography with mass selective detection. The ions monitored are 237 amu and 272 amu for quinoxyfen.

The method was validated by recovery experiments carried out in 2 batches by 2 analysts. Beer samples were fortified with quinoxyfen at concentration levels of 0.01 mg/kg to 0.5 mg/kg.

Table 30. Recovery of quinoxyfen from fortified beer samples (63705).

Fortification level (mg/L)	% Recovery[1]	Average % recovery	% RSD
0.01 0.01 0.01	75, 72 80, 72 92, 92	80.5 ± 9.4	11.6 (n=6)
0.05 0.05	86, 84 68, 81	79.8 ± 8.1	10.2 (n=4)
0.10 0.10	90, 92 72, 78	83 ± 9.6	11.6 (n=4)
0.50 0.50	94, 94 75, 86	87.3 ± 9	10.3 (n=4)
0.01 – 0.50	68 - 94	82.4 ± 8.8	10.6 (n=18)

1 Corrected for control value.

Mean control value = 0.000 mg/kg (n=6)

Method ERC 95.26.S1 – Determination of residues of quinoxyfen in hops by gas chromatography with mass selective detection (73994, Oberwalder, 1998; 81400, West, S., 2001); Independent laboratory validation (80416, Eckert, J. and West, S., 2000) was used in trials on hops conducted in the US. It is a modification of Method 95.26 which was originally developed and validated for grapes. The procedure for hops was the same as for grapes, with minor modifications such as increase in volume of extracting solvent and proportionate increase in the volumes of sodium bicarbonate and hexane. Recovery data were generated for hops by an independent laboratory by fortifying untreated control samples with quinoxyfen at concentration levels of 0.05 mg/kg and 0.1 mg/kg.

Table 31. Recovery of quinoxyfen from fortified samples of hops (73994; 80416; 81400).

Fortification level (mg/L)	% Recovery	Average % recovery	% RSD
0.05 0.05 0.05	113 117 87	106 ± 16	15.4 (n=3)
0.10 0.10 0.10	100 102 104	102 ± 2	2 (n=3)
0.05 –0.10	87 - 117	104 ± 10.6	10.6 (n=6)

Method ERC 94.7 – Determination of residues of quinoxyfen in skimmed milk, whole milk, and cream by GC-MSD (135047, Class, 2003) was developed for quantitative determination of quinoxyfen residues in skimmed milk, whole milk and cream. This method was used in the cattle feeding study. Quinoxyfen was extracted from the milk fraction by shaking with methanol. After addition of sodium bicarbonate solution, quinoxyfen was partitioned into hexane which was then evaporated to dryness. The residue was reconstituted in 1% acetone in hexane and applied to an aminopropyl SPE cartridge using a further volume of 1% acetone in hexane to elute the residue.

For cream samples, the eluate was evaporated to dryness and reconstituted in 5% methanol in dichloromethane prior to clean-up using gel permeation chromatography. For skimmed milk and whole milk, the residue was reconstituted in hexane and further purified using a silica SPE cartridge using 10% methyl-tertiary-butyl ether in hexane to elute quinoxyfen. The eluates were evaporated to dryness and the residue reconstituted in 0.1% corn oil in tri-methyl pentane. Quinoxyfen was quantified by gas chromatography with mass selective detection. The 237 m/z fragment ion was used for quantification, and the 272 m/z and 274 m/z fragment ions were used for confirmation.

The method was validated at fortification levels of 0.001 to 0.1 mg/kg quinoxyfen.

Table 32. Recovery of quinoxyfen from fortified samples of whole milk, skimmed milk, and cream (Method ERC 94.7) (135047).

Fortification level (mg/kg)	% Recovery[1]		
	SKIMMED MILK	WHOLE MILK	CREAM
0.001	87, 93	88, 79	64, 100
0.001	90, 96	83, 78	97, 110
0.001	99, 86	77, 85	90, 83
0.01	86, 85	80, 81	99, 99
0.1	79, 80	78, 84	87, 86
Mean	88 ± 6.5	81 ± 3.6	92 ± 12.6
Mean control values	0.000 mg/kg (n=6)	0.000 mg/kg (n=6)	0.000 (n=6)
% RSD over the validated range (0.001 – 0.1 mg/kg)	7.3 (n=10)	4.4 (n=10)	13.8 (n=10)
% RSD over the lowest validated range	5.6 (n=6)	5.3 (n=6)	17.6 (n=6)

1Corrected for appropriate control value

The independent laboratory validation consisted of fortification of skimmed milk and cream samples with quinoxyfen at concentration levels of 0.001 mg/kg and 0.1 mg/kg.

Table 33: Summary of ILV results for recovery of quinoxyfen from fortified skimmed milk and cream (Method ERC 94.7) (130457)

Matrix	Fortification level (mg/kg)	n	% Recovery (range)	Average % recovery	% RSD
Skimmed milk (1.5% fat)	0.001	5	76 - 113	101	15
	0.1	5	85 - 98	91	5
	Overall fortification	10	76 - 113	96 ± 11.5	12
Cream (30% fat)	0.001	4	69 - 81	75[1]	9
	0.1	4	70 - 78	74[2]	4
	Overall fortification	8	69 - 81	74 ± 4.4	6

[1] One result, 39% was identified by Dixon-Test as an outlier and was not included in the calculations.

[2] One result, 55%, was identified by Dixon-Test as an outlier and was not included in the calculations.

Method ERC 94.20 – Determination of residues of quinoxyfen in bovine muscle, kidney, and fat by GC-MSD (63682, Hastings, M. and Gambie, A., 1995); Independent laboratory validation (135044, Class, 2003) is applicable to the quantitative determination of quinoxyfen residues in bovine muscle, kidney, and fat down to the lowest validated level of 0.01 mg/kg. Quinoxyfen was extracted from the tissue by shaking with methanol. After addition of water, quinoxyfen was partitioned into hexane which was then evaporated to dryness. The residue was reconstituted in dichloromethane prior to clean-up using gel permeation chromatography. The eluate was evaporated to dryness and reconstituted in 0.1% corn oil in tri-methyl pentane. Quantification was by gas chromatography with mass selective detection. The 237 m/z fragment ion was used for quantification, and the 272 m/z and 274 m/z fragment ions were used for confirmation.

The method has been validated by the analysis of untreated and fortified samples for residues of quinoxyfen in muscle, kidney, and fat over the range of 0.01-1.0 mg/kg.

Table 34. Recovery of quinoxyfen from fortified samples of bovine muscle, kidney, and fat (Method ERC 94.20) (63682).

Fortification level (mg/kg)	% Recovery[1]		
	MUSCLE	KIDNEY	FAT
0.01	92, 100	91, 91	104, 116
0.01	93, 113	97, 98	108, 100
0.01	102, 112	94, 98	92, 95
0.10	110, 107	78, 86	109, 109
1.0	99, 96	95, 94	80, 93
Overall Mean	102 ± 7.7	92 ± 6.2	101 ± 10.7
Mean at lowest validated value (0.01 mg/kg)	102 ± 9.0	95 ± 3.3	102 ± 8.8
Mean control values	0.0007 mg/kg (n=6)	0.000 mg/kg (n=6)	0.000 (n=6)
% RSD over the validated range (0.01- 1.0 mg/kg)	7.6 (n=10)	6.7 (n=10)	10.6 (n=10)
% RSD over the lowest validated range (0.01 mg/kg)	8.8 (n= 6)	3.5 (n=6)	8.6 (n=6)

[1] Corrected for appropriate control value

For the independent laboratory validation, bovine meat and fat samples were fortified with quinoxyfen at levels of 0.01 mg/kg and 1.0 mg/kg.

Table 35. Summary of ILV recovery results for quinoxyfen residues in fortified bovine meat and fat (Method ERC 94.20) (135044).

Matrix	Fortification level (mg/kg)	n	% Recovery (range)	Average % recovery	% RSD
Bovine meat	0.01	4[1]	68 - 93	84	14
	1.0	5	86 - 105	92	9
	0.01- 1.0	8	80 - 105	88 ± 10.6	12
Bovine fat	0.01	5	70 - 96	80	13
	1.0	5	80 - 96	84	8
	0.01 -1.0	10	70 - 96	82 ± 8.2	10

[1] One result, 60% was caused by a partial loss of extract during partition and was not included in the calculation.

Method ERC 94.30 – Determination of residues of quinoxyfen in bovine liver by GC-MSD (63683, Hastings and Gambie, 1995); Independent laboratory validation (135046, Class, 2003) was used in the cattle feeding study. The liver sample was first digested with aqueous hydrochloric acid/pepsin in order to extract quinoxyfen residue. After centrifuging, the remaining solid tissue was extracted with methanol and again centrifuged. The methanol was decanted and removed by evaporation. The residue was reconstituted in the original acid/pepsin extract which was then treated with buffer and β-glucuronidase to breakdown any conjugated residues. After addition of water, quinoxyfen was partitioned into hexane which was then evaporated to dryness. The residue was reconstituted in dichloromethane prior to clean-up using gel permeation chromatography. The eluate was evaporated to dryness and reconstituted in 0.1% corn oil in tri-methyl pentane, and quinoxyfen was quantified by gas chromatography with mass selective detection. Ion 237 m/z is used for quantification, and ions 272 m/z and 274 m/z are used for confirmation.

The method has been validated by analysis of untreated and fortified samples for residues of quinoxyfen in liver over the range of 0.01 – 1.0 mg/kg. An independent laboratory validation resulted in unacceptable results, while in the repeated tests the recoveries remained poor (Table 37).

Table 36. Recovery of quinoxyfen from fortified samples of bovine liver (Method ERC 94.30) (63683).

Fortification level (mg/kg)	% Recovery	Average % Recovery	% RSD
0.01	67, 73	67 ± 3.8	5.6 (n=6)
0.01	61, 67		
0.01	68, 68		
0.10	62, 63		
1.0	62, 64		
0.01 - 1.0	61 - 73	66 ± 3.7	5.7 (n=10)

Mean control value = 0.000 (n=6)

Table 37. Summary of ILV recovery results for quinoxyfen residues in fortified bovine Liver, Second Attempt (Method ERC 94.30) (135046).

Fortification level (mg/kg)	% Recovery	Average % Recovery	% RSD
0.01	49	44 ± 4.9	11 (n=5)
0.01	46		
0.01	46		
0.01	36		
0.01	44		
1.0	53	54 ± 2.2	4.1 (n=4)
	56		
	55		
	51		
0.01 - 1.0			

Control values < 0.0005 and 0.0009 mg/kg. One sample at the 1.0 mg/kg fortification level (recovery 41% was excluded because of partial loss of hexane extract on evaporation.

Method ERC 98.05 – Determination of quinoxyfen residues in eggs by GC-MSD (64399, Khoshab et.al, 1998); Independent laboratory validation (135045, Class, 2003)-was used in the poultry feeding study. Quinoxyfen was extracted from whole egg and egg yolk by macerating and shaking with an acidic acetone solution. After addition of sodium bicarbonate solution, quinoxyfen was partitioned into hexane which was then evaporated to dryness. The residue was reconstituted in 2% acetone in hexane and applied to an aminopropyl SPE cartridge using a further volume of 2% acetone in hexane to elute quinoxyfen. The eluate was evaporated to dryness and reconstituted in dichloromethane prior to clean-up using gel permeation chromatography. The eluate was evaporated to dryness and reconstituted in 0.1% corn oil in tri-methyl pentane containing 1,4-dibromonaphthalene as an internal standard. Quinoxyfen was quantified by gas chromatography with mass selective detection. The 1,4-dibromonaphthalene quantification ion is 286 amu; the quinoxyfen quantification ion is 272 amu, with confirmation ions 237 amu and 307 amu.

The method has been validated by the analysis of untreated samples and samples of whole eggs and egg yolks fortified with quinoxyfen over the range of 0.01 mg/kg to 1.0 mg/kg. Results of the independent laboratory validation were consistent with the initial validation results.

Table 38. Recovery of quinoxyfen from fortified samples of eggs (64399).

Fortification level (mg/kg)	% Recovery[1]	Average % Recovery	% RSD
0.01	109, 105[2]	98 ± 7.3	7.4 (n=8)
0.01	101, 95		
0.01	101, 96		
0.01	85, 95		
0.10	93, 97	97 ± 2.6	2.7 (n=4)
0.10	98, 99		
0.5	92, 90	94 ± 4	4.3 (n=4)
0.5	96, 99		
1.0	93, 87	87 ± 4.5	5.2 (n=4)
1.0	87, 82		
0.01-1.0	82 - 109	95 ± 6.7	7.1 (n=20)

[1] Mean control values: whole eggs = 0.0000 (n=6); yolk = 0.0000 (n=6).

[2] Replicate samples.

Table 39. Summary of ILV recovery results for quinoxyfen residues in fortified eggs (135045).

Matrix	Fortification level (mg/kg)	n	% Recovery (range)	Average % recovery	% RSD
Whole eggs	0.01	5	73 - 104	85	15
	1.0	5	79 - 105	92	13
	0.01 - 1.0	8	73 - 105	88 ± 12.3	14
Egg yolk	0.01	5	73 - 78	76	3
	1.0	5	73 - 84	81	6
	0.01 – 1.0	10	73 - 84	78 ± 4.7	6

[1]Corrected from appropriate control value

Multi-residue Methods

Multi-residue method testing for quinoxyfen according to US FDA PAM I, Appendix II**,** as updated January 1994 was reported to the Meeting (80711, Hackert Anderson and West, S., 2001).

Quinoxyfen was analyzed according to the FDA Multiresidue Method Testing guidelines in PAM, Vol. I, Appendix II (1/94). Protocol A. Quinoxyfen was evaluated to see if it was naturally fluorescent. With an excitation wavelength of 245 nm and 300 nm, the compound showed no emission response above that of the methanol blank. Therefore, quinoxyfen does not naturally fluoresce.

Protocol C. The gas chromatographic behaviour of quinoxyfen was evaluated according with three column types (DB-1, DB-17, and DB-225) in combination with electron capture detection (ECD) or nitrogen-phosphorous detection (NPD). Quinoxyfen chromatographed within acceptable limits under Level I guidelines using the DB-1, DB-17, and DB-225 with ECD at 200ºC and also on the DB-1 with NPD at 200ºC.

Protocol D. Testing (without Florisil cleanup) for recovery of quinoxyfen from grapes utilizing NPD as a specific detector resulted in an average recovery of 27% at 0.1 mg/kg and 75% at 0.05 mg/kg.

Protocols E and F required Florisil cleanup. For the C1 elution system, the majority of quinoxyfen eluted in fraction 2. The majority of quinoxyfen eluted in the third fraction for the C2 system.

Protocol E testing with grapes yielded total average quinoxyfen recoveries of 37% (low fortification) and 102% (high fortification) for Florisil cleanup C1. The results show incomplete recovery of quinoxyfen at low fortification level (0.05 mg/kg) and complete recovery at the high fortification level (0.5 mg/kg). Since quinoxyfen is recoverable through the method with the C1 Florisil cleanup, the testing was repeated using the C2 Florisil cleanup. Average recoveries from grapes were 94% (low fortification) and 88% (high fortification) for Florisil cleanup C2, showing complete recovery through the method.

Protocol F testing with ground beef yielded total average quinoxyfen recoveries of 12% (low fortification) and 70% (high fortification) for Florisil cleanup C1 and 30% (low fortification) and 93% (high fortification) for Florisil cleanup C2. The results show incomplete or partial recovery at the low fortification level (0.05 mg/kg) and complete recovery at the high fortification level (0.5 mg/kg) with the C1 and C2 Florisil cleanup.

Multi-residue Method DFG S19 – Validation of method for determination of quinoxyfen in wheat, barley, grapes, strawberries, melons and other matrices by GC-ECD or GC-MSD (31651, Hastings and Schmidt, 1995)

A multi-residue method, DFG S19 was validated for use in the analysis of quinoxyfen in a number of plant matrices. The method involves extraction of plant samples with acetone: water solution (maintained constant at a ratio of 2:1, v/v). The extract was saturated with sodium chloride and diluted with dichloromethane, resulting in separation of excess water. The organic phase was separated and evaporated to dryness. The residue remaining was taken up in dichloromethane and was cleaned up by gel permeation chromatography. The eluate was concentrated and after supplemental clean up on a small silica gel column, quinoxyfen residues are determined by gas chromatography using capillary column and electron capture detector or mass selective detector.

Strawberry samples were extracted after adding sodium bicarbonate solution, since the original method resulted in unacceptable recoveries (about 60%).

Control samples of various matrices were each fortified with quinoxyfen at levels of 0.01 mg/kg and 0.5 mg/kg. The results are summarized in Table 40.

Table 40. Summary of recoveries of quinoxyfen from wheat, barley, melons, strawberries, and grapes at fortification levels of 0.01 and 0.5 mg/kg (MRM DFG S19) (31651).

Analyte	Recovery range (%)	Mean recovery (%)	% RSD
Winter wheat			
Grain	90 – 99	96	4 (n=4)
Straw	87 – 99	93	6 (n=4)
Winter barley			
Grain	84 – 98	91	7 (n=4)
Straw	79 – 100	92	10 (n=4)
Melons			
Peel	90 – 97	94	3 (n=1)
Pulp	95 – 97	96	1 (n=1)
Strawberries	69 – 88	78	10 (n=4)
Grapes	74 – 87	81	7 (n=4)

Method DFG S19 for the determination of residues of quinoxyfen on hops by GC-MSD (73994, Oberwalder, 1999), modified, was used for the supervised trials on hops conducted in Germany. The method was validated for hops by fortification of untreated samples with quinoxyfen at concentration levels of 0.01 mg/kg to 1.0 mg/kg. Recovery data are summarized in Table 41.

Table 41. Recovery of quinoxyfen in fortified samples of fresh and dried hops with MRM DFG S19 (73994).

Matrix	Fortification level (mg/L)	% Recovery
FRESH HOPS CONES	0.01	150[1]
	0.01	70
	0.02	85
	0.02	70
	0.02	90
	0.50	90
	0.50	73
	1.0	91
	1.0	93
Mean	0.01-1.0	85 ± 11
% RSD over the validated range (0.01-1.0 mg/kg)		12 (n=8)
DRIED HOPS CONES	0.02	105
	0.02	100
	0.50	93
Mean	0.02-0.50	99 ± 6
% RSD over the validated range (0.02 – 0.5 mg/kg)		6.1 (n=3)

1 Outlier according to Dixon Test; not included in calculation of the mean.

Stability of Pesticide Residues in Stored Analytical Samples

Frozen storage stability studies were reported to the Meeting for a variety of substrates that include animal tissues and plants. Control samples were fortified with known concentrations of quinoxyfen and then placed in frozen storage at approximately -20 C or less. The fortified samples were analyzed periodically for residues of quinoxyfen using the same analytical method as that used for the residue field trial or processing samples.

The stability of frozen samples was evaluated as part of the supervised trials on cherries (102721, 102722, Chen, 2002). The maximum storage interval for field-treated samples was 77 days. To evaluate stability of residues during this period, control samples of each matrix were fortified with 1.0 mg/kg quinoxyfen and analyzed after 80 days of frozen storage at -18ºC. All samples were analyzed within 1 day of extraction. Method ERC 95.26, which was validated and used for the determination of quinoxyfen in the supervised trials for cherries, was used for the storage stability test. Concurrent recoveries from samples fortified on the day of analysis were comparable to the test samples.

Table 42. Stability of quinoxyfen residues in cherries after frozen storage (102721, 102722).

Matrix	Fortification level (mg/kg)	No. of days in frozen storage	Quinoxyfen conc (mg/kg)	% Remaining	% Concurrent recovery[1]	Reference
Cherry fruit	1.0	80	0.908	91	94	Chen, H., 2002a IR-4 Study 07757
	1.0	80	0.926	93	94	
	1.0	80	0.918	92	95	
			Average	92	94	

[1] Concurrent recovery is from samples fortified on the day of analysis. % remaining was not corrected for the concurrent method recovery.

Numerous field trials on grapes were conducted in Europe, Australia, and the US. Throughout these trials, samples of grapes were stored frozen from 5.5 to about 11 months before analysis. A study was undertaken to determine the stability of quinoxyfen residues in samples of grapes stored

frozen (47234, Williams, 1996). Untreated samples of macerated grapes from one of the trials conducted in Europe were combined and mixed briefly in a Waring blender. Thirty six aliquots, each being 10 g of the grape sample, were fortified with 0.10 mg/kg quinoxyfen. A further 36 aliquots of 10 g each were left unfortified. All samples were stored below -16ºC and a proportion of both fortified and untreated samples were analyzed at intervals of 0, 3, 6, 9, and 12 months of storage.

Samples were analyzed according to Method ERC 94.29, which was previously validated over the range of to a lowest validated level of 0.01mg/kg for grapes and was used in the supervised trials. The results are summarized in Table 43.

Untreated samples of grapes (approximately 10 g each) were taken from the trial in the US in which portion of the harvest was processed and fortified with 0.10 mg/kg quinoxyfen (Thompson, 2001a; IR-4 Study 07256). Untreated juice and raisin samples were likewise collected after processing and fortified with 0.1 mg/kg quinoxyfen. All samples were then frozen at –18ºC. Frozen grape samples were analyzed after 206 days of storage, while juice and raisin samples were analyzed after 155 and 254 days of frozen storage, respectively. All samples were analyzed using method ERC 95.26, which had previously been validated for use on grapes and its processed fractions, with a lowest validated level of 0.01 mg/kg. The results are included in Table 43.

Table 43. Residues of quinoxyfen in grapes and grape processed fractions during frozen storage (47234).

Matrix	Storage Period, Days	Fortification mg/kg	% Remaining	Concurrent % Recovery[1]	Reference
Grapes	0	0.1	90	93	Khoshab, A. and Williams, M., 1996 (GHE-P-5423)
		0.1	93	93	
		0.1	88		
		0.1	90		
		0.1	102		
		0.1	100		
		Average	93.8	93	
Grapes	90	0.1	110	101	
		0.1	104	97	
		Average	107	99	
Grapes	180	0.1	96	93	
		0.1	96	95	
		Average	96	94	
Grapes	270	0.1	93	90	
		0.1	94	92	
		Average	93.5	91	
Grapes	365	0.1	97	100	
		0.1	98	99	
		Average	97.5	99.5	
Grapes	206	0.10	87	92	Thompson, 2001a (IR-4 Study 07256)
		0.10	86	91	
		0.10	85		
		Average	86	91.5	
Grape juice	255	0.01	93	89	
		0.01	93	93	
		0.01	81		
		Average	89	91	
Raisins	254	0.01	99	99	
		0.01	100	101	
		0.01	98		
		Average	99	100	

[1] Concurrent recovery is from samples fortified on the day of analysis. % remaining was not corrected for concurrent method recovery.

Samples of wheat grain and straw were taken from one of the supervised trials conducted in Europe. These samples were prepared with dry ice in a mill. Aliquots of each substrate (10g for grain

and 5g for straw) were weighed into 57 labelled 150 mL polypropylene pots. Thirty samples of grain were fortified with 0.1 mg/kg quinoxyfen, while 30 samples of straw were fortified with 1.0 mg/kg quinoxyfen. The remaining 27 samples of each substrate were retained as controls. All samples were then transferred to a freezer and kept frozen at temperatures ranging from -18ºC to -24ºC during the study with an average temperature of -23ºC. The storage intervals were 0, 98, and 267 days for grain and 0, 97, and 280 days for straw (31748, Gambie 1995). Grain samples were kept frozen and analyzed after 534 days. Straw samples were analyzed after 536 days (47586, Gambie, 1996)

Samples were analyzed using method ERC 94.5, which had previously been validated with a lowest validated level of 0.01 mg/kg for grain and 0.05 mg/kg for straw. The results from both studies are summarized in Table 44.

Table 44. Residues of quinoxyfen in cereal grain and straw during frozen storage (31748; 47586).

Matrix	Storage Period, Days	Fortification mg/kg	% Remaining	Concurrent% Recovery[1]	Reference
Grain	0	0.1	92	108	Gambie, A. and Long, T., 1995 (GHE-P-4409)
		0.1	81	109	
		0.1	97		
		0.1	91		
		0.1	100		
		0.1	103		
		Average	94	108	
Grain	98	0.1	100	102	
		0.1	99	101	
		0.1	100		
		Average	99	102	
Grain	267	0.1	106	89	
		0.1	92	101	
		0.1	97		
		Average	98	95	
Grain	453	0.1	106	105	Gambie, A., 1996 (GHE-P-5438)
		0.1	111	107	
		0.1	113		
		Average	110	106	
Grain	534	0.1	75	73	
		0.1	77	75	
		0.1	74		
		Average	75	74	
Straw	0	0.1	91	88	Gambie, A. and Long, T., 1995 (GHE-P-4409)
		0.1	95	87	
		0.1	92		
		0.1	92		
		0.1	97		
		0.1	96		
		Average	94	88	
Straw	97	0.1	72	75	
		0.1	79	73	
		0.1	80		
		Average	77	74	
Straw	280	0.1	85	88	Gambie, A. and Long, T., 1995 (GHE-P-4409)
		0.1	86	87	
		0.1	87		
		Average	86	87	
Straw	456	0.1	87	101	Gambie, A., 1996 (GHE-P-5438)
		0.1	96	94	
		0.1	93		
		Average	92	97	

Matrix	Storage Period, Days	Fortification mg/kg	% Remaining	Concurrent% Recovery[1]	Reference
Straw	536	0.1	75	78	
		0.1	78		
		0.1	74		
		Average	75	78	

[1] Concurrent recovery is from samples fortified on the day of analysis. % remaining was not corrected for concurrent method recovery.

The stabilities of frozen samples of hops, lettuce, strawberry, peppers, and cantaloupe (melon) were evaluated as part of the supervised trials (83727, Thompson, 2001; 208698, Barney, W., 2005; 208697, Barney, 2005; 8006, Chen, 2003; 208696, Corley, 2004). To evaluate the stability of residues during this period, control samples of commodity were fortified with quinoxyfen and analyzed after some days of frozen storage at -15ºC. Samples were generally analyzed on the day of extraction. No samples were analyzed on day 0. Method ERC 95.26 was used for the storage stability tests.

Table 45. Residues of quinoxyfen in hops (dried), lettuce, strawberry, peppers, and melon during frozen storage.

Matrix	Storage Period, Days	Fortification mg/kg	% Remaining	Concurrent % Recovery[1]	Reference
Hops	113	0.5	105	109	83727
		0.5	104	107	
		0.5	110	106	
		Average	106	108	
Lettuce (leaf)	280	0.10	93	95	208698
		0.10	94	96	
		0.10	88	95	
		Average	92	95	
Strawberry	162	0.10	92	98	208697
		0.10	97	94	
		0.10	94	97	
		Average	94	96	
Melon (cantaloupe)	251	0.50	89	94	208696
		0.50	95	95	
		0.50	91	93	
		Average	92	94	
Peppers, Bell	318	1.0	98	-	8006
		1.0	104	-	
		1.0	104	-	

[1] Concurrent recovery is from samples fortified on the day of analysis. % remaining was not corrected for concurrent method recovery.

Samples of whole milk from the dairy cow feeding study were stored for up to 87 days prior to analysis. In order to assess the stability of residues of quinoxyfen in stored frozen samples, twelve 10-g samples of untreated whole milk were fortified with 0.1 mg/kg and kept frozen until analysis. In order to simulate the conditions in the study, milk was initially stored for 4 days at + 4ºC, prior to deep freezing (31599, Gambie and Long, 1995).

Fortified samples, together with untreated samples were analyzed using method ERC 94.7, which has previously been validated with an LOQ of 0.001 mg/kg for milk. Samples were removed from storage and analyzed after 0, 161 and 238 days of frozen storage. Two untreated samples were fortified with 0.1 mg/kg on each day of analysis and analyzed for concurrent recoveries. The results are summarized in Table 46.

Table 46. Residues of quinoxyfen in whole milk samples during frozen storage (31599).

Matrix	Storage Period, Days	Fortification mg/kg	% Remaining	Concurrent % Recovery
Milk	0	0.1	93	90
		0.1	90	89
		0.1	91	
		0.1	90	
		0.1	90	
		0.1	89	
		Average	90	90
Milk	161	0.1	82	76
		0.1	81	85
		0.1	83	
		Average	82	80
Milk	238	0.1	86	89
		0.1	91	88
		Average	88	88

The liver, kidney, muscle, subcutaneous and peritoneal fat samples were stored up to 244, 190, 209, and 218 days, respectively during the cow feeding study (31599, Gambie, and Long,1995). In order to evaluate the stability of quinoxyfen residues on frozen storage, tissue samples from the control group were fortified with 0.1 mg/kg quinoxyfen and frozen at < 20ºC until analysis. Liver samples were analyzed using method ERC 94.30, which has been previously validated with an LOQ of 0.01 mg/kg. The rest of the tissues were analyzed with method ERC 94.20, with a lowest validated level of 0.01 mg/kg. Concurrent recoveries were run with control samples fortified with 0.1 mg/kg quinoxyfen on the day of analysis. The results are summarized in Table 47.

Table 47. Residues of quinoxyfen in animal tissue samples during frozen storage (31599).

Matrix	Storage Period, Days	Fortification mg/kg	% Remaining	Concurrent% Recovery[1]
Liver	242	0.1	64 (107[1])	61
		0.1	60 (100[1])	61
		0.1	44 (44[1])	
		Average	56 (93[1])	61
Liver	292	0.1	65	67
		0.1	69	66
		0.1	64	
		Average	66	66.5
Kidney	188	0.1	90	94
		0.1	94	91
		0.1	94	
		Average	93	92.5
Muscle	194	0.1	87	91
		0.1	86	91
		0.1	84	
		Average	86	91

Matrix	Storage Period, Days	Fortification mg/kg	% Remaining	Concurrent% Recovery[1]
Subcutaneous fat	207	0.1 0.1 0.1 Average	93 94 101 96	90 89 89.5
Peritoneal fat	216	0.1 0.1 0.1 Average	80 79 87 82	79 89 84

[1] Values in parenthesis are corrected for concurrent recoveries. No other % Remaining values were corrected for concurrent method recoveries.

USE PATTERN

Quinoxyfen is a protectant fungicide for the control of powdery mildew diseases in a range of crops. Quinoxyfen does not control existing or latent powdery mildew infections and therefore, it must be applied before symptoms of the disease appear, on a protectant schedule. The product is diluted with water and applied as foliar spray or broadcast treatment using conventional spray equipment.

Quinoxyfen is registered for use in a wide range of crops in several countries. Only the registered uses in countries where supervised trials have been conducted or in countries with GAPs similar to where the supervised trials were carried out, are provided and summarized in Table 48. This summary is based on official labels provided by the sponsor and by use information supplied by the governments of Australia, Germany, and the Netherlands.

Table 48. Summary of GAP uses for quinoxyfen.

Crop	Country	Formulation	Application				PHI days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hL	No. or max (kg ai/ha/ season)	
Blueberry	Germany	250 g/L SC	Foliar	0.075		3	14
Cereals (Wheat, barley, oats, rye, triticale)	UK	500 g/L SC	Foliar	0.15	0.0375 – 0.075	2 0.3 kg ai/ha	~60 Zadoks 49 (first awns)
Cereals (Wheat, barley, oats, rye, triticale)	UK	250 g/L SC	Foliar	0.15	0.0375 – 0.075	2 0.3 kg ai/ha	~60 Zadoks 49 (first awns)
Cereals (Wheat, barley, oats, rye, triticale)	UK	66.7 g/L + 250 g/L Fenpropimorph	Foliar	0.1 – 0.2		2	~60 Zadoks 49 (first awns)
Cherry	USA	250 g/L SC (2.08 lbs/gal)	Foliar	0.12		5, maximum 0.63 kg ai/ha/year 7-day intervals	7
Currants	Germany	250 g/L SC	Foliar	0.075		3	14
Gooseberries	Germany	250 g/L SC	Foliar	0.075		3,	14
Grapes	Australia	250 g/L SC	Foliar	0.05 (calculated)	0.0025 at 7-10-day intervals; 0.005 at 10-14-day intervals	3	14

Crop	Country	Formulation	Application				PHI days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hL	No. or max (kg ai/ha/ season)	
Grapes, table and wine	France	250 g/L SC	Foliar	0.05		3, 7-10-day intervals between 2 leaves unfolded and end of bunch closure	21
Grapes, table and wine	Germany	200 g/L + 60 g/L fenarimol	Foliar	0.08	0.005	4, 10-14-day intervals	21
Grapes, table and wine	Italy	250 g/L SC	Foliar		0.0075	5, 8-14-day intervals	28
Grapes, table and wine	Spain	250 g/L SC	Foliar	0.075	0.0075	5, 10-18-day intervals	30 (wine) 21 (table)
Grapes	USA	250 g/L SC	Foliar	0.018 – 0.12 0.036 at 7 day interval 0.072 at 14 day interval 0.12 at 21 day interval		5, 7-21-day intervals; maximum 0.60 kg ai/ha/year	14
Grapes, and wine	France	200 g/L (+ 60 g/L fenarimol) SC	Foliar	0.04		3, 7 – 10 day-intervals	21
Grapes, table and wine	Italy	200 g/L (+ 60 g/L fenarimol) SC	Foliar		0.005 – 0.0075	5, 8 – 10 day intervals	28
Grapes, table and wine	Spain	200 g/L (+ 60 g/L fenarimol) SC	Foliar		0.005 – 0.0075	5, 10-19-day intervals	30 (wine) 21 (table)
Hops	Germany	250 g/L SC	Foliar	0.0675 – 0.15	0.005 – 0.011	4, 10-14-day intervals, maximum 0.5 kg ai/ ha/ season	28
Hops	USA	250 g/L SC	Foliar	0.073 – 0.15		4, 7-day intervals; maximum 0.60 kg ai/ha/year	21
Lettuce, head and leaf	USA	250 g/L SC	Foliar	0.073 – 0.11	0.026 -0.039 (calculated)	4, maximum 0.44 kg ai/ha/season. 10 – 14 day intervals	1
Melons	Italy	250 g/L SC	Foliar		0.004 – 0.006	10-12 days intervals	7
Melons	Italy	200 g/L (+ 60 g/L fenarimol) SC	Foliar		0.005 – 0.006	10-12-days interval	7

Crop	Country	Formulation	Application				PHI days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hL	No. or max (kg ai/ha/ season)	
Melons	Spain	250 g/L SC	Foliar	0.075	0.0075	3, 5 – 7 day interval	7
Melons	Spain	200 g/L (+ 60 g/L fenarimol) SC	Foliar		0.005 – 0.0075	3, 10-14 day interval	7
Melons	USA	250 g/L SC	Foliar	0.073 – 0.11	0.039 (calculated)	4, maximum 0.44 kg ai/ha/season	3
Peppers (all)	USA	250 g/L SC	Foliar	0.15	0.02 (calculated)	4, maximum 0.60 kg ai/ha/season 7 day interval	3
Strawberry	Germany	250 g/L SC	Foliar	0.125	0.006	2, 7-21-day intervals. Field and glasshouse	14
Strawberry	USA	250 g/L SC	Foliar	0.073 – 0.11	0.026 – 0.039 (calculated)	4, maximum 0.44 kg ai/ha/season 10 – 14 day interval	1
Sugarbeets	France	500 g/L SC	Broadcast	0.15	0.05 – 0.1	1	28
Sugarbeets	Germany	500 g/L SC	Broadcast	0.12	0.031- 0.062	2	28
Sugarbeets	UK	500 g/L	Broadcast	0.15	0.038 – 0.075	2 0.2 kg ai/ha	28
Sugarbeets	UK	250 g/L	Broadcast	0.15	0.038 – 0.075	2 0.2 kg ai/ha	28
Watermelons	Italy	250 g/L SC	Foliar		0.004 – 0.006	10-12 days intervals	7
Watermelons	Italy	200 g/L (+ 60 g/L Fenarimol) SC	Foliar		0.005 – 0.006	10-12-days interval	7
Watermelons	Spain	250 g/L SC	Foliar	0.075	0.0075	3, 5 – 7 day interval	7
Watermelons	Spain	200 g/L (+ 60 g/L Fenarimol) SC	Foliar		0.005 – 0.0075	3, 10-14 day interval	7
Wheat and Barley	France	500 g/L SC	Foliar	0.15	0.05 – 0.1	1	56
Wheat and Barley	France	200 g/L + (66.7g/L fenpropimorph) SC	Foliar	0.1	0.03 – 0.07	1	56
Wheat and Barley	Germany	500 g/L SC	Foliar	0.15	0.025 – 0.075	2 0.25 kg ai/ha	49 (first awns visible)
Wheat and Barley	Germany	500 g/L SC	Foliar	0.25	-	1	BBCH 25 - 32
Wheat	Netherlands	500 g/L SC	Foliar	0.15	0.025-0.075	1	60

Crop	Country	Formulation	Application				PHI days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hL	No. or max (kg ai/ha/ season)	
Wheat	Netherlands	500 g/L SC	Foliar	0.15	0.025-0.075	1	60

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RESIDUES RESULTING FROM SUPERVISED TRIALS

The results of supervised trials are shown in Tables 50 to 64. Where multiple samples were taken from a single plot or multiple analyses conducted on a single sample, the average value is reported. Where results from separate plots with distinguishing characteristics such as different formulations, varieties or treatment schedules were reported, results are listed for each plot Results have not been corrected for concurrent method recoveries unless indicated. The following table summarizes information on residues resulting from supervised trials.

Table 49: Field trials.

Group	Commodity	Table No.
Stone fruit	Cherries	50
Berries and other small fruits	Grapes	51
	Strawberries	52
	Currants	53
Fruiting vegetables, cucurbits	Melons	54 - 55
Fruiting vegetables other than cucurbits	Peppers	56
Leafy vegetables	Lettuce	57
Root and tuber vegetables	Sugar Beets	58
Cereal grains	Wheat	59
	Barley	60
Straw, fodder and forage of cereal grains and grasses (straws and fodder dry)	Wheat	61
	Barley	62
Dried herbs	Hops	63
Miscellaneous fodder and forage crops	Sugar beet tops	64

Stone fruit – adapted in part from the Evaluation of the USA

A total of 13 supervised field trials on cherries were conducted in major cherry growing areas of the USA in 2000 and 2001 (102722; 102721, Chen, 2002). Each treated plot received five foliar-directed applications of a suspension concentrate formulation containing 250 g/L quinoxyfen at the rate of approximately 0.12 kg ai/ha (0.11 lbs/acre) per application. There were no adjuvants in the tank mix. The retreatment interval was 6 – 8 days, typically 7 days. The period of time from harvest to analysis ranged from 35 to 77 days. Cherries were pitted prior to analysis.

Table 50. Quinoxyfen residues in cherries (pitted) from foliar directed application in the USA.

Cherries country, year (variety)	Application					PHI days	Residues[1], mg/kg	Reference
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
GAP, USA	250 g/L SC	0.12			5, max 0.63 kg ai/ha/ year	7		
Trial 00-M121 Michigan, USA, 2000 (Montmorency)	250 g/L SC	0.12	0.013	929- 951	5, total = 0.62 kg ai/ha/yr	7	0.13	102722
Trial 00-M122 Michigan, USA, 2000 (Montmorency)	250 g/L SC	0.12	0.013	932- 946	5, total = 0.62 kg ai/ha/yr	7	0.08	102722
Trial 00-M123 Michigan, USA, 2000 (Montmorency)	250 g/L SC	0.12	0.013	930- 948	5, total = 0.62 kg ai/ha/yr	7	0.11	102722
Trial 00-M124 Michigan, USA, 2000 (Montmorency)	250 g/L SC	0.12	0.013	930- 945	5, total = 0.62 kg ai/ha/yr	7	0.14	102722
Trial 00-M125 Michigan, USA, 2000 (Emperor Francis)	250 g/L SC	0.13	0.022	571- 575	5, total = 0.63 kg ai/ha/yr	7	0.12	102722
Trial 00-M126 Michigan, USA, 2000 (Hedelfinger)	250 g/L SC	0.13	0.022	571- 579	5, total = 0.63 kg ai/ha/y	7	0.13	102722
Trial 00-WA39 Washington, USA, 2000 (Bing)	250 g/L SC	0.13	0.009-0.010	1312-1399	5, total = 0.63 kg ai/ha/yr	7	0.14	102722
Trial 00-WA40 Washington, USA, 2000 (Bing)	250 g/L SC	0.12	0.011	1102-1159	5, total = 0.61 kg ai/ha/yr	7	0.11	102722
Trial 00-WA41 Washington, USA, 2000 (Montmorency)	250 g/L SC	0.12	0.005-0.006	2144-2178	5, total = 0.62 kg ai/ha/yr	6	0.05	102722
Trial 00-CO01 Colorado, USA, 2000 (Montmorency)	250 g/L SC	0.11- 0.12	0.006	1765-1914	5, total = 0.60 kg ai/ha/yr	6	0.15	102722
Trial 00-PA01 Pennsylvania, USA , 2000 (Montmorency)	250 g/L SC	0.12 – 0.13	0.011	1122-1159	5, total = 0.63 kg ai/ha/yr	6	0.27	102722
Trial 01-CA49 California, USA, 2001 (Brooks)	250 g/L SC	0.12	0.009-0.010	1239-1360	5, total = 0.61 kg ai/ha/yr	7	0.03	102721
Trial 01-CA50 California, USA, 2001 (Brooks)	250 g/L SC	0.13	0.008-0.009	1469-1624	5, total = 0.63 kg ai/ha/yr	8	0.08	102721

[1] Average of duplicate field samples from the same plot.

Berries and other small fruits

A total of 57 supervised trials were conducted on grapes in the following countries: France (9), Germany (6), Italy (11), Spain (6), US (13), Canada (4), and Australia (8). Results are summarized in Table 51.

A typical residue decline curve from the foliar application to grapes is that from Trial R94-049 in Spain. Six applications were made at 9 – 10 day intervals, and grape samples were taken at intervals of 1 – 62 days after the final treatment. The half-life is 19 days for first-order kinetics.

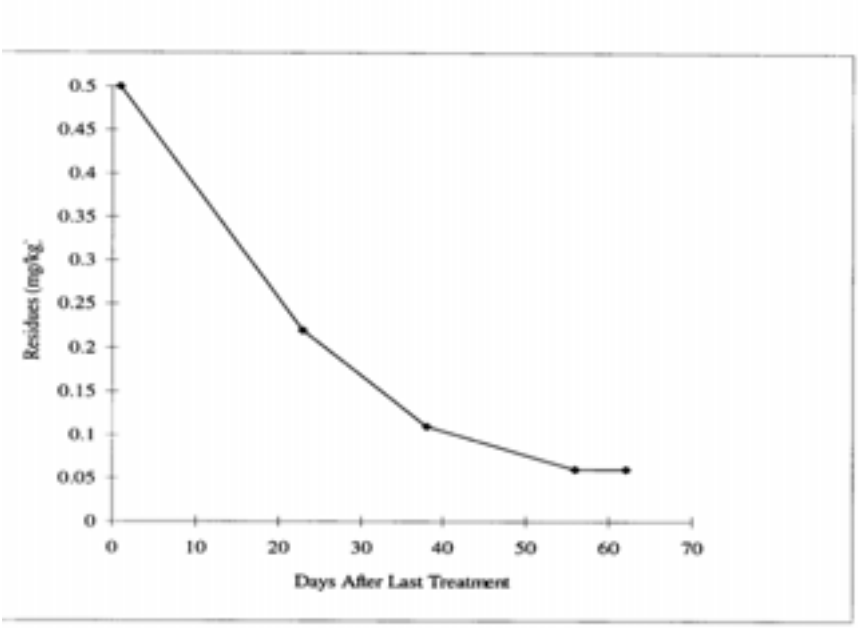


Table 51. Quinoxyfen residues in grapes from foliar applications in France, Germany, Italy, Spain, USA, Canada and Australia.

Grapes	Application[1]					PHI	Residues mg/kg	Reference
country, year (variety)	Form	kg ai/ha	kg ai/hL	water, L/ha	no.	days		
GAP, France (Table and wine)	250 g/L SC	0.05			3 appl/yr at 7-10-day intervals	21		
Trial D3T6S361 France, 1993 (Cinsault, Wine)	500 g/L SC	0.029 – 0.045	0.0074-0.0076	390-730	6 at 8-11 days interval	64	0.03	42674
Trial D3T6S361 France, 1993 (Cinsault, Wine)	500 g/L SC	0.031 – 0.058	0.0074-0.0076	410- 770	8 at 8-11 days interval	43	0.04	
Trial D3T6S361 France, 1993 (Cinsault, Wine)	500 g/L SC	0.031-0.055	0.0075	410-730	10 at 8-11 days interval	22	0.06	42674
Trial R94-014 France, 1994 (Italia, table)	250 g/L SC	0.05	0.025	200	6 at 6-13 days interval	0 7 15 22 30	0.12 0.06 0.05 0.04 0.04	43622
Trial R94-015 France, 1994 (Gamay, Wine)	250 g/L SC	0.05	0.025	200	6 at 8-13 days interval	0 6 13 20 27	0.39 0.10 0.07 0.13 0.07	43621
Trial R94-016B France, 1994 (Pinot Noir, Wine)	250 g/L SC	0.05	0.027	190	6 at 8-12 days interval	32	0.04	43620

Grapes country, year (variety)	Application[1] Form	kg ai/ha	kg ai/hL	water, L/ha	no.	PHI days	Residues mg/kg	Reference
Trial R94-017 France, 1994 (Pinot Meunierr, Wine)	250 g/L SC	0.05	0.024	210	6, at 11-13 days interval	0 10 21 28 34	0.05 0.04 <u>0.02</u> 0.02 0.02	43816
Trial R94-017 France, 1994 (Pinot Noir, Wine)	250 g/L SC	0.05	0.027	190	6, at 8-13 days interval	0 11 18 25 32	0.12 0.06 <u>0.05</u> 0.04 0.05	
Trial R95-037 France, 1995 (Chenin, Wine)	250 g/L SC	0.06	0.047	130	6, at 11-14 days interval	0 5 9 15 20	0.10 0.08 0.11 0.08 <u>0.09</u>	45716
GAP, Germany (Table and wine)	250 g/L SC Or 200 g/L + 60 g/L fenarimol SC	0.08	0.005	1600	Max 4, at 10-14 day interval	21		
Trial R95-038A Germany, 1995 (Silvaner, Wine)	200 g/L SC + 60 g/L fenarimol	0.068-0.072	0.010- 0.012	610-810	7, at 11-12 days interval	1 5 10 15 21	0.35 0.24 0.21 0.21 0.24	45717
Trial R95-038A Germany, 1995 (Silvaner, Wine)	200 g/L SC + 60 g/L fenarimol	0.020-0.021	0.003- 0.004	610- 810	7, at 11-12 days interval	1 5 10 15 21	0.05 0.06 0.06 0.06 <u>0.04</u>	
Trial R95-038B Germany, 1995 (Silvaner, Wine)	200 g/L SC + 60 g/L fenarimol	0.020-0.021	0.003- 0.004	610- 810	7, at 11-12 days interval	1 5 10 15 21	0.20 0.25 0.28 0.10 <u>0.36</u>	45717
Trial R95-038B Germany, 1995 (Silvaner, Wine)	200 g/L SC + 60 g/L fenarimol	0.019-0.022	0.003- 0.004	600- 840	7, at 11-12 days interval	1 5 10 15 21	0.04 0.06 0.05 0.06 <u>0.05</u>	
Trial R95-039A Germany, 1995 (Riesling, Wine)	200 g/L SC + 60 g/L fenarimol	0.066-0.077	0.009- 0.012	600-840	7, at 11-13 days interval	21	0.21	45718
Trial R95-039B Germany, 1995 (Riesling, Wine)	200 g/L SC + 60 g/L fenarimol	0.062-0.068	0.009- 0.012	560-770	7, at 11-12 days interval	21	0.16	
GAP, Italy (Table and wine)	250 g/L SC OR 200 g/L +60 g/L fenarimol		0.008		Max 5/year, at 8 – 14 days interval	28		

Grapes country, year (variety)	Application[1] Form	kg ai/ha	kg ai/hL	water, L/ha	no.	PHI days	Residues mg/kg	Reference
Trial R94-046A Italy, 1994 (Wine-White Marzemino)	250 g/L SC	0.094	0.008	1200	6 at 11-12 days interval	0 6 10 15 20 30	0.50 0.36 0.20 0.20 0.13 0.17	42673
Trial R94-046B Italy, 1994 (Wine-White Marzemino)	250 g/L SC	0.125	0.010	1200 - 1300	6 at 10-12 days interval	0 6 10 15 20 30	0.65 0.34 0.48 0.43 0.36 0.30	
Trial R94-047A Italy, 1994 (Italia, Table)	250 g/L SC	0.96- 0.103	0.008	1300 - 1400	6 at 10-11 days interval	0 4 9 14 19 30	0.30 0.24 0.24 0.26 0.24 0.18	43416
Trial R94-047B Italy, 1994 (Italia, Table)	250 g/L SC	0.136-0.138	0.010	1400	6 at 10-11 days interval	0 4 9 14 19 30	0.55 0.25 0.52 0.36 0.34 0.10	
Trial R94-048A Italy, 1994 (Cardinale, Table)	250 g/L SC	0.083-0.111	0.01	830-1100	6 at 9-11 days interval	21 32	0.10 0.06	43179
Trial R94-048B Italy, 1994 (Cardinale, Table)	250 g/L SC	0.05- 0.083	0.008	670-1100	6 at 9-11 days interval	21 32	0.07 0.04	
Trial R95-027 Italy, 1995 (Prosecco, Wine	250 g/L SC	0.062-0.065	0.006	1000	7 at 3-13 days interval	20	0.30	44917
Trial R95-028A Italy, 1995 (Garganega, Wine)	200 g/L SC+ 60 g/L fenarimol	0.07-0.072	0.007	1000	7 at 10-12 days interval	21	0.49	45173
Trial R95-028B Italy, 1995 (Prosecco, Wine)	200 g/L SC+ 60 g/L fenarimol	0.07-0.071	0.007	1000	7 at 10-13 days interval	20	0.28	45173
Trial R95-029A Italy, 1995 (Italia, Table)	200 g/L SC+ 60 g/L fenarimol	0.062-0.065	0.006	1000	7 at 10-13 days interval	20	0.32	45715
Trial R95-029B Italy, 1995 (Italia, Table)	200 g/L SC+ 60 g/L fenarimol	0.069-0.073	0.007	1000	7 at 10-13 days interval	20	0.25	45715
GAP, Spain (Table and wine)	250 g/L SC	0.075	0.008		Max 5/ season, at 10 – 18 day intervals	30, wine grapes; 21, table grapes		

Grapes country, year (variety)	Application[1] Form	kg ai/ha	kg ai/hL	water, L/ha	no.	PHI days	Residues mg/kg	Reference
Trial R94-049 Spain, 1994 (Malabeo, Wine)	250 g/L SC	0.042 0.052 0.039 0.047 0.050 0.050	0.008	520- 690	6 at 9-10 days interval	1 23 38 56 62	0.50 <u>0.22</u> 0.11 0.06 0.06	43180
Trial R94-050 Spain, 1994 (Malabeo, Wine)	250 g/L SC	0.038 0.040 0.044 0.040 0.058 0.056	0.0075	510- 780	6 at 9-10 days interval	62	0.04	43181
Trial R95-032 Spain, 1995 (Italia, Table)	200 g/L SC+ 60 g/L fenarimol	0.070	0.007	950	6 at 11-14 days interval	21	<u>0.08</u>	45531
Trial R95-033 Spain, 1995 (Blanca Apireta, Table)	250 g/L SC	0.060- 063	0.01 –0.013	480- 610	5 at 12-14 days interval	0 5 10 15 21	0.15 0.10 0.06 0.10 <u>0.04</u>	45532
Trial R95-034 Spain, 1995 (Palomino Fino, Wine)	250 g/L SC	0.060- 073	0.010	570- 700	6 at 13-15 days interval	21	<u>0.02</u>	45513
Trial R95-035 Spain, 1995 (Macabeo, Wine)	200 g/L SC + 60 g/L fenarimol	0.070- 0.073	0.014	500- 520	7 at 9-15 days interval	20	0.15	45692
GAP, USA	250 g/L SC	0.12 0.036 at 7 day interval 0.072 at 14 day interval 0.12 at 21 day interval			5, Max 0.60 kg ai/ha/year at 7 –21 days interval	14		
Trial 99-NY06 New York, USA, 1999 (Chardonnay)	250 g/L SC	0.094- 0.118	0.006 –0.007	1600	5, at interval of 6-7 days; Total = 0.567 kg ai/season	14	<u>0.22</u>	83731
Trial 99-PA02 Pennsylvania, USA, 1999 (Cayuga)	250 g/L SC	0.118 – 0.123	0.012	1000	5, at interval of 6-7 days; Total = 0.60 kg ai/season	15	<u>0.13</u>	
Trial 99-CA38 California, USA, 1999 (Perlette)	250 g/L SC	0.22 0.22 0.12 0.12 0.12	0.009 –0.017	1300- 1400	5, at interval of 6-8 days; Total = 0.8 kg ai/season	14	<u>0.06</u>	
Trial 99-CA39 California, USA, 1999 (Thompson seedless)	250 g/L SC	0.118 – 0.121	0.009	1328- 1400	5, at interval of 6-8 days; Total = 0.598 kg ai/season	14	<u>0.09</u>	

Grapes country, year (variety)	Application[1] Form	kg ai/ha	kg ai/hL	water, L/ha	no.	PHI days	Residues mg/kg	Reference
Trial 99-CA68 California, USA, 1999 (Thompson seedless)	250 g/L SC	0.120 – 0.121	0.009	1300 - 1400	5, at interval of 6-7 days; Total = 0.612 kg ai/season	14	0.15	
Trial 99-CA69 California, USA, 1999 (Thompson seedless)	250 g/L SC	0.115 – 0.123	0.009	1300 - 1400	5, at interval of 6-7 days; Total = 0.595 kg ai/season	14	0.08	
Trial 99-CA70 California, USA, 1999 (Thompson seedless)	250 g/L SC	0.119 – 0.121	0.009	1300	5, at interval of 6-7 days; Total = 0.598 kg ai/season	14	0.18	
Trial 99-CA71 California, USA, 1999 (Thompson)	250 g/L SC	0.169 – 0.120	0.009	1300	5, at interval of 6-7 days; Total = 0.592 kg ai/season	14	0.24	
Trial 99-CA96 California, 1999 (Sauvignon Blanc)	250 g/L SC	0.23 0.12 0.12 0.12 0.12	0.010- 0.018	1200	5, at interval of 6-8 days; Total = 0.71 kg ai/season	14	0.15	
Trial 99-ID16 Idaho, USA, 1999 (Chardonnay)	250 g/L SC	0.118 – 0.123	0.010	1100 - 1200	5, at interval of 6-7 days; Total = 0.607 kg ai/season	13	0.08	
Trial 99-WA10 Washington, USA, 1999 (Concord)	250 g/L SC	0.120 – 0.122	0.009- 0.012	1000- 1300	5, at interval of 6-7 days; Total = 0.605 kg ai/season	15	0.15	
Trial 99-WA11 Washington, USA, 1999 (Concord)	250 g/L SC	0.120 – 0.121	0.009- 0.012	1000 - 1400	5, at interval of 6-7 days; Total = 0.604 kg ai/season	14	0.13	
Trial 99-FL43 Florida, USA, 1999 (Summit Muscadine)	250 g/L SC	0.123- 0.128	0.013	960- 990	5, at interval of 7-8 days; Total = 0.625 kg ai/season	14	0.44	
GAP, USA (applied to Canadian trials)	250 g/L SC	0.018-0.12 0.036 at 7 day interval 0.072 at 14 day interval 0.12 at 21 day interval			5, Max 0.60 kg ai/ha/year at 7 –21days interval	14		
Trial 99-ON06 Ontario, Canada, 1999 (Concord)	250 g/L SC	0.117- 0.121	0.013	880- 920	5 at intervals of 6-7 days; Total = 0.594 kg ai/season	14	0.22	83731
Trial 99-ON06 Ontario, Canada, 1999 (C0ncord)	250 g/L SC	0.058- 0.062	0.007	880-930	5 at intervals of 6-7 days; Total = 0.30	14	0.09	

Grapes country, year (variety)	Application[1] Form	kg ai/ha	kg ai/hL	water, L/ha	no.	PHI days	Residues mg/kg	Reference
Trial 99-ON07 Ontario, Canada, 1999 (SU-23512)	250 g/L SC	0.115-0.120	0.013	860- 910	5 at intervals of 6-7 days; Total = 0.594 kg ai/season	14	0.29	
Trial 99-ON07 Ontario, Canada, 1999 (SU-23512)	250 g/L SC	0.058-0.061	0.007	870- 920	5 at intervals of 6-7 days; Total = 0.297 kg ai/season	14	0.13	
GAP, Australia	250 g/L SC	0.05 (calculated)	0.0025 at 7-10 day interval;0.005 at 10-14 day interval	1000 (for dilute)	3	14		
Trial 98483-01 Australia, 2000 (Rhine Reisling)	250 g/L SC	0.075	0.0025	3000	3	14	0.15	1951
		0.15	0.005	3000	3	14	0.45	
		0.30	0.010	3000	3	14	0.99	
Trial 98483-02 Australia, 2000 (Chardonnay)	250 g/L SC		0.0025	-	3	14	0.23	
			0.005	-	3	14	0.82	
			0.010	-	3	14	1.32	
Trial 98483-03 Australia, 2000 (Black Shiraz)	250 g/L SC	0.025	0.0025	1000	3	14	< 0.01	
		0.05	0.005	1000	3	14	0.09	
		0.10	0.010	1000	3	14	< 0.01	
		0.05	0.005	1000	3	14	0.09	
		0.10	0.010	1000	3	14	< 0.01	
Trial 99050-01 Australia, 1999 (Chardonnay)	250 g/L SC	0.025	0.0025	1000	7	14	0.06	2148
		0.050	0.005	1000	7	14	0.18	
		0.10	0.010	1000	7	14	0.39	
Trial 99050-02 Australia, 1999 (Verdelho)	250 g/L SC	0.025	0.0025	1000	6	82	0.02	
		0.043	0.005	850	6	82	0.05	
		0.083	0.010	830	6	82	0.09	
Trial 99051-01 Australia, 2000 (Cabernet)	250 g/L SC	0.035	0.0025	1400	3	0	0.05	2149
						3	0.05	
						7	0.04	
						14	0.03	
						21	0.03	
						28	0.05	
		0.073	0.005	1500	3	0	0.22	
						3	0.27	
						7	0.17	
						14	0.15	
						21	0.10	
						28	0.11	
		0.15	0.010	1500	3	0	0.50	
						3	0.43	
						7	0.23	
						14	0.25	
						21	0.18	
						28	0.19	

Grapes country, year (variety)	Application[1] Form	kg ai/ha	kg ai/hL	water, L/ha	no.	PHI days	Residues mg/kg	Reference
Trial 99051-03 Australia, 2000 (Chardonnay)	250 g/L SC	0.065	0.0025	2600	3	0	0.19	2149
						3	0.21	
						7	< 0.01	
						14	0.17	
						21	0.08	
						28		
		0.13	0.005	2600	3	0	0.60	
						3	0.51	
						7	0.39	
						14	0.54	
						21	0.30	
						28	0.22	
		0.26	0.010	2600	3	1	1.2	
						3	1.0	
						7	0.70	
						14	0.65	
						21	0.50	
						28	0.53	
Trial 99051-04 Australia, 2000 (Thompson seedless)	250 g/L SC	0.17	0.0025	6700	3	1	0.51	2149
						3	0.83	
						7	0.80	
						14	0.41	
						21	0.60	
						28	0.40	
		0.33	0.005	67 00	3	1	1.5	
						3	1.6	
						5	1.7	
						14	1.1	
						21	1.0	
						28	1.0	
		0.67	0.010	6700	3	1	3.2	
						3	4.3	
						7	3.3	
						14	3.2	
						21	3.1	
						28	2.6	

Note: For US trials, residue value is the average of duplicate samples.

A total of eleven supervised trials were conducted on strawberries in Germany from 1999 to 2000 (11110, Kunz, 2001). Eight supervised trials were conducted in strawberry growing areas in the USA (208697, Barney, W., 2005).

Table 52. Quinoxyfen residues in strawberries from foliar applications in Germany and the USA.

Strawberry country, year (variety)	Application[1] Form	kg ai/ha	kg ai/hL	water, L/ha	no.	PHI days	Residues mg/kg	Reference
GAP, Germany	250 g/L SC	0.12	0.006	2000	2	14		
Trial OR901 (Field) Germany, 1999 (Polka)	500 g/L SC	0.12	0.013	1000	2	21	0.01	11110
Trial B/FO 27/99 (Glasshouse) Germany, 1999 (Polka)	500 g/L SC	0.12	0.013	1000	2	0	0.08	11110
						7	0.04	
						14	0.02	
						21	0.01	

Strawberry country, year (variety)	Application[1]					PHI days	Residues mg/kg	Reference
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Trial RU-F-53 00 RPMZ 1/1 (Plastic tunnel) Germany, 1999 (Elsanta)	250 g/L SC	0.12	0.013	1000	2	0 7 14 21	0.37 0.01 <u>0.05</u> 0.01	11110
Trial RU-L-39 99 MZ (Plastic tunnel) Germany, 1999 (Honeoye Frigo)	500 g/L SC	0.12	0.013	1000	2	7 14 21	0.26 <u>0.07</u> 0.01	11110
Trial PSD-O-19/99 (Glasshouse) Germany, 1999 (Elsanta)	500 g/L SC	0.12	0.013	1000	2	21	0.04	11110
Trial 11312 (Plastic tunnel) Germany, 2000 (Elsanta)	250 g/L SC	0.12	0.013	1000	2	14 21	<u>0.16</u> 0.07	11110
Trial 11312 (plastic tunnel) Germany, 2000 (Elsanta)	500 g/L SC	0.12	0.013	1000	2	14 21	<u>0.09</u> 0.07	11110
Trial PSD-O-26/00 (Glasshouse) Germany, 2000 (Elsanta)	250 g/L SC	0.12	0.013	1000	2	14 21	<u>0.12</u> 0.02	11110
Trial B/FO 36/00 (Glasshouse) Germany, 2000 (Avalon grün)	250 g/L SC	0.12	0.013	1000	2	0 7 14	0.24 0.12 <u>0.04</u>	11110
Trial OR0001 (Field) Germany, 2000 (Elsanta)	250 g/L SC	0.12	0.013	1000	2	21	< 0.01	11110
Trial 00/022 (Field) Germany, 2000 (Elsanta)	250 g/L SC	0.12	0.013	1000	2	14	<u>0.01</u>	11110
GAP, USA	250 g/L SC	0.11		280	4, max 0.44 /kg ai/season	1		
Trial 02-WI12 Wisconsin, USA, 2002 (Honoeye)	250 g/L SC	0.14-0.15		400 – 440	4, max 0.58 kg ai/ ha/season	1	<u>0.16</u>	208697
Trial 02-NJ25 New Jersey USA, 2002 (Chandler)	250 g/L SC	0.14 - 0.15		500 - 520	4, max 0.58 kg ai/ ha/season	1	<u>0.18</u>	208697
Trial 02-NC16 North Carolina, USA, 2002 (Camarosa)	250 g/L SC	0.14 – 0.15		320 – 330	4, max 0.58 kg ai/ ha/season	1 3 6	<u>0.41</u> 0.25 0.18	208697
Trial 02-FL43, Florida, USA 2002 (Sweet Charlie)	250 g/L SC	0.14 – 0.15		370 - 380	4, max 0.58 kg ai/ ha/ season	1	<u>0.56</u>	208697
Trial 02-WA29 Washington, USA, 2002 (Totem)	250 g/L SC	0.15 - 0.17		390 - 660	4, max 0.65 kg ai/ ha/ season	1	0.05	208697

Strawberry country, year (variety)	Application[1]					PHI days	Residues mg/kg	Reference
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Trial 02-CA97 California, USA, 2002 (Hecker)	250 g/L SC	0.15		460 - 470	4, max 0.58 kg ai/ ha/ season	1 3 7	0.46 0.33 0.16	208697
Trial 02-CA98 California, USA, 2002 (Hecker)	250 g/L SC	0.14 - 0.15		460 - 480	4, max 0.58 kg ai/ ha/ season	1	0.24	208697
Trial 02-CA99 California, USA, 2002 (Hecker)	250 g/L SC	0.15		470 – 480	4, max 0.60 kg ai/ ha/ season	1	0.53	208697

Supervised trials were conducted on black currants in Germany (20062, Fuchsbichler, 2002; 20061, Fuchsbichler, 2002). The trials were conducted by a government agency in Germany to support the establishment of MRLs in the EU.

Table 53. Quinoxyfen residues in currants from foliar application in Germany (20061; 20062)[1].

Currants (black) Trial No., country, year (variety)	Application					PHI days	Residues[2] mg/kg
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.		
GAP, Germany	250 g/L SC	0.075	0.0075	1000	3	14	
RIP2003-487 (148) Germany, 2001 (Ben Navis)	250 g/L SC	0.075	0.0075	1000	3	14	0.20
RIP2003-490 (244) Germany, 2002 (Tenah)	250 g/L SC	0.075	0.0094	800	3	0 7 10 14 21	0.30 0.11 0.08 0.06 0.03
RIP2003-492 (246) Germany, 2002 (Titania)	250 g/L SC	0.075	0.017	429	3	14	0.05
RIP2003-449 (147) Germany, 2002 (Titania)	250 g/L SC	0.075	0.01	750	3	0 7 10 14 21	0.25 0.09 0.04 0.04 0.02
RIP2003-483 (146) Germany, 2002 (Blackdown)	250 g/L SC	0.075	0.0075	1000	3	0 7 10 14 21	0.63 0.43 0.39 0.40 0.34
RIP2003-489 (243) Germaany, 2002 (Titania)	250 g/L SC	0.075	0.0075	1000	3	0 7 10 14 21	0.60 0.47 0.44 0.30 0.11
RIP2003-491 (245) Germany, 2002 (Ben Tirran)	250 g/L SC	0.075	0.0075	1000	3	14	0.28

[1]Only the report on the analytical phase of the study was available to the Meeting.

[2]All control samples were <LOQ (0.01 or 0.02 mg/kg).

Fruiting vegetables, Cucurbits

Sixteen supervised trials on melons were conducted in Spain, Italy, and Greece during 1995 and 1996 (46957; 46958; 46956; 46954, Khoshab, 1995; 48081; 48080; 47141, Khoshab, 1996). Quinoxyfen was determined in the peel and the pulp. Residues in the whole fruit were calculated using percentage weight composition of melons from a standard text (The Composition of Foods by Paul, A. and Southgate, D), i.e., 59% pulp and 41% peel.

Table 54. Quinoxyfen residues on melons from supervised trials in Italy, Greece and Spain.

Melons	Application			PHI	Residues, mg/kg			
country, year (variety)	Form	kg ai/hL	no.	days	Whole fruit	Peel	Pulp	Reference
GAP, Italy	250 g/L OR 200 g/L + 60 g/L fenarimol	0.004- 0.006		7				
Trial R95-025A Italy, 1995 (0328 Numens)	200 g/L + 60 g/L fenarimol	0.0075	3	7	0.03	0.07	< 0.01	46957
Trial R95-025B Italy, 1995 (0328 Numens)	200 g/L 60 g/L fenarimol	0.0070	3	7	0.02	0.06	< 0.01	
Trial R96-041A Italy, 1996 (vector)	250 g/L	0.0075	3	8	0.03	0.04	0.02	48081
Trial R96-041B Italy, 1996 (vector)	200 g/L + 60 g/L fenarimol	0.0075	3	8	0.02	0.03	0.02	
Trial R96-043A Italy, 1996 (Supermarket)	250 g/L	0.0055	3	7	0.02	0.02	< 0.01	48080
Trial R96-043B Italy, 1996 (Supermarket)	200 g/L + 60 g/L fenarimol	0.0050	3	7	0.01	0.01	< 0.01	
Trial R96-097A Greece, 1996 (Yuppie Hylond)	250 g/L	0.0075	3	7	0.02	0.06	< 0.01	47141
Trial R96-097B Greece, 1996 (Yuppie Hylond)	200 g/L + 60 g/L fenarimol	0.0070	3	7	0.01	0.04	< 0.01	
GAP, Spain	200 g/L + 60 g/L fenarimol	0.005- 0.0075	3	7				
Trial R95-030A Spain, 1995 (Galia-Revigal)	250 g/L	0.0075	3	0 4 7	0.05 0.01 0.02	0.08 0.02 0.06	< 0.01 < 0.01 < 0.01	46958
Trial R95-030A Spain, 1995 (Galia-Revigal)	200 g/L + 60 g/L fenarimol	0.0070	3	7	0.02	0.04	ND	
Trial R95-030B Spain, 1995 (Doral)	250 g/L	0.0074	3	0 4 7	0.03 0.01 0.01	0.07 0.04 0.02	< 0.01 ND ND	

Melons	Application			PHI	Residues, mg/kg			
country, year (variety)	Form	kg ai/hL	no.	days	Whole fruit	Peel	Pulp	Reference
Trial R95-030B Spain, 1995 (Doral)	200 g/L + 60 g/L fenarimol	0.0070	3	0 4 7	0.02 0.02 0.01	0.06 0.06 0.03	ND ND ND	
Trial R95-031A Spain, 1995 (Cantaloupe)	250 g/L	0.0077	3	0 4 7	0.02 0.02 0.01	0.06 0.02 0.02	ND < 0.01 ND	46956
Trial R95-031B Spain, 1995 (Cantaloupe)	200 g/L + 60 g/L fenarimol	0.0072	3	0 4 7	0.02 < 0.01 < 0.01	0.04 0.01 < 0.01	ND ND ND	46956
Trial R96-045A Spain, 1996 (Regal)	250 g/L	0.0075	3	7	0.02	0.04	< 0.01	46954
Trial R96-045B Spain, 1996 (Regal)	200 g/L + 60 g/L fenarimol	0.0070	3	7	0.02	0.04	< 0.01	

ND = 0.002 mg/kg (20% of LOQ)

A total of 11 supervised field trials on cantaloupes were conducted in major growing areas of the USA and Canada in 2002 and 2003 (208696, Corley, 2004).

Table 55. Summary of quinoxyfen residue data on melons from treatments according to the GAP in the US (208696).

Melons	Application				PHI days	Residues[1] mg/kg
country, year (variety)	Form	kg ai/ha	water, L/ha	no.		
GAP, USA	250 g/L SC	0.073-0.11	280	4	3	
Trial 01-BC01 BC, Canada, 2001 (Athena)	250 g/L SC	0.14 – 0.15	540 - 560	4, total 0.58 kg ai/ha	4	0.03
Trial 01-ON01 ON, Canada, 2001 (Athena)	250 g/L SC	0.11 – 0.17	520 - 760	4, total 0.57 kg ai/ha	2	0.05
Trial 01-QC03 Quebec, Canada 2001 (Early Dawn)	250 g/L SC	014 – 0.15	470 - 490	4, total 0.58 kg ai/ha	4	0.03
Trial 01-CA60 CA, USA, 2001 (Aclaim)	250 g/L SC	0.15	370 - 480	4, total 0.58 kg ai/ ha	3	0.03
Trial 01-CA82 CA, USA, 2001 (Hale's Best)	250 g/L SC	0.15 – 0.16	470 - 480	4, total 0.62 kg ai/ ha	3	0.04
Trial 01-CA83 CA, USA, 2001 (Hearts of Gold)	250 g/L SC	0.15 – 0.16	480 - 490	4, total 0.62 kg ai/ ha	2	0.02
Trial 01-NM09 NM, USA, 2001 (Hale's Best Jumbo)	250 g/L SC	0.15	340 - 370	4, total 0.58 kg ai/ ha	3	0.02
Trial 01-NJ20 NJ, USA, 2001 (Ambrosia)	250 g/L SC	0.15	380 - 390	4, total 0.60 kg ai/ ha	0 3 7 14	0.04 0.03 0.03 0.02
Trial 01-TX323 TX, USA, 2001 (Explorer)	250 g/L SC	0.15 - 0.16	320 - 340	4, total 0.61 kg ai/ ha	0 3 7 14	0.07 0.02 0.02 < 0.01

Melons country, year (variety)	Application				PHI days	Residues[1] mg/kg
	Form	kg ai/ha	water, L/ha	no.		
Trial 01-TX324 TX, USA, 2001 (Mission)	250 g/L SC	0.15	290 - 300	4, total 0.60kg ai/ ha	2	0.05
Trial 01-GA16 GA, USA, 2001 (Vienna)	250 g/L SC	0.15	280 - 290	5, total 0.75 kg ai/ ha	2	< 0.01

[1] Average of duplicate samples from the same plot.

Fruiting vegetables, other then Cucurbits

Peppers, sweet

A total of eleven supervised trials were conducted on peppers (six on bell peppers and five on non-bell peppers) in commercial growing areas in the United States during 2001 (8006, Chen, 2003). Samples of marketable pepper were collected by hand from each plot, placed in plastic bags and shipped to the laboratory, where they were kept frozen at about -20ºC until analysis (255 days maximum).

Table 56. Quinoxyfen residues in sweet peppers from supervised trials in the USA (8006).

PEPPERS country, year (variety)	Application					PHI days	Residues[1] mg/kg
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.		
GAP, USA	250 g/L SC	0.15	0.020	750	4, max 0.60 kg ai/ha/yr	3	
Trial 01-NJ19 NJ, USA, 2001 (Bell- King Arthur)	250 g/L SC	0.14		370 - 390	4, total 0.58 kg ai/ha/yr	2	0.15
Trial 01-FL27 FL, USA, 2001 (Bell- Camelot)	250 g/L SC	0.14		320 – 340	4, total 0.58 kg ai/ha/yr	0 3 7 14	0.41 0.16 0.06 0.02
Trial 01-FL28 FL, USA, 2001 (Bell- Camelot)	250 g/L SC	0.14		320 - 340	4, total 0.58 kg ai/ha/yr	3	0.15
Trial 01-TX20 TX, USA, 2001 (Bell- Jupiter)	250 g/L SC	0.14		360 - 380	4, total 0.58 kg ai/ha/yr	2	0.17
Trial 01-CA58 CA, USA, 2001 (Bell - Jupiter)	250 g/L SC	0.14		460 - 480	4, total 0.58 kg ai/ha/yr	0 3 7 14	0.03 0.01 0.01 0.01
Trial 01-CA59 CA, USA, 2001 (Bell- Jupiter)	250 g/L SC	0.14		480– 490	4, total 0.58 kg ai/ha/yr	3	0.02
Trial 01-OH15 OH, USA, 2001 (Non-bell)	250 g/L SC	0.14		470- 550	4, total 0.58 kg ai/ha/yr	2	0.09
Trial 01-NM07 NM, USA, 2001 (Non-bell- Big Jim)	250 g/L SC	0.14		320 - 370	4, total 0.58 kg ai/ha/yr	4	0.12

PEPPERS country, year (variety)	Application					PHI days	Residues[1] mg/kg
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.		
Trial 01-FL29 FL, USA, 2001 (Non-bell -Cheyane)	250 g/L SC	0.14		320 - 330	4, total 0.58 kg ai/ha/yr	3	0.64
Trial 01-TX21 TX, USA, 2001 (Non-bell- Sonora Anaheim)	250 g/L SC	0.14		420 – 590	4, total 0.58 kg ai/ha/yr	2	0.23
Trial 01-GA14 GA, USA, 2001 (Non-bell - Jalapeno)	250 g/L SC	0.14		470 - 480	4, total 0.58 kg ai/ha/yr	5	0.52

[1] Average of duplicate samples in the same plot.

Leafy vegetables

Lettuce

A total of sixteen supervised trials were conducted on lettuce (eight on leaf lettuce and eight on head lettuce) in commercial growing areas in the United States during 2002 (208698, Barney, 2005). A minimum of 4 pounds of head lettuce (with wrapper leaves) or leaf lettuce (with wrapper leaves) was collected per sample. At all of the trials, lettuce samples were harvested randomly across the plots avoiding the ends of the plots. Two samples were collected from each treated plot and one from each untreated plot. Lettuce samples were placed in labelled sample bags and held in frozen storage until analysis (19 to 187 days).

Table 57. Quinoxyfen residues on lettuce with wrapper leaves from supervised trials in the USA (208698).

Lettuce (head and leaf) country, year (variety)	Application					PHI days	Residues[1] mg/kg
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.		
GAP, USA	250 g/L SC	0.11		280	4 , max of 0.44 kg ai/ha/season	1	
Trial 02-CO09 CO, USA, 2002 (Head -Raider)	250 g/L SC	0.14 – 0.15		360 - 400	4, total 0.58 kg ai/ ha/yr	1	1.4
Trial 02-FL40 FL, USA, 2002 (Head - Salinas)	250 g/L SC	0.15		330	4, total 0.58 kg ai/ ha/yr	1	5.3
Trial 02-NC15 NC, USA, 2002 (Head - Maverick)	250 g/L SC	0.14 – 0.15		321	4, total 0.58 kg ai/ ha/yr	1	1.2
Trial 02-CA89 CA, USA, 2002 (Head -Sharp Shooter)	250 g/L SC	0.15 – 0.16		550 - 740	5, total 0.74 kg ai/ ha/yr	1	0.86
Trial 02-CA91 CA, USA, 2002 (Head - Titan)	250 g/L SC	0.14 - 0.15		590 - 650	4, total 0.58 kg ai/ ha/yr	1	1.0

Lettuce (head and leaf) country, year (variety)	Application					PHI days	Residues[1] mg/kg
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.		
Trial 02-CA93 CA, USA, 2002 (Head – Sharp Shooter)	250 g/L SC	0.14 – 0.15		720 - 750	4, total 0.59 kg ai/ ha/yr	1	0.91
Trial 02-CA94 CA, USA, 2003 (Head – Wellton)	250 g/L SC	0.14		380 - 400	4, total 0.58 kg ai/ ha/yr	1	3.1
Trial 02-CA87 CA, USA, 2002 (Head – Empire)	250 g/L SC	0.14 – 0.15		370 - 380	4, total 0.58 kg ai/ ha/yr	1	2.1
Trial 02-CO10 CO, USA, 2002 (Leaf – Capistrano)	250 g/L SC	0.14 – 0.17		360 - 440	4, total 0.62 kg ai/ ha/yr	1	1.8
Trial 02-MD09 MD, USA, 2002 (Leaf – Grand Rapids)	250 g/L SC	0.14 – 0.15		400 - 410	4, total 0.58 kg ai/ ha/yr	1	2.9
Trial 02-CA90 CA, USA, 2002 (Leaf – Green Gene's #1)	250 g/L SC	0.15		450 - 620	4, total 0.74 kg ai/ ha/yr	1 4 7 14	2.2 1.4 0.76 0.29
Trial 02-CA92 CA, USA, 2002 (Leaf – Panther)	250 g/L SC	0.14 – 0.15		660 - 760	4, total 0.58 kg ai/ ha/yr	1	1.3
Trial 02-NM11 NM, USA, 2002 (Leaf – Salad Bowl)	250 g/L SC	0.14		380 - 510	4, total 0.57 kg ai/ ha/yr	1	3.4
Trial 02-CA95 CA, USA, 2002 (Leaf – Marin)	250 g/L SC	0.14 – 0.15		400 - 470	4, total 0.58 kg ai/ ha/yr	1	13
Trial 02-CA88 CA, USA, 2002 (Leaf – Waldemans Green)	250 g/L SC	0.15		370 - 380	4, total 0.58 kg ai/ ha/yr	1	4.3
Trial 02-FL41 FL, USA, 2003 (Leaf – Waldemans Dark Green MTO)	250 g/L SC	0.15		330	4, total 0.58 kg ai/ ha/yr	1 3 7 14	6.9 6.1 5.2 1.8

[1] Average of results from duplicate samples taken for each plot.

Root and Tuber vegetables

Sugar beet roots

A total of eight supervised trials were conducted on sugar beets in Northern Europe (Germany, Northern France and the UK) 76690, (Jones, 2000; 83187, Kang, 2001,). The first application was made at growth stage BBCH 16–33 and the second at growth stage BBCH 45–49. Samples were immediately frozen and kept in frozen storage at about -18ºC up to 4 months (1999 trials) or 179 days (2000 trials), until analysis.

Table 58. Quinoxyfen residues in sugar beet roots from supervised trials in Germany, UK, and France.

Sugar Beets	Application					PHI	Residues mg/kg	Reference
country, year (variety)	Form	kg ai/ha	kg ai/hL	water, L/ha	no.	days		
GAP, Germany	500 g/L SC	0.12	0.062		2	28		
Trial R99-021C Germany, 1999 (Aries)	500 g/L	0.15 0.15	0.075	200	2	28	< 0.01	76690
Trial CEMS-1348C Germany, 2000 (Beta vulgaris)	500 g/L	0.15 0.15	0.075	200	2	28	0.01	83187
Trial CEMS-1348D Germany, 2000 (Beta vulgaris)	500 g/L	0.16 0.15	0.075	210	2	28	0.01	83187
GAP, UK	500 g/L SC	0.15	0.075	200	2 0.2 kg ai/ha/yr	28		
Trial R99-021A UK, 1999 (Madison)	500 g/L	0.15 0.15	0.075	200	2	28	< 0.01	76690
Trial R99-021B UK, 1999 (Madison)	500 g/L	0.15 0.15	0.075	200	2	28	< 0.01	76690
Trial CEMS-1348A, UK, 2000 (BEA VA/ Chorus)	500 g/L	0.14 0.16	0.075	180-210	2	28	0.02	83187
Trial CEMS-1348B, UK, 2000 (BEA VA/ Jackpot)	500 g/L	0.15 0.14	0.075	190-210	2	28	0.01	83187
GAP, France	500 g/L	0.15	0.10		1	28		
Trial R99-021D France (northern), 1999 (Access)	500 g/L	0.15	0.075	200	2	28	< 0.01	76690

Cereal grains

Wheat

A total of 38 supervised trials were conducted on wheat during 1993 and 1994 (10 in Germany (31265, 31266, 31633, 31262, Gambie, 1995), eight in the UK (24035, 63697, Gambie and Nicholson, 1994; 31080, Gambie, 1995; 29405, Gambie, Nicholson, and Wood, 1995; 31268, Gambie and Wood, 1995), six in northern France (31620, 29912, 31241, Gambie, 1995), 10 in southern France (29406, 31644, 31234, Gambie, 1995), and four in Greece (31231, Gambie, 1995). The trials received either one application of a suspension concentrate formulation containing 500 g/L quinoxyfen at the rate of 0.25 kg ai/ha or two applications, one at 0.25 kg ai/ha, the second at 0.15 kg ai/ha. The applications were made around the stem elongation growth stages (BBCH 31-33) with a second one at around the booting stage when first awns are visible (BBCH 47–49). Samples of grain and straw were collected at harvest. Whole plants were cut above ground level by hand with subsequent threshing in a combine harvester. Samples were stored frozen at -18ºC until analysis, 9 to 15 months later.

Table 59. Quinoxyfen residues on wheat grain from foliar applications in France, Greece, Germany, and UK

Wheat grain	Application				PHI	Residues, mg/kg	Reference
country, year (variety)	Form	kg ai/ha	Water L/ha	no.	days	Grain[1]	
GAP, France	500 g/L SC	0.15	130-300	1	56		
Trial R93-28A S. France, 1993 (Durum, Lloyd)	500 g/L SC	0.25	230	1	79	< 0.01	29406
Trial R93-28A S. France, 1993 (Durum, Llyod)	500 g/L SC	0.15 +0.25	230-460	2	64	< 0.01	
Trial R93-28B S. France, 1993 (Winter, Soissons)	500 g/L SC	0.25	180	1	67	ND	
Trial R93-28B S. France, 1993 (Winter, Soissons	500 g/L SC	0.15 + 0.25	180	2	55	ND	
Trial R94-018 S. France, 1994 (Winter, Fortal)	500 g/L SC	0.25	300	1	99	ND	31644
Trial R94-018 S. France, 1994 (Winter, Fortal)	500 g/L SC	0.15 + 0.25	300	2	66	ND	31644
Trial R94-019A S. France, 1994 (Durum, Neodor)	500 g/L SC	0.25	200	1	87	ND	31234
Trial R94-019A S. France, 1994 (Durum, Neodor)	500 g/L SC	0.15 + 0.25	200	2	67	ND	
Trial R94-020A S. France, 1994 (Winter, Soisson)	500 g/L SC	0.25	316	1	83	ND	31234
Trial R94-020A S. France, 1994 (Winter, Soisson)	500 g/L SC	0.15 + 0.25	308	2	27	ND	
Trial R93-29A N. France, 1993 (Winter, Sidereal)	500 g/L SC	0.25	333	1	95	ND	29912
Trial R93-29A N. France, 1993 (Winter, Sidereal)	500 g/L SC	0.15 + 0.25	333	2	75	< 0.01	
Trial R93-30B N. France, 1993 (Winter, Arche)	500 g/L SC	0.25	200	1	115	ND	31620
Trial R93-30B N. France, 1993 (Winter, Arche)	500 g/L SC	0.15 + 0.25	200	2	87	ND	

Wheat grain	Application				PHI	Residues, mg/kg	Reference
country, year (variety)	Form	kg ai/ha	Water L/ha	no.	days	Grain[1]	
Trial R94-023A N. France, 1994 (Spring, Furio)	500 g/L SC	0.25	200	1	65	ND	31241
Trial R94-023A N. France, 1994 (Spring, Furio)	500 g/L SC	0.15 + 0.25	200	2	50	ND	
GAP, France applied to trials in Greece	500 g/L SC	0.15	130-300	1	56		
Trial R93-36A Greece, 1993 (Hard wheat, Mexicali)	500 g/L SC	0.25	200	1	82	ND	31231
Trial R93-36A Greece, 1993 (Hard wheat, Mexicali)	500 g/L SC	0.15 + 0.25	200	2	60	0.02	
Trial R93-36B Greece, 1993 (Soft wheat, Yecora)	500 g/L SC	0.25	200	1	58	0.02	
Trial R93-36B Greece, 1993 (Soft wheat, Yecora)		0.15 + 0.25	200	2	48	0.09	
GAP, Germany	500 g/L SC	0.15	200-400	2, 0.25 kg ai/ha/yr	49		
	500 g/L SC	0.25	-	1	BBCH 25 – 32		
Trial RF93-31S Germany, 1993 (Winter, Ares)	500 g/L SC	0.25	400	1	84	ND	31266
Trial RF93-31S Germany, 1993 (Winter, Ares)	500 g/L SC	0.15 + 0.25	400	2	70	ND	
Trial RF93-31C Germany, 1993 (Winter, Astron)	500 g/L SC	0.25	400	1	100	ND	31266
Trial RF93-31C Germany, 1993 (Winter, Astron)	500 g/L SC	0.15 + 0.25	400	2	78	< 0.01	
Trial RF93-32C Germany, 1993 (Winter, Astron)	500 g/L SC	0.25	400	1	86	ND	31265
Trial RF93-32C Germany, 1993 (Winter, Astron)	500 g/L SC	0.15 + 0.25	400	2	73	ND	

Wheat grain	Application				PHI	Residues, mg/kg	Reference
country, year (variety)	Form	kg ai/ha	Water L/ha	no.	days	Grain[1]	
Trial RF94-038A Germany, 1994 (Winter, Kanzler)	500 g/L SC	0.25	400	1	85	ND	31262
Trial RF94-038A Germany, 1994 (Winter, Kanzler)	500 g/L SC	0.18 + 0.25	437	2	50	ND	
Trial R94-041A Germany, 1994 (Winter, Kanzler)	500 g/L SC	0.25	400	1	84	ND	31633)
Trial R94-041A Germany, 1994 (Winter, Kanzler)	500 g/L SC	0.15 + 0.25	400	2	49	ND	
GAP, UK	250 g/L or 500 g/L SC	0.15	200-400	2	~60 Zadoks 49		
Trial R93-34A UK, 1993 (Winter, Mercia)	500 g/L SC	0.25	200	1	113	ND	24035
Trial R93-34A UK, 1993 (Winter, Mercia)	500 g/L SC	0.15 + 0.25	200	2	91	ND	
Trial R93-35A UK, 1993 (Winter, Brock)	500 g/L SC	0.15 + 0.25	200	2	62	ND	24035
Trial R93-35A UK, 1993 (Winter, Clarine)	500 g/L SC	0.15 + 0.25	200	2	69	0.05	
Trial R93-33A UK, 1993 (Winter, Apollo)	500 g/L SC	0.25	200	1	113	ND	29405
Trial R93-33A UK, 1993 (Winter, Apollo)	500 g/L SC	0.15 + 0.25	200	2	70	< 0.01	
Trial R94-002A UK, 1994 (Winter, Spark)	500 g/L SC	0.25	200	1	104	ND	31080
Trial R94-002A UK, 1994 (Winter, Spark)	500 g/L SC	0.15 + 0.25	200	2	71	< 0.01	31080

[1] ND = 0.002 mg/kg for wheat grain (< 20% of LOQ).

Barley

A total of 22 supervised trials were conducted on barley during 1993 and 1994, eight in Germany (31263, 31261, 31648, 31635, Gambie, 1995); seven in the UK (24035, Gambie and Nicholson, 1994; 31267, Gambie and Wood, 1995; 31235, Gambie, 1995), five in northern France (31620, 29912, 31646, Gambie, 1995) and two in southern France in 1998 (69430, Khoshab and Clements, 1999). The applications were made around the stem elongation growth stages (BBCH 31-33) with a second one at around the booting stage when first awns are visible (BBCH 45-49). Samples of grain and

straw were collected at harvest. Whole plants were cut above ground level by hand with subsequent threshing in a combine harvester. Samples were stored frozen at -18ºC until analysis, 8 to 18 months later.

Residues of quinoxyfen were determined by gas chromatography with mass selective detection, following method ERC 94.5.

Table 60. Quinoxyfen residues in barley grain from supervised trials in Germany, UK, and France.

BARLEY	Application				PHI	Residues, mg/kg	Reference
country, year (variety)	Form	kg ai/ha	Water L/ha	no.	days	Grain[1]	
GAP, France	500 g/L SC	0.15	130-300	1	56		
Trial R98-002A S. France, 1998 (Winter, Majestic)	500 g/L SC	0.15 + 0.16	253	2	65	0.04	69430
Trial R98-002A S. France, 1998 (Winter, Nevada)	500 g/L SC	0.15 + 0.16	246	2	55	< 0.01	
Trial R93-30A N. France, 1993 (Winter, Energy)	500 g/L SC	0.25	200	1	75	0.01	31620
Trial R93-30A N. France, 1993 (Winter, Energy)	500 g/L SC	0.15 + 0.25	200	2	55	0.11	
Trial R93-29B N. France, 1993 (Winter, Plaisant)	500 g/L SC	0.25	300	1	58	< 0.01	29912
Trial R93-29B N. France, 1993 (Winter, Plaisant)	500 g/L SC	0.15 + 0.25	300	2	47	0.12[2]	
Trial R94-021 N. France, 1994 (Spring, Alexis)	500 g/L SC	0.25	260	1	65	ND	31646
GAP, Germany	500 g/L SC	0.1 - 0.15	200-400	2	49		
	500 g/L SC	0.25	-	1	BBCH 25 – 32		
Trial RF93-114A Germany, 1993 (Winter, Igri)	500 g/L SC	0.25	400	1	66	0.02	31263
Trial RF93-114A Germany, 1993 (Winter, Igri)	500 g/L SC	0.15 + 0.25	400	2	58	0.04	
Trial RF93-115S Germany, 1993 (Winter, Iastrid)	500 g/L SC	0.25	400	1	75	ND	31261
Trial RF93-115S Germany, 1993 (Winter, Iastrid)	500 g/L SC	0.15 + 0.25	400	2	65	ND	
Trial RF94-039A Germany, 1994 (Winter, Grete)	500 g/L SC	0.25	400	1	66	< 0.01	31635

Trial RF94-039A Germany, 1994 (Winter, Grete)	500 g/L SC	0.15 + 0.25	400	2	54	0.05	
Trial RF94-040A Germany, 1994 (Winter, Jana)	500 g/L SC	0.25	400	1	72	< 0.01	31648
Trial RF94-040A Germany, 1994 (Winter, Jana)	500 g/L SC	0.15 + 0.25	400	2	55	0.01	
GAP, UK	250 g/L OR 500 g/L	0.15	200-400	1-2	~60 Zadoks 49		
Trial R93-34A UK, 1993 (Winter, Pasoral)	500 g/L SC	0.25	200	1	96	ND	24035
Trial R93-34A UK, 1993 (Winter, Pasoral)	500 g/L SC	0.15 + 0.25	200	2	82	ND	
Trial R93-85A UK, 1993 (Spring, Alexis)	500 g/L SC	0.25	200	1	76	0.02	31267
Trial R93-85A UK, 1993 (Spring, Alexis)	500 g/L SC	0.15 + 0.25	200	2	64	0.15	
Trial R94-003A UK, 1994 (Winter, Halcyon)	500 g/L SC	0.25	200	1	91	ND	31235
Trial R94-003A UK, 1994 (Winter, Halcyon)	500 g/L SC	0.15 + 0.25	200	2	70	< 0.01	

[1] ND = 0.002 mg/kg for barley grain (< 20% of LOQ).

[2] Trials not included in estimation of MRL due to very late application at growth stage BBCH 58.

Straw, fodder and forage of cereal grains and grasses (straws and fodders dry)

Wheat

Table 61. Quinoxyfen residues on wheat straw from foliar applications in France, Germany, Greece, and UK.

Wheat Straw	Application[1]				PHI	Residues, mg/kg	Reference
country, year (variety)	Form	kg ai/ha	Water L/ha	no.	days	Straw[1]	
GAP, France (South)	500 g/L SC	0.15	130-300	1	56		
Trial R93-28A S. France, 1993 (Durum, Lloyd)	500 g/L SC	0.25	230	1	79	0.10	29406
Trial R93-28A S. France, 1993 (Durum, Llyod)	500 g/L SC	0.15 + 0.25	230-460	2	64	0.24	
Trial R93-28B S. France, 1993 (Winter, Soissons)	500 g/L SC	0.25	180	1	67	0.33	
Trial R93-28B S. France, 1993 (Winter, Soissons	500 g/L SC	0.15 + 0.25	180	2	55	0.32	

Wheat Straw	Application[1]				PHI	Residues, mg/kg	Reference
country, year (variety)	Form	kg ai/ha	Water L/ha	no.	days	Straw[1]	
Trial R94-018 S. France, 1994 (Winter, Fortal)	500 g/L SC	0.25	300	1	99	< 0.05	31644
Trial R94-018 S. France, 1994 (Winter, Fortal)	500 g/L SC	0.15 + 0.25	300	2	66	0.13	31644
Trial R94-019A S. France, 1994 (Durum, Neodor)	500 g/LSC	0.25	200	1	87	0.07	31234
Trial R94-019A S. France, 1994 (Durum, Neodor)	500 g/L SC	0.15 + 0.25	200	2	67	0.17	
Trial R94-020A S. France, 1994 (Winter, Soisson)	500 g/L SC	0.25	320	1	83	< 0.05	31234
Trial R94-020A S. France, 1994 (Winter, Soisson)	500 g/L SC	0.15 + 0.25	310	2	27	0.06	
Trial R93-29A N. France, 1993 (Winter, Sidereal)	500 g/L SC	0.25	330	1	95	0.19	29912
Trial R93-29A N. France, 1993 (Winter, Sidereal)	500 g/L SC	0.15 + 0.25	330	2	75	0.23	
Trial R93-30B N. France, 1993 (Winter, Arche)	500 g/L SC	0.25	200	1	115	< 0.05	31620
Trial R93-30B N. France, 1993 (Winter, Arche)	500 g/L SC	0.15 + 0.25	200	2	87	0.13	31620
Trial R94-023A N. France, 1994 (Spring, Furio)	500 g/L SC	0.25	200	1	65	0.19	31241
Trial R94-023A N. France, 1994 (Spring, Furio)	500 g/L SC	0.15 + 0.25	200	2	50	0.58	
GAP, France (South) applied to trials in Greece	500 g/L SC	0.15	130-300	1	56		
Trial R93-36A Greece, 1993 (Hard wheat, Mexicali)	500 g/L SC	0.25	200	1	82	0.11	31231
Trial R93-36A Greece, 1993 (Hard wheat, Mexicali)	500 g/L SC	0.15 + 0.25	200	2	60	1.06	
Trial R93-36B Greece, 1993 (Soft wheat, Yecora)	500 g/L SC	0.25	200	1	58	2.99	

Wheat Straw	Application[1]				PHI	Residues, mg/kg	Reference
country, year (variety)	Form	kg ai/ha	Water L/ha	no.	days	Straw[1]	
Trial R93-36B Greece, 1993 (Soft wheat, Yecora)		0.15 + 0.25	200	2	48	7.22	
GAP, Germany	500 g/L SC	0.1 - 0.15	200-400	2	49		
	500 g/L EC	0.25	-	1	BBCH 25- 32		
Trial RF93-31S Germany, 1993 (Winter, Ares)	500 g/L SC	0.25	400	1	84	ND	31266
Trial RF93-31S Germany, 1993 (Winter, Ares)	500 g/L SC	0.15 + 0.25	400	2	70	< 0.05	
Trial RF93-31C Germany, 1993 (Winter, Astron)	500 g/L SC	0.25	400	1	100	0.07	31266
Trial RF93-31C Germany, 1993 (Winter, Astron)	500 g/L SC	0.15 + 0.25	400	2	78	0.11	
Trial RF93-32C Germany, 1993 (Winter, Astron)	500 g/L SC	0.25	400	1	86	0.13	31265
Trial RF93-32C Germany, 1993 (Winter, Astron)	500 g/L SC	0.15 + 0.25	400	2	73	0.36	
Trial RF94-038A Germany, 1994 (Winter, Kanzler)	500 g/L SC	0.25	400	1	85	< 0.05	31262
Trial RF94-038A Germany, 1994 (Winter, Kanzler)	500 g/L SC	0.18 + 0.25	440	2	50	0.26	
Trial R94-041A Germany, 1994 (Winter, Kanzler)	500 g/, SC	0.25	400	1	84	< 0.05	31633
Trial R94-041A Germany, 1994 (Winter, Kanzler)	500 g/, SC	0.15 + 0.25	400	2	49	0.12	
GAP, UK	250 g/, or 500 g/L SC	0.15	200-400	1-2	~60 Zadoks 49		
Trial R93-34A UK, 1993 (Winter, Mercia)	500 g/, SC	0.25	200	1	113	0.19	24035
Trial R93-34A UK, 1993 (Winter, Mercia)	500 g/, SC	0.15 + 0.25	200	2	91	0.27	
Trial R93-35A UK, 1993 (Winter, Brock)	500 g/, SC	0.15 + 0.25	200	2	62	0.37	31268

Wheat Straw	Application[1]				PHI	Residues, mg/kg	Reference
country, year (variety)	Form	kg ai/ha	Water L/ha	no.	days	Straw[1]	
Trial R93-35A UK, 1993 (Winter, Clarine)	500 g/, SC	0.15 + 0.25	200	2	69	1.61	
Trial R93-33A UK, 1993 (Winter, Apollo)	500 g/, SC	0.25	200	1	113	0.09	29405
Trial R93-33A UK, 1993 (Winter, Apollo)	500 g/, SC	0.15 + 0.25	200	2	70	0.57	
Trial R94-002A UK, 1994 (Winter, Spark)	500 g/, SC	0.25	200	1	104	0.21	31080
Trial R94-002A UK, 1994 (Winter, Spark)	500 g/, SC	0.15 + 0.25	200	2	71	0.87	31080

[1] ND = 0.01 mg/kg for straw (< 20% of LOQ).

Barley

Table 62. Quinoxyfen residues in barley straw from foliar applications in Germany, UK, and France.

Barley straw	Application				PHI	Residues, mg/kg	Reference
country, year (variety)	Form	Kg ai/ha	Water L/ha	no.	days	Straw[1]	
GAP, France	500 g/L SC	0.15	130-300	1	56		
Trial R98-002A S. France, 1998 (Winter, Majestic)	500 g/L SC	0.15 + 0.16	253	2	65	1.34	69430
Trial R98-002A S. France, 1998 (Winter, Nevada)	500 g/L SC	0.15 + 0.16	246	2	55	1.77	
Trial R93-30A N. France, 1993 (Winter, Energy)	500 g/L SC	0.25	200	1	75	1.23	31620
Trial R93-30A N. France, 1993 (Winter, Energy)	500 g/L SC	0.15 + 0.25	200	2	55	2.10	
Trial R93-29B N. France, 1993 (Winter, Plaisant)	500 g/L SC	0.25	300	1	58	1.13	29912
Trial R93-29B N. France, 1993 (Winter, Plaisant)	500 g/L SC	0.15 + 0.25	300	2	47	1.77	
Trial R94-021 N. France, 1994 (Spring, Alexis)	500 g/L SC	0.25	260	1	65	0.13	31646
GAP, Germany	500 g/L SC	0.15	200-400	2, 0.25 kg ai/ha/season	49		
	500 g/L SC	0.25	-	1	BBCH25 – 32		

Barley straw	Application				PHI	Residues, mg/kg	Reference
country, year (variety)	Form	Kg ai/ha	Water L/ha	no.	days	Straw[1]	
Trial RF93-114A Germany, 1993 (Winter, Igri)	500 g/L SC	0.25	400	1	66	1.56	31263
Trial RF93-114A Germany, 1993 (Winter, Igri)	500 g/L SC	0.15 + 0.25	400	2	58	1.76	
Trial RF93-115S Germany, 1993 (Winter, Iastrid)	500 g/L SC	0.25	400	1	75	0.22	31261
Trial RF93-115S Germany, 1993 (Winter, Iastrid)	500 g/L SC	0.15 + 0.25	400	2	65	0.38	
Trial RF94-039A Germany, 1994 (Winter, Grete)	500 g/L SC	0.25	400	1	66	0.29	31635
Trial RF94-039A Germany, 1994 (Winter, Grete)	500 g/L SC	0.15 + 0.25	400	2	54	0.54	
Trial RF94-040A Germany, 1994 (Winter, Jana)	500 g/L SC	0.25	400	1	72	0.20	31648
Trial RF94-040A Germany, 1994 (Winter, Jana)	500 g/L SC	0.15 + 0.25	400	2	55	0.86	
GAP, UK	250 g/L OR 500 g/L	0.15	200-400	1-2	~60 Zadoks 49		
Trial R93-34A UK, 1993 (Winter, Pasoral)	500 g/L SC	0.25	200	1	96	0.30	24035
Trial R93-34A UK, 1993 (Winter, Pasoral)	500 g/L SC	0.15 + 0.25	200	2	82	0.58	
Trial R93-85A UK, 1993 (Spring, Alexis)	500 g/L SC	0.25	200	1	76	2.94	31267
Trial R93-85A UK, 1993 (Spring, Alexis)	500 g/L SC	0.15 + 0.25	200	2	64	5.25	
Trial R94-003A UK, 1994 (Winter, Halcyon)	500 g/L SC	0.25	200	1	91	0.22	31235
Trial R94-003A UK, 1994 (Winter, Halcyon)	500 g/L SC	0.15 + 0.25	200	2	70	1.15	

[1] ND = 0.002 mg/kg for barley grain (< 20% of LOQ).

Dried herbs

Hops

Table 63. Quinoxyfen residues on dried hops from foliar applications in the US and Germany.

Hops, dry country, year (variety)	Application					PHI days	Residues mg/kg	Reference
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
GAP, Germany	250 g/L SC	0.15	0.011		4 or max 0.5 kg ai/ha/season	28		
Trial RF98-200A Germany, 1998 (Hallertauer tradition)	250 g/L SC	0.16 - 0.24	0.007		3, total 0.62 kg ai/ha/season	27[1] 35	(0.03) 0.04 0.04	73994
Trial RF98-200B Germany, 1998 (Hallertauer tradition)	250 g/L SC	0.16- 0.24	0.007		3, total 0.63 kg ai/ha/season	27[1] 35	(0.02) 0.03 < 0.02	73994
Trial RF98-200C Germany, 1998 (Hallertauer tradition)	250 g/L SC	0.160 - 0.24	0.007		3, total 0.61 kg ai/ha/season	27[1] 35	(0.03) 0.04 0.07	73994
Trial RF98-200D Germany, 1998 (Spalter)	250 g/L SC	0.20 - 0.251	0.007		3, total 0.69 kg ai/ha/season	28[1] 35	(0.19) 0.27 0.05	73994
Trial G99033R Germany, 1999 (Spalter)	250 g/L SC	0.24- 0.250	0.007		3, total 0.69 kg ai/ha/season	28 30	0.76 0.28	74050
Trial G99085R Germany, 1999 (Perle)	250 g/L SC	0.15- 0. 25	0.007		3, total 0.60 kg ai/ha/season	28 35	0.55 0.34	74050
Trial G99086R Germany, 1999 (Perle)	250 g/L SC	0.16- 0.25	0.007		3, total 0.62 kg ai/ha/season	28 35	0.37 0.23	74050
Trial G99087R Germany, 1999 (Perle)	250 g/L SC	0.16- 0.24	0.007		3, total 0.62 kg ai/ha/season	28 35	0.41 0.28	74050
GAP, USA	250 g/L SC	0.15			4 or max 0.6 kg ai/ha/season	21		
Trial 99-WA09 Washington, USA 1999 (Nugget hops)	250 g/L SC	0.15- 0.22	0.018		3, total 0.59 kg ai/ha/season	20	0.39[2]	83727
Trial 99-OR12 Oregon, USA 1999 (Nugget hops)	250 g/L SC	0.15- 0.23	0.020		4, total 0.75 kg ai/ha/season	21	1.22[2]	83727
Trial 99-ID03 Idaho, USA, 1999 (Nugget tops)	250 g/L SC	0.15- 0.22	0.021		3, total 0.61 kg ai/ha/season	21	2.17[2]	83727

[1] Residues in parenthesis were levels on fresh hops as actually determined. The residues in dried hops were estimated assuming fresh hops have a moisture content of 80% and dried hops, about 10%, i.e., a concentration of about 70% in residues after drying.

[2] Average of two replicate samples (from the same plot).

Miscellaneous Fodder and Forage crops

Sugar beet Tops

The eight supervised trials conducted on sugar beets in Northern Europe (Germany, Northern France and the UK) were described above. Samples were immediately frozen and kept in frozen storage at about -18ºC up to 4 months (1999 trials) or 179 days (2000 trials), until analysis.

Table 64. Quinoxyfen residues in sugar beet tops from foliar applications in Germany, the UK, and northern France.

Sugar beet tops country, year (variety)	Application					PHI days	Residues mg/kg	Reference
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
GAP, Germany	500 g/L SC	0.12	0.062	200-400	2	28		
Trial R99-021C Germany, 1999 (Aries)	500 g/L SC	0.15	0.075	200	2	28	0.27	76690
Trial CEMS-1348C Germany, 2000 (Beta vulgaris)	500 g/L SC	0.153 0.152	0.075	202-204	2	28	0.10	83187
Trial CEMS-1348D Germany, 2000 (Beta vulgaris)	500 g/L SC	0.155 0.154	0.075	205-207	2	28	0.10	83187
GAP, UK	500 g/L SC	0.15	0.075	200-400	2	28		
Trial R99-021A UK, 1999 (Madison)	500 g/L SC	0.15	0.075	200	2	Whole plant 0 7 14 21 Tops 28	 1.0 0.43 0.10 0.06 0.22	76690
Trial R99-021B UK, 1999 (Madison)	500 g/L SC	0.15	0.075	200	2	Whole plant 0 7 14 21 Tops 28	 0.71 0.69 0.24 0.14 0.37	76690
Trial CEMS-1348A, UK 2000 (BEA VA/ Chorus)	500 g/L SC	0.136 0.158	0.075	180-210	2	Whole Plant 0 7 14 21 Tops 28	 1.3 0.57 0.41 0.23 0.36	83187

Sugar beet tops country, year (variety)	Application					PHI days	Residues mg/kg	Reference
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Trial CEMS-1348B, UK 2000 (BEA VA/ Jackpot)	500 g/L SC	0.154 0.144	0.075	192-205	2	Whole plant 0 7 14 21 Tops 28	 1.4 0.55 0.23 0.14 <u>0.13</u>	83187
GAP, France	500 g/L SC	0.15	0.10		1	28		
Trial R99-021D France (northern), 1999 (Acces)	500 g/L SC	0.15	0.075	200	2	28	<u>0.07</u>	76690

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

In storage

No information was provided on the fate of quinoxyfen residues under commercial storage conditions.

In processing

Two processing studies were conducted in northern France and the United Kingdom in 1994 to determine the residues in processed fractions of flour and bread from winter wheat samples treated with quinoxyfen (31600; 31607, Gambie and Press, 1995).

Grains from foliar application to wheat at GAP rate were first cleaned to remove non-wheat material such as broken kernels and straw. Before milling, samples were conditioned by adding water and again cleaned. The samples were then milled at a feed rate of 5 kg per hour. Each passage and consequent sieving in the milling resulted in flour fractions called respectively the 1st, 2nd and 3rd reduction flour. In all milling experiments, approximately 200 g of sample material were taken for residue analysis from the 1st reduction, the white bread flour and the finished offal. These samples were rapidly deep frozen. The flour fractions were blended by weight and starting with 5 kg of flour, dough's were mixed, dough pieces moulded and placed in greased aluminium-coated tins. Baking was done in a rotary oven at 240ºC for 30 minutes for white bread and 15 minutes for wholemeal bread, with steam injection at the start of baking.

All grain samples (RAC) and processed fractions for residue analysis were frozen at -20ºC until analysis about 280 days later. Residues of quinoxyfen in the RAC were determined by gas chromatography with mass selective detection, following method ERC 94.5, which had a lowest validated level of 0.01 mg/kg for grain. Residues of quinoxyfen in flour, bran and bread were determined using gas chromatography with mass selective detection, following method ERC 95.16, which had a lowest validated level of 0.01 mg/kg.

Table 65. Residues in wheat grain and processed fractions.

Processed Fraction	Residues mg/kg	Reference
Wheat grain (RAC)	< 0.01	31600
Flour, 1st reduction	ND	
Flour, 74% extraction	ND	

Processed Fraction	Residues mg/kg	Reference
Wholemeal flour Bran White bread Wholemeal bread	< 0.01 0.01 ND < 0.01	
Wheat grain (RAC) Flour, 1st reduction Flour, 76% extraction Bran White bread	ND ND < 0.01 0.01 ND	31607

ND = < 0.002 mg/kg

The residues of quinoxyfen after processing barley grain following treatment with quinoxyfen into beer was investigated in the UK in 1994 (31624, Gambie and Teasdale, 1995). To ensure that the grain for brewing had sufficient residues, spring barley was treated with two applications of a suspension concentrate formulation containing quinoxyfen at 500 g/L, at double the maximum recommended application rate. After collecting about 1 kg of grain for the determination of residues in the RAC, the remaining grain was bulked and sent for processing into beer.

The grain samples were cleaned by sieving prior to processing following commercial practices. Barley malt was prepared. A 200g sample of malt was frozen for residue analysis. The remainder was milled and then mashed and the wort filtered. The spent grains from the filtration process were homogenized and a 1kg sub-sample frozen for analysis. After adjustment of the pH the wort was boiled and the sugar content adjusted prior to fermenting. After storage of the green beer for one month at 0-4ºC, the beer was filtered, bottled, and pasteurised. Five litres of beer in bottles were stored at 10ºC prior to shipment for residue analysis.

Grain, malt, and spent grain samples were analyzed by method ERC 94.5 using gas chromatography with mass selective detection. The LOQ for the method was 0.01 mg/kg. Procedural recoveries were 88% for grain, 90% for malt, and 88% for spent grains. Residues of quinoxyfen in beer were quantified by gas chromatography with mass selective detection according to method ERC 95.19. The LOQ for the method was 0.01 mg/kg. Procedural recovery for beer was 73%.

Table 66. Residues in barley grain and processed fractions (31624).

Matrix	Residues mg/kg	Processing factor
Barley grain (RAC)	0.02	-
Malt	0.01	0.5
Spent grain	0.01	0.5
Beer	ND (< 0.002)	0.1

Several processing studies were carried out in 1995 in Italy, France, and Germany on red and white wine grapes (46975; 47145; Khoshab, 1996 and 47868 Khoshab and Volle, 1996). In the trials in France, vines received seven sequential applications of a suspension concentrate formulation containing 250 g/L quinoxyfen, at 11–15 day intervals between post-flowering and 20–21 days before harvest. Grape samples were taken at normal harvest (20–21 days after final application). Samples of the grapes for the RAC were frozen immediately for residue analysis. Additional samples were taken and transported to the processing facility on the same day. Processing was carried out within 48 hours of harvest.

Residue trials on red and white wine grapes were also carried out at two locations in Germany during 1995. The wine grape vines received seven sequential applications of a suspension concentrate formulation containing a mixture of 200 g/L quinoxyfen and 60 g/L fenarimol, at a rate of about 0.07 kg ai/ha. Applications were made between post flowering and 22 days before harvest at approximately equal intervals. Grape samples were taken at normal harvest. Samples for the RAC were immediately frozen after harvest. Additional samples taken for wine processing were processed within 8 hours of harvest.

Trials on white wine grapes were conducted in Italy in the same period. The vines were also treated with seven sequential applications of a suspension concentrate formulation containing 250 g/L quinoxyfen at the rate of 0.065 kg ai/ha. Applications were made between post flowering and 21 days before harvest at approximately equal intervals. Grape samples were taken at normal harvest (21 days after the last application). Samples for the RAC were frozen immediately for residue analysis. Additional samples were collected and transported the same day for processing into wine. Processing started within 7 hours after arrival at the processing facility.

For the trials in France, processing was carried out according to the VITI R&D methods VINIF/001 and VINIF/002 for white and red grapes, respectively. The processing method used by the German and Italian trials was according to the BBA Guideline Part IV, 3-4. Both these methods follow commercial practices.

In addition to the grapes (RAC), samples of pomace, must and wine were taken in each trial for residue analysis. Grapes were separated from stalks and prepared in a cutter without dry ice. Grape pomace samples were prepared in a homogeniser with dry ice. No preparations were required for must and wine. All prepared samples were stored deep frozen until analysis. Samples were kept frozen at about -20ºC for a period of 9 to 12 months before analysis. All samples were analyzed following the methods described below.

Residues of quinoxyfen in grape samples were determined using method ERC 94.29 which has a lowest validated level of 0.01 mg/kg. Residues in grape pomace, must and wine samples were determined using method ERC 95.26 which has a lowest validated level of 0.05 mg/kg for pomace and 0.01 mg/kg for must and wine.

Table 67. Residues in grapes and processed fractions.

Matrix	Residues mg/kg	Processing factor	Reference
Red wine grapes (RAC)	0.08		47868
Must	< 0.01	0.13	
Pomace	0.25	3.1	
Wine (2 months)	ND (< 0.002)	0.03	
Wine (6 months)	ND < 0.002)	0.03	
White wine grapes (RAC)	0.15		47868
Must	< 0.01	0.07	
Pomace	0.78	5.2	
Wine (2 months)	ND (< 0.002)	0.01	
Wine (6 months)	ND (< 0.002)	0.01	
Red wine grapes (RAC)	0.46		46975
Must	0.02	0.04	
Pomace	1.00	2.2	
Young wine	ND (< 0.002)	0.004	
Mature wine	ND (< 0.002)	0.004	
White wine grapes (RAC)	0.14		46975
Must	0.01	0.07	
Pasteurized must	< 0.01	0.07	
Pomace	0.72	5.1	
Young wine	ND (< 0.002)	0.01	

Matrix	Residues mg/kg	Processing factor	Reference
Pasteurized young wine	ND (< 0.002)	0.01	
Mature wine	ND (< 0.002)	0.01	
Pasteurized mature wine	ND (< 0.002)	0.01	
White wine grapes	0.52		47145
Must	0.03	0.06	
Pomace	1.72	3.3	
Wine (2 months)	ND (< 0.002)	0.004	
Wine (6 months)	ND (< 0.002)	0.004	
		AVERAGE (n = 5)	MEDIAN
Grapes (RAC)		-	
Must		0.07	
Pomace		3.8	
Young wine/ 2 months		0.01	0.01
Mature wine/ 6 months		0.01	0.01

One of the trials conducted in California in 1999 to determine the residues of quinoxyfen in grapes (RAC) included a determination of the residues on juice and raisins (83731, Thompson, 2001). Mature fruits were harvested 14 days after the last application and sent to the processing laboratory on the day of sampling and kept frozen until processing. A representative sample was taken for RAC residues and the rest of the bulk sample was processed into grape juice and raisins, simulating commercial practices.

Grapes were passed through a crusher/destemmer. The stems were discarded, the crush was transferred to a steam-jacketed kettle, and pectinase enzyme was added. The crush was then heated to 120–130ºF for a minimum of 2 hours. The depectinized grape crush slurry was passed through a screw press to extract unclarified juice and wet pomace, which was discarded. The unclarified juice was heated to inactivate the added enzyme and then cooled in a refrigerator for argol settling. After 34 days, the argol-settled juice was filtered using a plate-and frame filter press with depth filter pads and filter aid. The filtered juice was heated to canning temperature, put in jars and sealed. Once cool, the jars were weighed, labelled, bagged, and placed in frozen storage (-10º to -5ºF) until shipment to the analytical laboratory for analysis of residues.

Grape bunches were spread on stainless steel drying trays and placed outdoors with adequate protection from birds, insects, and animals. The grapes were turned about once a week during the drying process, which took approximately 1 month. When dry, the moisture content was measured, and the dried grapes were placed in plastic bags and stored at 70ºF for approximately 13 days to achieve moisture equilibrium. The dried grapes were frozen, gently rubbed on 4-mesh screen for destemming and cap stem removal, and immersed in 77ºF water for approximately 15 minutes for rehydration to raisins. Excess water was drained, and the raisins were placed in plastic bags and returned to storage to achieve moisture equilibrium. After 12 days the moisture content was again measured , and the raisins were weighed, bagged, labelled and placed in frozen storage (-10º to -5ºF) until shipment to the analytical laboratory.

Samples were analyzed using method ERC 95.26, which had a lowest validated level of 0.01 mg/kg quinoxyfen for grapes and processed fractions. Residues of quinoxyfen were quantified by gas chromatography with mass selective detection.

Table 68. Residues in grapes, juice, and raisins (83731).

Matrix	Residues mg/kg	Processing factor
Grapes (RAC)	0.177	
Raisins	0.117 (0.109, 0.110, 0.115, 0.132)[1]	0.66
Juice	< 0.01 (< 0.01 (4))[1]	0.06

[1] Replicate sub-samples.

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Two cattle feeding studies were conducted in 1994. One of the studies was conducted using fourteen lactating Friesian cows aged between 4 and 9 years old with weight range of 480 to 644.5 kg and daily milk yield of at least 10 kg/day (31599; Gambie and Long, 1995). The animals were divided into the following treatment groups:

Dose Group	Number of animals	Treatment	
		ppm total diet per day	mg/animal/day
1	3	Untreated	0
2	3	0.2	4
3	3	0.6	12
4	5	2.0	40

The animals received a feed concentrate twice daily. On each occasion, the appropriate amount of quinoxyfen was added to the feed concentrate as a corn oil solution, based on a daily food consumption of 20 kg. The untreated group of animals received 20 ml corn oil. The animals were dosed for 28 days. Milk samples were taken throughout the study for residue analysis. In addition whole milk samples from days 14 and 27 were separated into cream and skimmed milk and samples taken for both residue analysis and determination of fat content. Milk samples were stored frozen at about -20ºC until analysis in about 51 days. .

After 28 days of dosing, all cows except two from the highest treatment group were sacrificed. The remaining two cows were maintained on a basal diet from day 29 and were sacrificed 7 and 14 days after the end of the dosing period (days 36 and 43, respectively).

At sacrifice, samples of the following tissues were taken for residue analysis: subcutaneous fat, peritoneal fat (perirenal and omental pooled fat), skeletal muscle (pooled from the pectoralis and abductor muscle of the thigh), liver and kidney. The samples were coarsely chopped before freezing and storage at -20ºC until analysis in 76-255 days.

A supplementary study (31634; Gambie, Teasdale, and Press, 1995) was undertaken with higher feeding levels of quinoxyfen. The study used six lactating Friesian cows aged between 4 and 6 years old with a weight range of 500-620 kg and daily milk yield at least 11 kg/day. Three cows were treated at dose level of 20 ppm total diet/day, equivalent to 400 mg/animal/day. The other three animals received no treatments. The animals were dosed for 28 days and 16 hours after the last dose, they were sacrificed. The same tissue samples as the other study were collected and frozen.

Samples of whole milk, skimmed milk, and cream were analyzed for residues of quinoxyfen using method ERC 94.7, which had a lowest validated level of 0.001 mg/kg. Mean procedural recoveries were 81% for whole milk and 80% for cream. The fat content of whole milk and cream samples was determined by the Rose-Gottlieb gravimetric method (BS 1741: Part 3: 1987).

Method ERC 94.30 was used for the determination of residues of quinoxyfen in liver, where quantification was done by gas chromatography with mass selective detection. The method had an LOQ of 0.01 mg/kg. Procedural recoveries for liver samples were below 70%.

For analysis of residues of quinoxyfen in kidney, muscle and fat, method ERC 94.20, which had a lowest validated level of 0.01 mg/kg, was used. Mean procedural recoveries of up to 94% for kidney, 92% for muscle, and 84% to 92% for fat were obtained from samples fortified at levels of 0.01mg/kg to 1.0 mg/kg.

In all these methods, quinoxyfen residues were quantified by gas chromatography with mass selective detection. All residues equivalent to less than 20% of the lowest validated level were classified as "not detected" (ND).

From the first study (31599), milk sampled on Days 14 and 27/28 was separated into cream and skimmed milk and both the fat content and quinoxyfen residues were determined.

During the withdrawal period from day 29 to 42, residues in whole milk decreased from 0.007 to < 0.001 mg/kg within 4 days after dosing ceased. After 14 days depuration period, no residues were detected in milk. Tissue residues were determined after 7 and 14 day depuration period. After 7 and 14 days, residues in peritoneal fat were 0.05 mg/kg and < 0.01 mg/kg, respectively. Residues in other tissues were either < 0.01 mg/kg or not detected during the depuration period.

Table 69. Mean residues of quinoxyfen in whole milk over 28 days oral administration of quinoxyfen to dairy cows (31599; 31634).

DAY	Concentration of quinoxyfen, mg/kg				
	Treatment groups (ppm feed/day)				
	Untreated	0.2	0.6	2.0	20
-1	ND	ND	ND	ND	ND
1	-	-	-	< 0.001	0.017
3	-	-	-	0.006	0.085
5	-	-	-	0.007	-
7	< 0.001	0.001	0.002	0.007	0.37
10	-	-	-	0.009	0.19
14	ND	0.001	0.002	0.009	0.18
18	-	-	-	0.009	-
21	ND	< 0.001	0.002	0.007	0.15
24	-	-	-	0.007	0.16
28*	-	< 0.001	0.002	0.007	-
MEAN	< 0.0005	0.001	0.002	0.007	0.16
30	-	-	-	0.005	0.11
32	-	-	-	< 0.001	-
35	-	-	-	< 0.001	-
37	-	-	-	< 0.001	-
40	-	-	-	< 0.001	-
42	-	-	-	ND	-

* Final day of dosing; - No analysis; ND = not detected (< 0.0002 mg/kg)

Table 70. Residues in whole milk, skim milk, and cream on days 14 and 27/28 following oral administration of quinoxyfen to dairy cows (31599; 31634).

Treatment, ppm in feed/day		Quinoxyfen residues[1] (mg/kg)			Quinoxyfen ratio: cream/whole milk	Fat ratio: cream/whole milk
		Whole milk	Skim milk	Cream		
Day 14						
0.2	Min	< 0.001	ND[2]	< 0.001	-	12.5
	Max	< 0.001	ND	0.007	>7	9.8
	Mean	< 0.001	ND	0.003	>3	10.7
0.6	Min	0.002	ND	0.016	8.0	11.6
	Max	0.002	ND	0.022	11.0	15.2

Treatment, ppm in feed/day		Quinoxyfen residues[1] (mg/kg)			Quinoxyfen ratio: cream/whole milk	Fat ratio: cream/whole milk
		Whole milk	Skim milk	Cream		
	Mean	0.002	ND	0.018	9.2	12.9
2.0	Min	0.010	0.005	0.046	4.6	3.1
	Max	0.007	< 0.001	0.077	11.0	12.9
	Mean	0.0088	0.0022	0.068	7.9	9.3
Day 27						
0.2	Min	< 0.001	< 0.001	0.006	>6	12.2
	Max	< 0.001	< 0.001	0.007	>7	12.9
	Mean	< 0.001	< 0.001	0.0067	>7	12.0
0.6	Min	0.002	ND	0.011	5.5	12.3
	Max	0.002	ND	0.02	10.0	14.8
	Mean	0.002	ND	0.015	7.3	13.4
2.0	Min	0.009	0.004	0.041	4.6	9.7
	Max	0.007	0.001	0.079	11.3	13.7
	Mean	0.007	0.002	0.059	8.6	11.4

[1]Min and Max refer to the ratio of quinoxyfen in cream/quinoxyfen in whole milk on a per cow basis within each treatment group.

.[2]ND = not detected (< 0.0002)

Table 71. Summary of residues in tissues following 28 days oral administration of quinoxyfen to dairy cows (31599; 31634).

Treatment, ppm feed/day (Reference)		Residues, mg/kg				
		Liver	Kidney	Skeletal muscle	Subcutaneous fat	Peritoneal fat
0.2 (GHE-P-4161)	Min	ND	ND	ND	ND	< 0.01
	Max	ND	< 0.01	ND	< 0.01	0.02
	Mean	ND	< 0.01	ND	< 0.01	0.01
0.6 (GHE-P-4161)	Min	ND	ND	ND	< 0.01	< 0.01
	Max	< 0.01	< 0.01	ND	< 0.01	0.02
	Mean	< 0.01	< 0.01	ND	< 0.01	0.012
2.0 (GHE-P-4161)	Min	< 0.01	< 0.01	< 0.01	0.02	0.09
	Max	< 0.01	< 0.01	< 0.01	0.04	0.10
	Mean	< 0.01	< 0.01	< 0.01	0.03	0.09
20.0 GHE-P-4185)	Min	0.04	0.07	0.06	0.78	1.1
	Max	0.21	0.29	0.18	2.0	3.2
	Mean	0.12	0.19	0.11	1.4	2.2

ND = 0.002 mg/kg

A poultry feeding study conducted in 1995 consisted of four groups of 10 Isa Brown laying hens (5-6 months old, weighing *ca* 1-2 kg) fed at the following dose levels: 0.1, 0.3, and 1.0 mg/kg of diet/ day based on the average daily intake of food in the group (31744, Jack. and Dunsire, 1995). The fourth group of hens served as controls and were dosed with empty gelatin capsules. The hens were

fed gelatin capsules containing radiolabelled (mixture of ^{14}C-quinoline label and ^{14}C-phenoxy label) ^{14}C-quinoxyfen. Each daily dose was administered in a single capsule at the same time each day for 28 days.

Within each dose group, the ten hens were subdivided into 3 sub-groups of 3 or 4 hens. All samples were pooled by sub-group prior to analysis. Eggs were collected daily from all hens and pooled for residue analysis. On days 14 and 28, the egg samples were separated into whites and yolks, which were analyzed separately.

At the end of the dosing period (approximately 23 hours after last dose on day 28), the hens were sacrificed and the following edible tissues retained for analysis: skin with fat, breast muscle, thigh muscle, liver, kidney, and abdominal fat pad. An approximately equal weight of tissue from each bird in each sub-group was combined and thoroughly mixed.

With the exception of eggs, samples not analyzed immediately were stored frozen at -20ºC until analysis. Eggs were stored at 4ºC until analysis. After combustion of the samples, radioactivity was measured by LSC.

Table 72. Residues in eggs over 28 days oral administration of ^{14}C-quinoxyfen to laying hens (31744).

Dose period, Day	Residues (TRR), µg equivalent/g Treatment (ppm feed/day)		
	0.1 ppm	0.3 ppm	1.0 ppm
1	0.000	0.000	0.000
2	0.000	0.001	0.003
3	0.001	0.002	0.006
4	0.001	0.004	0.009
5	0.002	0.005	0.014
6	0.002	0.006	0.018
7	0.002	0.007	0.021
8	0.002	0.007	0.024
9	0.002	0.008	0.023
10	0.002	0.008	0.023
11	0.002	0.008	0.024
12	0.002	0.008	0.023
13	0.002	0.008	0.023
14 (whites)	0.000	0.001	0.002
15 (yolks)	0.008	0.025	0.071
15	0.002	0.008	0.023
16	0.003	0.008	0.025
17	0.003	0.009	0.024
18	0.003	0.009	0.025
19	0.003	0.009	0.023
20	0.003	0.009	0.024
21	0.003	0.010	0.024
22	0.003	0.009	0.024
23	0.003	0.009	0.023
24	0.003	0.009	0.025
25	0.003	0.010	0.025
26	0.003	0.010	0.025
27	0.003	0.011	0.025

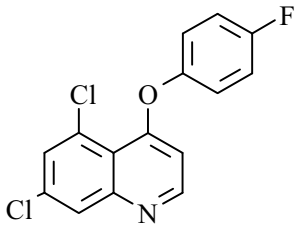
Dose period, Day	Residues (TRR), µg equivalent/g Treatment (ppm feed/day)		
	0.1 ppm	0.3 ppm	1.0 ppm
28 (white)	0.000	0.001	0.002
28 (yolks)	0.010	0.036	0.072

Table 73. Residues in poultry tissues following 28 days oral administration of ^{14}C-quinoxyfen to laying hens (31744).

Treatment, ppm feed/day (Reference)		Residues (TRR), mg equivalent/kg					
		Liver	Kidney	Breast muscle	Thigh muscle	Skin with fat	Abdominal fat pad
0.1 (GHE-P-4394)	Min	0.009	0.003	0.000	0.000	0.003	0.007
	Max	0.009	0.004	0.000	0.000	0.008	0.013
	Mean	0.009	0.003	0.000	0.000	0.005	0.010
0.3 (GHE-P-4394)	Min	0.035	0.016	0.000	0.001	0.012	0.028
	Max	0.040	0.018	0.000	0.001	0.017	0.037
	Mean	0.038	0.017	0.000	0.001	0.014	0.034
1.0 (GHE-P-4394)	Min	0.087	0.039	0.001	0.005	0.034	0.080
	Max	0.097	0.049	0.002	0.009	0.063	0.12
	Mean	0.093	0.045	0.001	0.007	0.051	0.10

APPRAISAL

Chemical name: 5,7-dichloro-4-quinolyl 4 fluorophenyl ether



Animal metabolism

The Meeting received results of an animal metabolism study in lactating goats. Two goats were orally dosed with phenoxy ^{14}C-quinoxyfen (purity > 98%), twice daily for five consecutive days, at a rate of 10.7 mg quinoxyfen/kg feed. Similarly, two goats were treated with quinoline ^{14}C-quinoxyfen, twice daily for five consecutive days, at a rate of 11.7 mg quinoxyfen/kg feed.

Urine, faeces and cage wash accounted for 77–80% of the total administered dose. Milk contained 0.5–0.9%, liver 0.9–1.3%, and kidney 0.0–0.05%. The radioactive residue appeared to plateau in milk on day 4. Total radioactive residues (TRRs) in tissues and milk from use of the phenoxy labelled quinoxyfen were 0.34 mg/kg in kidney, 1.0 mg/kg in liver, 0.032 mg/kg in muscle, 0.20 mg/kg in omental fat, and 0.11 mg/kg in milk (16 hours after final dose); and from the use of the quinoline labelled quinoxyfen, 1.5 mg/kg in liver, 0.22 mg/kg in kidney, 0.032 mg/kg in muscle, 0.32 mg/kg in perirenal fat, and 0.064 mg/kg in milk.

Quinoxyfen was identified in milk (30–40% TRR), kidney (2–4% TRR), liver (10–20% TRR), and fat (50–97% TRR). DCHQ (5,7-dichloro-4-hydroxyquinoline) and/or 4-fluorophenol, resulting from cleavage of the ether linkage, was/were found in small amounts (< 5% TRR) in milk, kidney, and liver. Enzyme deconjugation of liver extracts indicated additional substantial quantities of these two compounds (13–20% TRR) present as conjugates. Less than 5% TRR was attributed to isomeric hydroxy quinoxyfens in liver, milk, and subcutaneous fat. 2-Oxo-quinoxyfen was found in milk at a maximum of 1.4% TRR.

The metabolism in goat and rat are qualitatively similar. Cleavage of the ether linkage to form 4-fluorophenol and DCHQ is seen in both animals. Isomers of fluorophenyl-ring hydroxylated quinoxyfen were found in the rat (bile and faeces), whereas isomers of quinoline-ring hydroxylated quinoxyfen (2-oxo) were found in the goat metabolism study. The latter were at very low levels (< 0.1% of the administered dose for the 2-oxo quinoxyfen) in the rat.

The Meeting concluded that the major metabolite in ruminant commodities from the oral administration of quinoxyfen is the parent quinoxyfen. Degradation from cleavage of the ether linkage generates free DCHQ and 4-fluorophenol. Another minor pathway involves formation of hydroxy derivatives.

Plant metabolism

The Meeting received plant metabolism studies for the foliar application of phenoxy- and quinoline-labelled [^{14}C] quinoxyfen, in separate experiments, to winter wheat, sugar beets, grapes, cucumber and tomato.

In each crop tested, parent quinoxyfen was found to be a significant to very major portion of the TRR: 8–27% TRR in wheat straw, 25% TRR in sugar beet root, 19–30% TRR in sugar beet tops, 93–98% TRR in grapes, 64–74% TRR in cucumber fruit, and 63–65% TRR in tomato fruit. DCHQ (7% TRR) and 4-fluorophenol (17% TRR) were found in sugar beet tops, indicative of ether bond cleavage. CFBPQ (2-chloro-10-fluoro(1)benzopyrano (2,3,4-de)quinoline), a product of photolysis, was also found in sugar beet tops (3–5% TRR) and possibly in wheat straw at 2% TRR. The 2-oxo quinoxyfen and p-hydroxyphenoxy quinoxyfen metabolites were tentatively identified at low concentrations (< 5% TRR) in several crops. Much of the unidentified extractable radioactivity in wheat straw was found to be multicomponent and of an acidic anionic nature (about 20% TRR) not related to conjugates of quinoxyfen with natural products.

About ≥ 20% of the TRR in mature wheat straw was characterized as lignin, and 25% was associated with cellulose. About 13–53% TRR in wheat grain (100% TRR = 0.03 mg/kg) was associated with starch. About 10% TRR in tomato was associated with lignin, cellulose, and hemicellulose.

Quinoxyfen was shown to have no tendency to translocate in grape vines. Radiolabeled material applied only to some vine leaves did not move to the fruit or to untreated leaves.

The Meeting concluded that quinoxyfen is a major portion of the residue when applied in a foliar fashion to several crop types (grapes, cucumber and tomato). In wheat quinoxyfen was extensively metabolized with portions of the molecule becoming associated with natural plant constituents. Minor metabolic pathways were cleavage of the ether bond, photolysis, and hydroxylation of the quinoline or phenyl rings.

Environmental fate

The Meeting received information on the aqueous hydrolysis, aerobic soil metabolism, aqueous photolysis, and soil photolysis of quinoxyfen. Confined rotational crop studies with radiolabeled quinoxyfen were also provided.

Quinoxyfen is stable under aqueous hydrolysis at pH 7 and 9, but degrades slowly under acidic hydrolysis conditions. The half-life at pH 4 at 25 °C is about 75 days. The hydrolysis product was identified as DCHQ.

Quinoxyfen is relatively stable under conditions of dark aerobic soil metabolism. After 200 days, 53–81% of the quinoxyfen applied to various soil types remained. Some 0–27% had been converted to 2-oxo quinoxyfen, and 0–5% was present as DCHQ. About 15–25% of the original quinoxyfen had become bound to the soil. Less than 2% had been converted to carbon dioxide. Half lives of 90 to 500 days were calculated for the various soil types.

Quinoxyfen degraded in aqueous solution under artificial light (298 nm) to yield CFBPQ and DCHQ (minor). Half lives under typical use conditions were calculated to vary from 7 to 16 hours. Other work under natural sunlight conditions in water and water sediment systems showed the rapid loss of quinoxyfen, with no quinoxyfen remaining after 1 day. CFBPQ formed, but rapidly degraded

In contrast, quinoxyfen degraded slowly on the surface of sandy loam soil when exposed to simulated natural light. The half life was estimated to be equivalent to > 2 years in spring in England. DCHQ was identified in minor amounts, but the major metabolite remained unknown.

The uptake of radiolabeled quinoxyfen from soil into three succeeding crops (turnips, sunflower and cabbage) was reported. The quinoxyfen was applied at a rate equivalent to 400 g ai/ha, typical of the maximum seasonal use rate. Mature crop parts contained very low levels of radioactive residue, 0.4–3.5 μg/kg.

The Meeting concluded that quinoxyfen is relatively stable under aerobic conditions in soil and at neutral and alkaline pH in water, it undergoes rapid photolytic degradation in water systems, and that residues of quinoxyfen in rotational crops are unlikely.

Methods of Analysis

The Meeting received information for analytical methods for the quantitative determination of quinoxyfen in a variety of crops. The methods were used for data collection in the supervised field trials and livestock feeding studies, and several of the methods were validated by independent laboratories for use as enforcement methods. The methods were typically validated at 0.01 mg quinoxyfen/kg matrix for fruits, vegetables, and grains, with some exceptions (sugarbeet tops 0.2 mg/kg; barley and wheat straw and hops, 0.05 mg/kg). The methods were validated at 0.001 mg/kg for milk, and at 0.01 mg/kg for muscle, kidney, fat, and eggs. Bovine liver was problematic, and adequate recoveries were not achieved at levels of 0.01–1.0 mg/kg. Recovery in liver was 40% to 60%.

The various analytical methods for determination of residues of quinoxyfen in plant and animal matrices follow similar partitioning, clean-up and quantification procedures. Generally, quinoxyfen residues are extracted from plants and animal tissues samples with acidic acetonitrile. After addition of sodium bicarbonate solution to an aliquot of the extract, quinoxyfen is partitioned into hexane, which is then evaporated to dryness. The residue is reconstituted in hexane prior to an aminopropyl solid phase extraction using 1% acetone in hexane to elute quinoxyfen residues. The eluate is evaporated to dryness and reconstituted in 0.1% corn oil in tri-methyl pentane (TMP). Quinoxyfen is quantified either by gas chromatography with mass selective detection (GC-MSD) or by HPLC with UV absorbance. Specific methods differ in the clean-up steps, e.g., the use of gel permeation chromatography for livestock matrices.

The Meeting concluded that adequate analytical methods exist for the determination of quinoxyfen in crops and livestock commodities (except liver) both for data collection and MRL enforcement purposes.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the stability of quinoxyfen in a variety of crop and livestock matrices. In all cases, quinoxyfen was shown to be stable in the macerated matrices under conditions of frozen storage for an interval at least as great as the storage interval of supervised field trial or livestock feeding samples.

Quinoxyfen was stable under conditions of frozen storage for at least 80 days in cherries, 365 days in grapes, 255 days in grape juice and raisins, 530 days in wheat grain and straw, 110 days in dried hops, 280 days in lettuce, 980 days in strawberry, 320 days in bell peppers, 250 days in melons, 240 days in milk, 190 days in kidney and muscle, and 220 days in fat. Quinoxyfen appeared to be unstable in liver, 60% remaining at 240 days, but the correction for the average concurrent recovery yields a percent remaining of 93%.

The Meeting concluded that quinoxyfen is stable in a variety of analytical crop, processed commodity, and livestock commodity samples under frozen storage conditions.

Residue definition

The plant and ruminant metabolism studies show that a major portion of the residue is parent quinoxyfen. There was no indication that substantial portions of quinoxyfen exist as conjugates in the metabolic mixtures. In plant studies, significant degradation with reincorporation of the radiolabel into natural products was indicated.

No metabolism study in poultry was provided. The poultry feeding study utilized radiolabeled quinoxyfen, but no attempts were made to identify the radiolabeled residues in eggs and tissues.

The available analytical methods determine only quinoxyfen.

The residue definition in Australia, the European Union, and the United States is quinoxyfen.

Ruminant feeding studies show that quinoxyfen preferentially accumulates in fat as opposed to muscle (10:1). Likewise the quinoxyfen ratio between cream and whole milk was about 8 to 1. The goat metabolism study indicated that the TRR in the various fats was about 10× those in muscle. Finally, the octanol/water partition coefficient for quinoxyfen is 4.7.

The Meeting concluded that the residue definition for both enforcement and dietary exposure considerations for plant commodities and for farm animal commodities is quinoxyfen. The Meeting also decided that quinoxyfen is fat-soluble.

Results of supervised trials on crops

The Meeting received supervised trials data for the foliar application of quinoxyfen as a suspension concentrate formulation (SC) to a variety of crops, including cherries, grapes, strawberries, currants, melons, peppers, lettuce, sugar beets, wheat, barley, and hops.

Cherries

Field trials are reported from the USA (GAP: 250 g/L SC, 0.12 kg ai/ha, five applications per season, 7 day PHI). The ranked order of residue values on cherries (pitted) for 13 trials conducted at maximum GAP is: 0.03, 0.05, 0.08 (2), 0.11 (2), 0.12, 0.13 (2), 0.14 (2), 0.15, and 0.27 mg/kg. The Meeting estimated an STMR of 0.12 mg/kg, HR of 0.27 mg/kg and a maximum residue level of 0.4 mg/kg.

Grapes

Field trials are reported from France (GAP: 250 g ai/L SC, 0.05 kg ai/ha, three applications per year at 7–10 day intervals, 21 day PHI), Germany (GAP: 250 g ai/L EC, 0.005 kg ai/hL, four applications maximum at 10–14 day intervals, 21 day PHI, the application volume depends on the growth stage), Italy (GAP: 250 g ai/L SC, 0.008 kg ai/hL, five applications maximum per year at 8–14 day intervals, 28 day PHI), Spain (GAP: 250 g ai/L SC, 0.075 kg ai/ha, 0.008 kg ai/hL, five applications maximum per year at 10–18 day intervals 30 day PHI for wine grapes, 21 day PHI for table grapes), US (GAP: 250 g ai/L SC, 0.12 kg ai/ha, five applications maximum per season or 0.60 kg ai/ha/year at 7–21 day interval, 14 day PHI), Canada (GAP: no label, use USA), and Australia (GAP: 250 g ai/L SC, 0.005 kg ai/hL, three applications maximum at 7–14 day intervals, 14 day PHI).

The trials in France and Germany consisted of 6, 7, or 10 repeat applications. Applications made more than 30 days before harvest will not contribute significantly to the final residue. With the 6–13 day retreatment intervals and a 21 day PHI, only the last three applications will contribute to the residue. The ranked order of residues from trials conducted at the maximum GAP with the additional repeat applications (n=9) in France and Germany is: 0.02, 0.04, 0.04, 0.05, 0.05, 0.06, 0.09, 0.13, and 0.36 mg/kg.

The ranked order of the residue values on grapes for eight trials conducted at the maximum GAP in Italy is: 0.04, 0.06, 0.07, 0.10, 0.17, 0.18, 0.30, 0.49 mg/kg. The ranked order of the residue values on grapes for trials conducted at the maximum GAP in Spain is: 0.02, 0.04, 0.08, 0.22 mg/kg.

The ranked order for 13 trials in the US conducted at the maximum GAP is: 0.06, 0.08 (2), 0.09, 0.13 (2), 0.15 (3), 0.18, 0.22, 0.24, 0.44 mg/kg. The ranked order for two trials in Canada conducted at the maximum GAP of the US is: 0.22, 0.29 mg/kg.

Fifteen trials conducted in Australia comply with the PHI of 14 days, 0.01, 0.05, 0.06, 0.09 (2), 0.15 (2), 0.17, 0.18, 0.23, 0.41, 0.45, 0.54, 0.82, 1.1 mg/kg

The trial residue values from France, Germany, Italy, Spain, Canada, US, and Australia appear to be from the same population and are combined (n = 51) in rank order: 0.01, 0.02 (2), 0.04 (4), 0.05 (3), 0.06 (4), 0.07, 0.08 (3), 0.09 (4), 0.10, 0.13 (3), 0.15 (5), 0.17 (2), 0.18 (3), 0.22 (3), 0.23, 0.24, 0.29, 0.30, 0.36, 0.41, 0.44, 0.45, 0.49, 0.54, 0.82, and 1.1 mg/kg. The Meeting estimated an STMR of 0.13 mg/kg, HR of 1.1 mg/kg and a maximum residue level of 2 mg/kg.

Strawberries

Field trials were reported to the Meeting from Germany (GAP: 250 g ai/L SC, 0.12 kg ai/ha, 0.006 kg ai/hl, two applications per season, 14 day PHI) and the USA (250 g ai/L SC, 0.11 kg ai/ha, four applications per season (0.44 kg ai/ha/season), 1 day PHI).

The residue values in ranked order from the eight trials in Germany at maximum GAP were: 0.01, 0.02, 0.04, 0.05, 0.07, 0.09, 0.12, and 0.16 mg/kg.

The residue values in ranked order from the six trials in the USA at maximum GAP are: 0.16, 0.18, 0.24, 0.41, 0.46, and 0.56 mg/kg. The values of Germany and the USA are not from the same population.

Using the residue values (n=6) from the USA, the Meeting estimated as STMR of 0.32 mg/kg, HR of 0.56 mg/kg and a maximum residue level of 1 mg/kg.

Currants

Supervised field trial studies for the foliar application of quinoxyfen to black currants in Germany were reported to the Meeting. The GAP is: 240 g ai/L, 0.075 kg ai/ha, 0.0075 kg ai/hL, three applications per year, 14 day PHI.

The residue values in ranked order (n=7) for trials conducted at maximum GAP were: 0.04, 0.05, 0.06, 0.20, 0.28, 0.30, and 0.40 mg/kg.

The Meeting estimated an STMR of 0.20 mg/kg, HR of 0.40 mg/kg and a maximum residue level of 1 mg/kg.

Melons

Field trials on melons were reported from Spain (GAP: 250 g ai/L SC, 0.0075 kg ai/hL, three applications per year, 7 day PHI), Italy (GAP: 250 g ai/L SC, 0.006 kg ai/hL, 7 day PHI), Greece (No label available, use GAP Italy), and the USA (GAP: 250 g ai/L SC, 0.11 kg ai/ha, four applications per year, 3 day PHI).

The residues in ranked order for whole melons from six trials at maximum GAP in Italy and two trials in Greece are: 0.01 (2), 0.02 (4), and 0.03 (2) mg/kg; and the residues in ranked order for the pulp only were: < 0.01 (6) and 0.02 (2) mg/kg.

The residues in ranked order for whole melons from eight trials at maximum GAP in Spain are: 0.01 (4) and 0.02 (4) mg/kg; and the residues in ranked order for the pulp only are: ND - < 0.01 (8) mg/kg.

The residues in ranked order for whole melons (cantaloupes) from six trials at maximum GAP in the USA (taking into account the permitted maximum total seasonal rate) are: < 0.01, 0.02, 0.03 (3), and 0.05 mg/kg. No data were provided on pulp.

The data from the various countries are from the same population and are combined (n=22) in ranked order, for whole melon: 0.01 (7), 0.02 (9), 0.03 (5), and 0.05 mg/kg; and for pulp (n=16), 0.01 (14) and 0.02 (2) mg/kg.

The Meeting estimated an STMR of 0.01 mg/kg and HR of 0.02 mg/kg for quinoxyfen in melon pulp and a maximum residue level of 0.1 mg/kg for quinoxyfen in/on whole melon, except watermelon in both cases.

Peppers

A field trial residue study was reported from the USA (GAP: 250 g ai/L SC, 0.15 kg ai/ha, four applications per year, 0.60 kg ai/ha/year, 3 day PHI).

The residues (n=11) in ranked order for quinoxyfen residues on peppers from application at maximum GAP were: 0.01, 0.02, 0.09, 0.12, 0.15 (2), 0.16, 0.17, 0.23, 0.52, and 0.64 mg/kg.

The Meeting estimated an STMR of 0.15 mg/kg, HR of 0.64 mg/kg and a maximum residue level of 1 mg/kg for peppers (bell and non-bell).

Lettuce

A field study report was provided for the foliar application of quinoxyfen to lettuce (leaf and head) in the USA. The GAP in the USA is: 250 g ai/L SC, 0.11 kg ai/ha, four applications per season and 0.44 kg ai/ha/season, and a PHI of 1 day.

Seven trials on head lettuce were at maximum GAP, with residues in ranked order of: 0.91, 1.0, 1.2, 1.4, 2.1, 3.1, and 5.3 mg/kg. Six trials on leaf lettuce were at maximum GAP, with residues in ranked order of: 1.3, 2.9, 3.4, 4.3, 6.9, and 13 mg/kg.

The Meeting estimated an STMR of 1.4 mg/kg, HR of 5.3 mg/kg and a maximum residue level of 8 mg/kg for lettuce (head).

The Meeting estimated an STMR of 3.8 mg/kg, HR of 13 mg/kg and a maximum residue level of 20 mg/kg for lettuce (leaf).

Sugar beet roots

Field trial data were received from Germany (GAP: 500 g ai/LC SC, 0.12 kg ai/ha, two applications per season, 28 day PHI), UK (GAP: 500 g ai/L SC, 0.15 kg ai/ha, two applications, 28 day PHI), and France (GAP: 500 g ai/L SC, 0.15 kg ai/ha, one application, 28 day PHI).

The residue values for trials conducted in the three European countries at maximum GAP in ranked order are: < 0.01 (4), 0.01 (3), 0.02 mg/kg.

The Meeting estimated an STMR of 0.01 mg/kg and a maximum residue level of 0.03 mg/kg for sugar beet roots.

Wheat grain

Wheat grain trials were reported from France, Germany, and the UK. The GAPs are: 500 g ai/L SC, 0.15 kg ai/ha, one application in France with a PHI of 56 days; 500 g ai/L SC, 0.25 kg ai/ha, one

application in Germany at growth stages BBCH 25–32 (tillering), and in the UK 500 g ai/L SC, 0.15 kg ai/ha, two applications until growth stage BBCH 49 (about 60 days PHI).

Some trials in Greece were evaluated against the GAP of France. The trials in North France were evaluated against the GAP of Germany.

The trials in Greece were not within the maximum GAP of France. The trials were conducted at rates in excess of the maximum GAP, and they resulted in finite residue values (> LOQ). Some trials (n=21) in France, Germany, and the UK conducted at or in excess of the maximum GAP of the respective countries yielded residue values below the LOQ. The residue values in ranked order were: < 0.01 (21) mg/kg.

The Meeting estimated an STMR of 0.01 mg/kg and a maximum residue level of 0.01 (*) mg/kg for wheat grain.

Barley grain

Barley grain trials were reported from France, Germany, and the UK. The GAPs are: 500 g ai/L SC, 0.15 kg ai/ha, one application in France with a PHI of 56 days; 500 g ai/L SC, 0.25 kg ai/ha, one application in Germany at growth stages BBCH 25–32 (tillering), and in the UK 500 g ai/L SC, 0.15 kg ai/ha, two applications until growth stage BBCH 49 (about 60 days PHI).

All trials in Europe were conducted above the maximum GAP. Eight trials provided residue values below the limit of quantitation. The ranked order of residues is < 0.01 (8) mg/kg.

The Meeting estimated an STMR of 0.01 mg/kg and a maximum residue level of 0.01 (*) mg/kg for barley grain.

Wheat straw

Wheat trials were reported from France, Germany, and the UK. The GAPs are: 500 g ai/L SC, 0.15 kg ai/ha, two applications per season one application in France with a PHI of 56 days; 0.25 kg ai/ha, one application in Germany at growth stages BBCH 25–32 (tillering), and in the UK 500 g ai/L SC, 0.15 kg ai/ha, two applications until growth stage BBCH 49 (about 60 days PHI). Some trials in Greece were evaluated against the GAP of France (South). The trials in the UK and in North France were evaluated against the GAP of Germany.

The residues in rank order in wheat straw (n=16) were: < 0.05 (5), 0.07, 0.09, 0.11, 0.13 (2), 0.19 (2), 0.21, 0.23, 0.27, 0.36 mg/kg. On a dry weight basis (88% DM) the values are: < 0.06 (5), 0.08, 0.10, 0.12, 0.15 (2), 0.22 (2), 0.24, 0.26, 0.31, 0.41 mg/kg. The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.14 mg/kg.

Barley straw

Barley trials were reported from France, Germany, and the UK. The GAPs are: 500 g ai/L SC, 0.15 kg ai/ha, two applications per season one application in France with a PHI of 56 days; 0.25 kg ai/ha, a application in Germany at growth stages BBCH 25–32 (tillering), and 500 g ai/L SC, 0.15 kg ai/ha, two applications until growth stage Zadoks 49 (about 60 days PHI) in the UK. The trials in the UK and in North France were evaluated against the GAP of Germany.

The residues in barley straw in rank order (n=6) are: 0.22 (2), 0.30, 0.58, 1.23, 2.94 mg/kg. On a dry weight basis (89% DM) the values are: 0.25 (2), 0.34, 0.65, 1.38, 3.30 mg/kg. The Meeting estimated a maximum residue level of 5 mg/kg and an STMR of 0.50 mg/kg. The highest residue is 3.3 mg/kg.

Hops (dry)

Hops trials were reported from Germany (GAP: 250 g/L SC, 0.011 kg ai/hL, four applications or 0.5 kg ai/ha/season, PHI 28 days) and from the USA (GAP: 250 g/L SC, 0.15 kg ai/ha, four applications or 0.6 kg ai/ha/season, PHI 21 days).

Six trials in Germany were conducted at the maximum seasonal GAP, but with three applications rather than four. The sum of the three applications was within 30% of the seasonal maximum GAP. The residue values in ranked order were: 0.03, 0.04, 0.07, 0.37, 0.41, and 0.55 mg/kg.

Four trials in the USA were conducted at the maximum season GAP, with three applications rather than four. The residue values in ranked order were: 0.39, 1.2, and 2.2 mg/kg.

The trials in the USA and in Germany are not from the same population. The three trials in the USA provide insufficient data for the estimation of an STMR and maximum residue level.

Using the six trials from Germany, the Meeting estimated and STMR of 0.22 mg/kg and a maximum residue level of 1 mg/kg for residues of quinoxyfen in hops (dry).

Spices

Using a default processing (dehydration) factor of 10, the Meeting estimated a maximum residue level of 10 mg/kg and an STMR of 1.5 mg/kg for dried chili peppers based on the maximum residue level and STMR of pepper

Sugar beet tops

Field trial data were received from Germany (GAP: 500 g ai/LC SC, 0.12 kg ai/ha, two applications per season, 28 day PHI), UK (GAP: 500 g ai/L SC, 0.15 kg ai/ha, two applications, 28 day PHI), and France (GAP: 500 g ai/L SC, 0.15 kg ai/ha, one application, 28 day PHI).

Three trials from Germany and four trials from the UK were conducted at the maximum GAP. The residue values from Germany in ranked order were: 0.10 (2) and 0.27 mg/kg. The residue values from the UK in ranked order are: 0.13, 0.22, 0.36, and 0.37 mg/kg. The combined values (n=7) in ranked order were: 0.10 (2), 0.13, 0.22, 0.27, 0.36, and 0.37 mg/kg.

The Meeting estimated an STMR of 0.22 mg/kg and a highest residue level of 0.37 mg/kg.

Fate of residues in storage

The effect of storage upon the fate of quinoxyfen residues was not reported to the Meeting.

Fate of residues during processing

Information on the fate of quinoxyfen in the processing of wheat, barley, and grapes was reported to the Meeting. No information was supplied on the fate of radiolabeled quinoxyfen under general processing conditions.

Winter wheat which had received foliar treatment with quinoxyfen was processed into flour and bread in separate studies in France and the UK. The wheat grain contained no residues (< 0.01 mg/kg) and while there was no apparent concentration of residue in bran, flour, or bread, no processing factors could be calculated.

Barley in the UK was treated at an exaggerated rate with quinoxyfen, and the grain at normal harvest was processed into malt and beer by a simulated commercial process. The processing factor for malt was 0.5 and that for beer was < 0.1. Using the STMR for barley (0.01 mg/kg), the Meeting estimated an STMR-P of 0.001 mg/kg for beer.

Processing studies for the conversion of grapes to wine were reported from France, Germany, and Italy. In all cases, the grapes had quantifiable field incurred residues of quinoxyfen. Three trials were conducted for the preparation of white wine and two for the preparation of red wine. The processing factor varied from 0.004 to 0.03, with a median and average value of 0.01. Applying this processing factor to the STMR of grapes (0.15 mg/kg), the Meeting estimated an STMR-P of 0.015 mg/kg for wine (from grapes).

A processing study for the conversion of grapes to raisins and grape juice was reported from the USA. Grapes with a quantifiable field incurred residue of quinoxyfen were processed in separate commercial-type procedures into raisins and pasteurised grape juice. The processing factors for raisins and juice were 0.66 and 0.06, respectively. Using the STMR value for grapes (0.15 mg/kg), the Meeting estimated STMR-Ps of 0.099 mg/kg and 0.009 mg/kg for raisins and grape juice, respectively.

Farm animal dietary burden

The Meeting estimated the dietary burden of quinoxyfen residues in farm animals on the basis of the diets listed in Appendix IX of the *FAO Manual*. Calculation from MRLs, highest residues and STMR-P values provides the levels in feed suitable for estimating MRLs for animal commodities, while calculation from STMR and STMR-P values for feed is suitable for estimating STMR values for animal commodities. The percentage of dry matter is taken as 100% when MRLs and STMR values are already expressed as dry weight.

Estimated maximum dietary burden of farm animals

Commodity	Group	Residue (mg/kg)	Basis of Residue	Dry matter (%)	Diets			Residue contribution (mg/kg)		
					Beef cattle	Dairy cattle	Poultry	Beef cattle	Dairy cattle	Poultry
Barley grain	GC	0.01	MRL	88	50	30	80	0.006	0.005	0.009
Sugar beet leaves (tops)	AV	0.37	HR	23	20	10	-	0.32	0.16	-
Barley Straw	AS	3.3	HR	89	10	60		0.33	1.98	
TOTAL					80	100	80	0.66	2.14	0.01

The calculated maximum dietary burdens for beef cattle, dairy cows and poultry are 0.66, 2.1, and 0.01 ppm, respectively.

Estimated STMR dietary burden of farm animals

Commodity	Group	Residue (mg/kg)	Basis of Residue	Dry matter (%)	Diets			Residue contribution (mg/kg)		
					Beef cattle	Dairy cattle	Poultry	Beef cattle	Dairy cattle	Poultry
Barley grain	GC	0.01	STMR	88	50	30	80	0.006	0.003	0.009
Sugar beet leaves (tops)	AV	0.22	STMR	23	20	10	-	0.19	0.10	-
Barley Straw	AS	0.50	STMR	89	10	60	-	0.05	0.30	
TOTAL					80	100	80	0.25	0.40	0.01

The calculated STMR dietary burdens for beef cattle, dairy cows and poultry are 0.25, 0.40, and 0.01 ppm, respectively.

Farm animal feeding studies

The Meeting received two feeding studies for dairy cattle and a radiolabeled quinoxyfen feeding study for poultry (chickens). Friesian cows were fed for 28 consecutive days with diets containing 0.2, 0.6, 2.0, or 20 ppm quinoxyfen. Residues in whole milk reached a plateau by day 7 of 0.001, 0.002, and 0.007 mg/kg for 0.2, 0.6, and 2.0 ppm dosing levels, respectively. At the 20 ppm dosing level, the quinoxyfen residue spiked to 0.37 mg/kg on day 7 and then declined to an apparent plateau of 0.16 ppm by the final day.

At the 0.2 ppm feeding level, the maximum and average (n=3 cows) residue in whole milk (day 27) was < 0.001 mg/kg. In cream, the maximum residue was 0.007 mg/kg and the average residue was 0.003 mg/kg. At the 0.6 ppm feeding level, the maximum and average (n=3 cows) in whole milk (day 27) was 0.002 mg/kg and 0.002 mg/kg, respectively. In cream, the maximum residue was 0.02 mg/kg, and the average residue was 0.015 mg/kg.

At the 0.2 ppm feeding level, the maximum and average residues in liver, kidney, muscle, and fat were ND and ND, < 0.01 and < 0.01 mg/kg, ND and ND, and 0.02 and 0.01 mg/kg, respectively. At the 0.6 feeding level, the maximum and average residues in liver, kidney, muscle, and fat were < 0.01 and < 0.01 mg/kg, < 0.01 and < 0.01 mg/kg, ND and ND, and 0.02 and 0.012 mg/kg, respectively.

At the 0.6 ppm feeding level, the maximum and average residues in milk were 0.002 mg/kg each. The maximum and average values in cream were 0.022 mg/kg and 0.016 mg/kg, respectively. The maximum and average residue values in liver and kidney were < 0.01 mg/kg each. The maximum and average values in muscle were ND (< 0.002 mg/kg). The maximum and average values in fat were 0.02 and 0.12 mg/kg, respectively.

At the 2 ppm feeding level, the maximum and average residues in milk were 0.010 and 0.0088 mg/kg, respectively. The maximum and average residues in cream were 0.077 and 0.068 mg/kg, respectively. The maximum and average residues in liver, kidney, and muscle were < 0.01 mg/kg. The maximum residue in fat was 0.10 mg/kg, and the average was 0.09 mg/kg.

Quinoxyfen total residues, mg/kg

Dietary burden (ppm)		Cream	Milk	Muscle		Liver		Kidney		Fat	
Feeding level [ppm]											
		Mean	Mean	Highest	Mean	Highest	Mean	Highest	Mean	Highest	Mean
MRL, beef cattle	(0.66)			(< 0.01)		(< 0.01)		(< 0.01)		(0.02)	
	[0.6]			[< 0.002]		[< 0.01]		[< 0.01]		[0.02]	
MRL, dairy cattle	(2.1)	(0.068)	(0.0088)								
	[2]	[0.068]	[0.0088]								
STMR beet cattle	(0.25)				(0.002)		(0.002)		(< 0.01)		(0.01)
	[0.2]				[< 0.002]		[< 0.002]		[< 0.01]		[0.01]
STMR dairy cattle	(0.40)	(0.01)	(0.002)								
	[0.2/0.6]	[0.003/0.016]	[< 0.001/0.002]								

A *poultry feeding* study consisted of four groups of 10 Isa Brown laying hens fed at the following dose levels: 0.1, 0.3, and 1.0 ppm of diet/ day. The hens were fed gelatin capsules containing a *radiolabelled* (mixture of ^{14}C-quinoline label and ^{14}C-phenoxy label) ^{14}C-quinoxyfen. Each daily dose was administered in a single capsule at the same time each day for 28 days. Only TRR was determined in the eggs and tissues. These levels were very low at a 0.1 ppm diet with maximum values of 0.003 mg/kg in eggs, 0.009 mg/kg in liver, 0.004 mg/kg in kidney, 0.0 mg/kg in muscle, and 0.013 mg/kg in fat. At the 1.0 ppm feeding level, TRR values were 0.025 mg/kg in eggs, 0.097 mg/kg in liver, 0.049 mg/kg in kidney, 0.009 mg/kg in muscle, and 0.063 mg/kg in fat. However, the TRR was not characterized or identified.

Animal commodity maximum residue levels

The Meeting estimated the following maximum residue levels for mammalian commodities, based on the cow feeding studies and the calculated dietary intake (see above): muscle, 0.01 (*) mg/kg; fat, 0.02 mg/kg; edible offal, 0.01 (*) mg/kg; milk fat, 0.2 mg/kg; milk, 0.01 mg/kg. The Meeting likewise estimated the following STMR values: muscle, 0.002 mg/kg; fat, 0.01 mg/kg; edible offal 0.01 mg/kg; milk fat, 0.02 mg/kg; milk, 0.002 mg/kg. The milk fat estimations assume that cream is 50% fat. Although the metabolic profile in poultry was not determined, the feeding study with radiolababelled quinoxyfen demonstrated very low levels of total residue at a feeding level of 0.1 ppm, the estimated dietary burden of poultry. Therefore, the MRLs for poultry commodities are estimated at the LOQs of the analytical method, 0.01 (*) mg/kg for each of poultry egg and edible offal, and 0.02 mg/kg meat (fat). The STMRs are based on the TRR values and are estimated to be: eggs, 0.003 mg/kg; offal, 0.009 mg/kg; muscle, 0 mg/kg; and fat 0.013 mg/kg.

RECOMMENDATIONS

The Meeting estimated the maximum residue levels and STMR values shown below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue: Plant and animal commodities.

Definition of the residue (for compliance with MRL and estimation of dietary intake): *quinoxyfen.*

The residue is fat soluble.

CCN	Commodity Name	MRL, mg/kg		STMR or STMR-P, mg/kg	HR, mg/kg
		New	Previous		
GC0640	Barley	0.01 (*)		0.01	
	Beer			0.001	
FS0013	Cherries	0.4		0.12	0.27
FB0278	Currants, black	1		0.20	0.40
DF0269	Dried grapes (=Currant, Raisins and Sultanas)			0.099	
MO105	Edible offal (mammalian)	0.01 (*)		0.01	
PE0112	Eggs	0.01 (*)		0.003	
FB0269	Grapes	2		0.13	1.1
JF0269	Grape juice			0.009	
DH1100	Hops, dry	1		0.22	
VL0482	Lettuce, head	8		1.4	5.3
VL0483	Lettuce, leaf	20		3.8	13
MM0095	Meat (from mammals other than marine mammals)	0.01 * (muscle) 0.02 (fat)		0.01 fat 0.002 muscle	
VC0046	Melons, except watermelon	0.1		0.01	0.02
ML0106	Milks (excl. processed products)	0.01		0.002	
FM0183	Milk fats	0.2		0.02	
VO0051	Peppers	1		0.15	0.64
?	Peppers, chili, dried	10		1.5	
PO 0111	Poultry, edible offal of	0.01 (*)		0.009	
PM 0110	Poultry meat	0.02		0.013 fat 0.0 muscle	
FB0275	Strawberry	1		0.32	0.56
VR0596	Sugar beet root	0.03		0.01	
AV1051	Sugar beet leaves or tops			0.22	
GC0654	Wheat	0.01 (*)		0.01	
	Wine of grapes			0.015	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDI) of quinoxyfen, based on the STMRs estimated for 15 commodities for the thirteen GEMS/Food cluster diets were in the range of 0% to 1% of the ADI (Annex 3 of the 2006 JMPR Report). The Meeting concluded that the long-term intake of residues of quinoxyfen resulting from its uses that have been considered by JMPR is unlikely to present a public health concern.

Short-term intake

The 2005 JMPR decided that an acute RfD is unnecessary. The Meeting therefore concluded that the short-term intake of quinoxyfen residues is unlikely to present a public health concern.

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Gambie, A.	31261	1995g
Gambie, A.	31262	1995h
Gambie, A.	31263	1995i
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THIABENDAZOLE (065)

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EXPLANATION

Thiabendazole was evaluated several times by JMPR in the period 1970-1981. The 1997 JMPR reviewed it under the CCPR Periodic Review Programme and proposed withdrawal of the existing CXL for citrus fruit of 10 mg/kg. The residue definition agreed for compliance with MRLs and estimation of dietary intake for plant commodities is thiabendazole; that for compliance with MRLs for animal commodities is the sum of thiabendazole and 5-hydroxythiabendazole. For estimation of dietary intake for animal commodities the definition is the sum of thiabendazole, 5-hydroxythiabendazole and its sulfate conjugate.

The 2000 JMPR received new data from trials on oranges and mandarins carried out in Spain in 1998, on the basis of which an MRL of 3 mg/kg was proposed. At the 35th CCPR meeting, the delegation from Morocco indicated that the proposed MRL for thiabendazole on citrus of 3 mg/kg would need to be increased to support that country's use pattern. At that meeting the delegation of Morocco was invited to submit data to JMPR. At the 36th CCPR meeting the proposed Codex MRL for citrus was returned to Step 6 pending receival of the Moroccan data. Residue trials data to support the use of thiabendazole as a post-harvest treatment on citrus fruits (oranges and mandarins) in Morocco, generated by Morocco Institutions in 2003 and 2004, were submitted in early 2006 for evaluation.

RESIDUE ANALYSIS

Two methods from the open literature were used to analyse the samples from the supervised trials conducted in Morocco. The first was a spectroscopic method with UV detection (Gneagi *et al*., 1974). Under this method the sample was homogenised using ethyl acetate, anhydrous sodium sulfate and aqueous sodium hydroxide. Then, the homogenate was filtered, and an aliquot part was separated from the ethyl acetate extract with a hydrochloric acid solution. The aqueous phase was made alkaline, then extracted with ethyl acetate and the absorbance of the organic layer was measured at 302 nm. The method was validated for thiabendazole recovery only for whole fruits at 0.1, 0.5 and 1 mg/kg fortification levels (one fortified sample at each fortification level) and was found to yield 97–102% recovery in 2003 and 96–99% in 2004. The limit of quantification (LOQ) achieved was 0.1 mg/kg.

The second method, which was used in 2004, was an HPLC method, based on the CEN method EN 14333-3. The extraction and clean up procedure was the same as for the UV method, the difference being that the residue was determined by HPLC reversed phase chromatography with UV (285 nm) and fluorescence detection (emission 385 nm; excitation 315 nm). The recoveries for thiabendazole were tested only for whole fruits at one fortification level (0.1 mg/kg, 3 replicate fortified samples) and were found to be in the range 82–84%, with the latter method providing an improved LOQ (0.01 mg/kg).

USE PATTERN

Table 1 shows the registered use of thiabendazole on citrus in Morocco, as indicated on the official label.

Table 1. Registered post-harvest uses of thiabendazole on citrus in Morocco.

Country	Form	Application			Waiting or withholding period (days)
		Method	Concentration kg ai/hL wax	Number	
Morocco	SC 500 g/L	In mixture with wax	0.375	1	Not stated

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CITRUS

The Meeting received information on supervised residue trials of thiabendazole carried out in Morocco with post-harvest application to:

Oranges (Table 2)
Clementine mandarins (Table 3)

The trials were conducted in 2003 and 2004. Thiabendazole, formulated as a 500 SC, was applied post-harvest on oranges or clementine mandarins as a spray application mixed with wax at the rate of 0.375 kg ai/hL, i.e., according to Moroccan GAP.

In 2003 trials were carried out with several varieties of oranges. Treated fruit was stored at 4–6 ^{o}C and samples were taken < 2 days, 3, 4, 5, 11 or 15 days after application. Whole orange fruits were analysed immediately after sampling. The UV spectroscopic analytical method, reported previously was used.

In 2004 the product was applied to oranges and mandarins. Fruits were stored at 4-6^{0}C and samples were collected < 2, 7 and 15 days after application. Peel and pulp were analysed separately and the residues in whole fruits were calculated on the basis of results for peel and pulp and relative weights. Some of the samples were analysed using the spectroscopic method, while others were analysed using the HPLC method.

One or more samples were analysed at each time interval. Since they were replicate samples, the mean values are reported. No correction for recovery was applied to the analytical results. Control samples were also analysed and have shown no interference or contamination. Residues used for MRL derivation are double-underlined in Tables 2 and 3.

Table 2. Thiabendazole residues in oranges following post-harvest treatment in Morocco.

Country Location/ Variety	Year	Application			Days after Treatment	Residues in whole fruit (mg/kg)	Author Year (Trial No.)
		Product	kg ai/hL	Type/ Number of treatments			
Morocco Casablanca/ Navel	2004	500SC	0.375	Spray with wax/1	< 2 7 15	5.2 3.2 1.6	Benzine and Tarhy 2004 (0951)
Morocco Casablanca/ Navel	2004	500SC	0.375	Spray with wax/1	< 2 7 15	3.8 2.1 2.7	Benzine and Tarhy 2004 (0951)
Morocco Agadir/ Salustianas	2003	500SC	0.375	Spray with wax/1	6	4.2	Benzine and Tarhy 2003 (0952)

Country Location/ Variety	Year	Application			Days after Treatment	Residues in whole fruit (mg/kg)	Author Year (Trial No.)
		Product	kg ai/hL	Type/ Number of treatments			
Morocco Agadir/ Salustianas	2003	500SC	0.375	Spray with wax/1	< 2	3.3[1]	Benzine and Tarhy 2003 (0952)
Morocco Agadir/ Ortanique	2003	500SC	0.375	Spray with wax/1	3	2.5[2]	Benzine and Tarhy 2003 (0952)
Morocco Agadir/ Orantique Sanguine	2003	500SC	0.375	Spray with wax/1	3	1.6[2]	Benzine and Tarhy 2003 (0952)
Morocco Casablanca/ Sanguine	2003	500SC	0.375	Spray with wax/1	7	3.4[3]	Benzine and Tarhy 2003 (0952)
Morocco Agadir/ Salustianas	2003	500SC	0.375	Spray with wax/1	3-4	2.2 [3]	Benzine and Tarhy Morocco 2003 (0952)
Morocco Agadir/ Salustianas	2003	500SC	0.375	Spray with wax/1	5	1.6	Benzine and Tarhy Morocco 2003 (0952)
Morocco Casablanca/ Sanguine	2003	500SC	0.375	Spray with wax/1	11-13	3.4 [2]	Benzine and Tarhy Morocco 2003 (0952)
Morocco Casablanca/ Sanguine	2003	500SC	0.375	Spray with wax/1	11-13	4[4]	Benzine and Tarhy Morocco 2003 (0952)
Morocco Agadir/ Salustianas	2003	500SC	0.375	Spray with wax/1	3	1.8[3]	Benzine and Tarhy Morocco 2003 (0952)
Morocco Agadir/ Salustianas	2003	500SC	0.375	Spray with wax/1	4	1.8	Benzine and Tarhy Morocco 2003 (0952)
Morocco Casablanca/ Sanguine	2003	500SC	0.375	Spray with wax/1	< 2	2.1 [3]	Benzine and Tarhy Morocco 2003 (0952)
Morocco Casablanca/ M. Late	2003	500SC	0.375	Spray with wax/1	3	3.3[5]	Benzine and Tarhy Morocco 2003 (0952)

[1]: mean of 3 replicate samples
[2]: mean of 2 replicate samples
[3]: mean of 4 replicate samples
[4]: mean of 6 replicate samples
[5]: mean of 8 replicate samples

Table 3. Thiabendazole residues in mandarins following post-harvest treatment in Morocco.

Country Location/ Variety	Year	Application			Days after Treatment	Residues in whole fruit [mg/kg]	Author Year Trial No.
		Product	kg ai/hL	Type/Number of treatments			
Morocco Casablanca/ Clementine	2004	500SC	0.375	Spray with wax/1	< 2 7 15	3.5[1] 1.8[1] 1[1]	Benzine and Tarhy 2004 (0951)
Morocco Agadir/ Clementine	2004	500SC	0.375	Spray/1	< 2 7 15	2.7 2.6 1.3	Benzine and Tarhy 2004 (0951)
Morocco Berkane/ Clementine	2004	500SC	0.375	Spray/1	< 2 7 15	2.7[2] 2.3[2] 1.4[2]	Benzine and Tarhy 2004 (0951)
Morocco Casablanca/ Clementine	2004	500SC	0.375	Spray/1	7 15	2.9 3.5	Benzine and Tarhy 2004 (0951)
Morocco Berkane/ Clementine	2004	500SC	0.375	Spray/1	< 2 7 15	2.7[2] 2.3[2] 2.[2]	Benzine and Tarhy 2004 (0951)
Morocco Casablanca/ Clementine	2004-	500SC	0.375	Spray/1	< 2 7 15	1.3[3] 1.3[3] 1[3]	Benzine and Tarhy 2004 (0951)
Morocco Berkane/ Clementine	2004	500SC	0.375	Spray/1	< 2 7 15	2.8[1] 2.7[1] 2.1[1]	Benzine and Tarhy 2004 (0951)
Morocco Agadir/ Clementine	2004	500SC	0.375	Spray/1	< 2	2.4[3]	Benzine and Tarhy 2004 (0951)

[1]: mean of 2 replicate samples
[2]: mean of 3 replicate samples
[3]: mean of 4 replicate samples

FATE OF RESIDUES IN STORAGE AND PROCESSING

In commercial cold storage

Treated fruit were taken and analysed immediately after application (0 days) and after periods of 7 or 15 days in cold storage (4-6 °C), as per common commercial practice. This data shows that there is no significant decrease in residues within the first 7 days of storage, while some degradation occurred after 15 days of storage (Tables 3 and 4). However, given the variability in results and the fact that the reproducibility of the analytical methods was not assessed, no reliable conclusion about storage stability can be drawn. Data evaluated by the 2000 JMPR (Spanish trials) showed that the residues in oranges and mandarins remained stable or decreased slightly during storage up to 15 days.

In processing

The 2000 JMPR estimated processing factors from whole fruit to pasteurised juice. The best estimate from two studies, two replicate samples for each study, was 0.14. It also estimated a processing factor of 5.7 from whole fruits to dry citrus pomace (dried citrus pulp).

RESIDUES IN THE EDIBLE PORTION

Table 4 provides information on the distribution of residues between edible (pulp) and non-edible parts (peel). Values used for the estimation of the distribution factor are underlined.

Table 4. Distribution of residues between peel and pulp of citrus treated at 0.375 kg ai/hL.

Citrus Fruits –Trial No 0951/T04	Residue (mg/kg)				Distribution Factor (pulp)[4]
	DAT[1]	Whole fruit[2]	Peel	Pulp[3]	
Orange	< 2 7 15	5. 2 3.2 1.6	18 11 9.4	< 0.1 < 0.1 < 0.1	
Orange	< 2 7 15	<u>3.8</u> <u>2.1</u> <u>2.7</u>	10.3 7.6 4.9	<u>0.84</u> <u>0.34</u> <u>0.49</u>	0.21 0.15 0.17
Mandarin-	< 2 7 15	1.5 0.50 0.40	4.5 3.4 2.4	< 0.1 < 0.1 < 0.1	
Mandarin-	< 2 7 15	5.5 3.2 1.7	17 13 11	< 0.1 < 0.1 < 0.1	
Mandarin-	< 2 7 15	2.7 2.6 1.3	18 16 5.7	< 0.1 < 0.1 < 0.1	
Mandarin-	< 2 7 15	2.7 2.6 1.3	13 11 7.4	< 0.1 < 0.1 < 0.1	
Mandarin-	< 2 7 15	2.7 2.6 1.5	16 12 6.8	< 0.1 < 0.1 < 0.1	
Mandarin-	< 2 7 15	2.6 1.5 1.5	11 7 7	< 0.1 < 0.1 < 0.1	
Mandarin-	7 15	<u>2.9</u> <u>3.5</u>	8.4 7.5	<u>0.22</u> <u>0.37</u>	0.07 0.10
Mandarin-	< 2 7 15	2.2 2.2 2.2	12 11 8	< 0.1 < 0.1 < 0.1	
Mandarin-	< 2 7 15	2.5 1.7 1.6	13 8 8	< 0.1 < 0.1 < 0.1	
Mandarin-	< 2 7 15	3.4 3 2.4	18 15 11	< 0.1 < 0.1 < 0.1	
Mandarin-	< 2 7 15	0.24 0.11 <u>0.16</u>	0.86 0.4 0.5	< 0.01 < 0.01 <u>0.03</u>	
Mandarin-	< 2 7 15	<u>0.46</u> 0.30 0.33	2.05 0.94 1.2	<u>0.04</u> < 0.01 < 0.01	0.08
Mandarin-	< 2 7 15	<u>0.35</u> <u>0.24</u> 0.10	1.3 0.50 0.65	<u>0.09</u> <u>0.04</u> < 0.01	0.25 0.16
Mandarin-	< 2 7 15	<u>4.2</u> <u>4.5</u> <u>3.4</u>	15 8.8 8.3	<u>0.05</u> <u>0.07</u> <u>0.12</u>	0.01 0.01 0.03

Citrus Fruits –Trial No 0951/T04	Residue (mg/kg)				Distribution Factor (pulp)[4]
	DAT[1]	Whole fruit[2]	Peel	Pulp[3]	
Mandarin-	< 2 7 15	3.2 3.2 2.7	17 16 12	< 0.1 < 0.1 < 0.1	
Mandarin-	< 2 7 15	2.4 3.1 1.5	12 9.3 7	< 0.1 < 0.1 < 0.1	
Median Distribution Factor					0.1

[1] Days after treatment

[2] Results calculated from residues in pulp and peel

[3] Values <LOQ are not considered for estimation of the median DF.

[4] Calculated by dividing the residues measured in the pulp by the residues measured in whole fruit

RESIDUES IN ANIMAL COMMODITIES

The dietary burden of thiabendazole residues in farm animals was estimated by the 2000 JMPR on the basis of the data then evaluated (lower GAP compared with that of Morocco). New estimates were made using the highest residue found in whole citrus fruits from the Moroccan trials. These have shown that the additional intake resulting from the Moroccan GAP was 2% for beef cattle and 4 % for dairy cattle; and 2% and 3% respectively when using the median residue from whole fruits. This shows that the additional intake is very low compared with the intake from wet potato pomace, which is the main driver for estimation of animal burden.

APPRAISAL

Thiabendazole is authorised as a post-harvest fungicide on citrus in many countries. It was evaluated several times by JMPR in the period 1970-1981. The 1997 JMPR reviewed it under the CCPR Periodic Review Programme and proposed withdrawal of the existing CXL for citrus fruits of 10 mg/kg. The residue definition, agreed for compliance with MRLs and estimation of dietary intake for plant commodities, is thiabendazole; that for compliance with MRLs for animal commodities is the sum of thiabendazole and 5-hydroxythiabendazole. For estimation of dietary intake for animal commodities it is the sum of thiabendazole, 5-hydroxythiabendazole and its sulfate conjugate. The JMPR 2000 received new data from Spain on the basis of which an MRL of 3 mg/kg was proposed. At the CCPR meeting in 2003, the delegation from Morocco had commented that the proposed MRL for thiabendazole on citrus fruits of 3 mg/kg would need to be increased to support the country's use pattern. At that meeting the delegation of Morocco was invited to submit data to JMPR. During the CCPR meeting in April 2004 the MRL for citrus fruits was returned to Step 6 pending receival of the data.

The current Meeting received data from residue trials to support the uses of thiabendazole as a post-harvest treatment on citrus fruits in Morocco.

Methods of analysis

For the analysis of the samples from supervised trials conducted in Morocco two methods from the open literature were used. The first one is an old spectroscopic method with UV detection developed in France in 1974. Limited validation was carried out (on whole fruits at 0.1, 0.5 and 1 mg/kg fortification levels, one fortified sample at each fortification level). The recoveries yielded were 96–102%, with an LOQ of 0.1 mg/kg.

The second method was a reversed phase HPLC method (standard CEN method). The recoveries for thiabendazole were tested only for whole fruits at one fortification level (0.1 mg/kg, 3 replicate fortified samples) and were found to be in the range of 82–84%. The LOQ was estimated to be 0.01 mg/kg.

Results of supervised trials on crops

Citrus fruits

The Meeting received, for evaluation, post-harvest application data from trials conducted in Morocco during 2003 and 2004. Thiabendazole, formulated as 500 SC, was applied to oranges (15 trials) and clementine mandarins (eight trials) according to the nationally authorised use pattern (GAP). This consisted of a spray application, mixed with wax, at a rate of 0.375 kg ai/hL. Treated fruit samples were stored at 4–6°C from harvest to analysis. Samples were taken for analysis < 2 days, 3, 4, 5, 11 or 15 days after application. Whole orange samples were analysed in 2003, while in 2004 peel and pulp were analysed separately.

The residue levels in whole oranges treated according to Moroccan GAP, in ranked order, were: 1.6 (2), 1.8 (2), 2.1, 2.2, 2.5, 3.3 (2), 3.4 (2), 3.8, 4, 4.2 and 5.2 mg/kg.

Those in orange pulp were: < 0.1 and 0.84 mg/kg.

The residue levels in whole mandarins treated according to Moroccan GAP, in ranked order, were: 1.3, 2.4, 2.7 (3), 2.8, and 3.5 (2) mg/kg.

Those in mandarin pulp were: 0.03, 0.04, 0.09, < 0.1 (11), 0.12, and 0.37 mg/kg.

The Meeting agreed to combine the data for whole oranges and mandarins to provide a data set for whole citrus fruit. The combined citrus fruit data set (23 values), in ranked order were: 1.3, 1.6 (2), 1.8 (2) 2.1, 2.2, 2.4, 2.5, 2.7 (3), 2.8, 2.9, 3.3 (2), 3.4, (2), 3.5, 3.8, 4, 4.2, and 5.2 mg/kg. Residue levels in citrus pulp, in ranked order were: 0.03, 0.04, 0.09, < 0.1 (12), 0.12, 0.37 and 0.84 mg/kg.

On the basis of the trials carried out according to the Moroccan GAP the Meeting estimated a maximum residue level of 5 mg/kg for thiabendazole in citrus, replacing the previous recommendation of 3 mg/kg. The Meeting also estimated a median and a highest residue for whole fruits of 2.7 and 5.2 mg/kg respectively, for use in the calculation of the farm animal dietary burden. The Meeting recommended an STMR of 0.1 mg/kg and a HR of 0.84 mg/kg for citrus fruits.

Fate of residues in edible portion

The results from the 2004 trials provide information about distribution of residues between peel and pulp. These show that the majority of the residue remains on the peel. In only a few cases detectable residues of 0.03–0.84 mg/kg were found in the pulp. However, it should be noted that the sensitivity of the analytical method used on the majority of the samples was unsatisfactory (LOQ = 0.1 mg/kg). The distribution factors (DF) for residues into pulp ranged between 0.01 and 0.25, with a median DF of 0.1.

Farm animal dietary burden

The Meeting noted that the additional animal dietary burden, from residues in citrus treated according to the Morocco GAP, would be insignificant when compared to the contribution from wet potato peel as calculated by the 2000 JMPR. Thiabendazole accounted for 2% of the total dietary burden for beef cattle and 4% for dairy cattle when using the highest residue, and 2% and 3% respectively, when using the median from whole fruits.

RECOMMENDATIONS

On the basis of the data from supervised trials with post-harvest application of thiabendazole on citrus fruits (oranges and clementine mandarins) in Morocco carried out according to the GAP, the Meeting concluded that a residue concentration below is suitable for establishing a Codex Maximum Residue Limit and for assessing dietary intake.

Definition of the residue (for compliance with MRLs and for estimation of dietary intake): for plant commodities: *Thiabendazole.*

For compliance with MRLs for animal commodities: *Sum of thiabendazole and 5-hydroxythiabendazole.*

For estimation of dietary intake for animal commodities: *Sum of thiabendazole, 5-hydroxythiabendazole and its sulfate conjugate.*

Summary of recommendations for MRLs, STMRs and HRs for imidacloprid

CCN	Name	MRL, mg/kg		STMR mg/kg	HR mg/kg
		New	Previous		
FC0001	Citrus fruits	5 Po	3 Po	0.1	0.84

DIETARY RISK ASSESSMENT

Long-term intake

The IEDI of thiabendazole was estimated for the 13 cluster diets using the STMRs estimated by a previous JMPR for avocado, cattle kidney, cattle liver, cattle meat, cattle milk, mango, melon, papaya, pome fruit, potato and strawberry and the STMR for citrus fruits estimated by the current JMPR. The maximum ADI established in 1997 is 0.1 mg/kg bw and the calculated IEDIs were 2–20% of this ADI (See Annex 3 of the 2006 Report). The Meeting concluded that the intake of residues of thiabendazole resulting from the uses considered by a previous JMPR and by the current JMPR was unlikely to present a public health concern.

Short-term intake

The IESTIs of thiabendazole by general population and by children was calculated for commodities for which STMRs or HRs was estimated by the 2000 Meeting and the current Meeting (Annex 4 of 2006 Report). The current JMPR estimated two ARfDs for thiabendazole: one for general population; and the other for women of child-bearing age. The ARfD for general population is 1 mg/kg bw and the calculated IESTIs for children up to 6 years range from 0 to 60% and those for general population from 0 to 20% of this ARfD. The ARfD for women of child-bearing age is 0.3 mg/kg bw and the calculated IESTIs for women of child-bearing age range from 0 to 70% of this ARfD. The Meeting concluded that the short-term intake of residues of thiabendazole resulting from the use considered by the current JMPR is unlikely to present a public health concern.

REFERENCES

Author, Date, Title, Institute & report number, Submitting manufacturer and report number, GLP/Non-GLP.

Benzine, M. and M. Tarhy 2005. Residues of Thiabendazole in and on Citrus fruits from Post Harvest Applications in Supervised Trials in Morocco During 2004. EACCE and LOARC Morocco. File No. MK360/0951, Report Number 001/04, non GLP

Benzine, M. and M. Tarhy. 2003. Residues of Thiabendazole in and on Citrus fruits from Post Harvest Applications in Supervised Trials in Morocco During 2003. EACCE and LOARC Morocco. File No. MK360/0952, Report Number 002/03, non GLP

Gnaegi, F.; R. Mestres; J. Tourte, and M.Campo. 1974. Determination of benzimidazole and thiophanate fungicide residues in grapes, grape juice, and wine, and, in general, in fruits and vegetables. Travaux de la Societe de Pharmacie de Montpellier, 34(1), 91-100. CODEN: TSPMA6 ISSN: 0037-9115.

DIN EN 2001. Non-fatty foods-Determination of benzimidazole fungicides carbendazim, thiabendazole and benomyl (as carbendazim)-Part3: HPLC method with liquid/liquid clean up. EN 14333-3 (2001-11)

THIACLOPRID (223)

The first draft was prepared by Mr. Christian Sieke, Federal Institute for Risk Assessment, Germany

EXPLANATION

Residue and analytical aspects of thiacloprid were considered for the first time by the present meeting. The manufacturer submitted studies on metabolism, analytical methods, supervised field trials, processing, freezer storage stability, environmental fate in soil and rotational crop residues.

IDENTITY

Common name: Thiacloprid

Chemical name:

IUPAC: N-{3-[(6-Chloro-3-pyridinyl)methyl]-1,3-thiazolan-2-yliden}cyanamide

CA (index): Cyanamide, [3-[(6-chloro-3-pyridinyl)methyl]-2-thiazolidinylidene]-

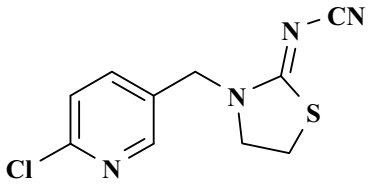
Manufacturer's code number: YRC 2894

CAS number: 111988-49-9

CIPAC number: not allocated

Molecular formula: $C_{10}H_9ClN_4S$

Structural formula:



Molecular mass: 252.73 g/mol

Formulations:

Formulation	Content of active ingredients	Trade names
SC 480	480 g/L Thiacloprid	Calypso
SC 240	240 g/L Thiacloprid	Calypso
OD 240	240 g/L Thiacloprid	Biscaya

PHYSICAL AND CHEMICAL PROPERTIES

A detailed chemical and physical characterisation of the active ingredient is given in Table 1.

References to test materials used:

1 Thiacloprid (batch 941013ELB01, purity 99.3%)

2 Thiacloprid (batch 950614ELB02, purity 99.7%)

3 Thiacloprid (batch 940629ELB04, purity 98.6%)

4 [methylene-^{14}C] thiacloprid, radiochemical purity > 98%, specific radioactivity 3.43 MBq/mg

Table 1. Physical and chemical data of thiacloprid.

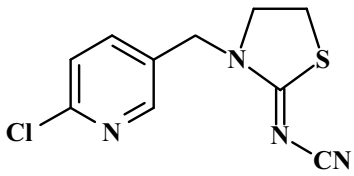
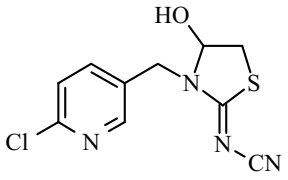
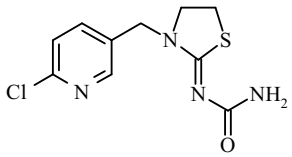
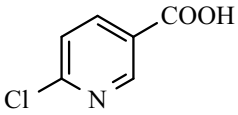
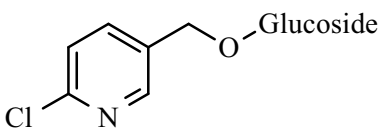
Property	Results	Test Material, Method	Reference
Physical state, colour	Active substance, pure: yellowish powder of crystals Active substance as manufactured: yellowish crystalline powder	Material 1 Technical ai	Krohn, J. 1996
Odour	Active substance, pure: no characteristic odour Active substance as manufactured: weak characteristic odour	Material 1 Technical ai	Krohn, J. 1996
Melting point	136 °C A second modification has a melting point of 128°C.	Material 1 EU A.1.	Krohn, J. 1996 Goehrt, A. 1995
Density	1.46 g/cm³ at 20°C	Material 1, OECD 109	Krohn, J. 1996
Vapour pressure	$1.61 \cdot 10^{-8}$ to $4.50 \cdot 10^{-8}$ Pa at 50°C $1.21 \cdot 10^{-7}$ to $1.61 \cdot 10^{-7}$ Pa at 60°C $1.68 \cdot 10^{-7}$ to $6.31 \cdot 10^{-7}$ Pa at 70 °C $3 \cdot 10^{-10}$ Pa at 20 °C (extrapolated) $8 \cdot 10^{-10}$ Pa at 25 °C (extrapolated)	Material 2, OECD 104 ≅ EU A.4	Krohn, J. 1996
Volatility	Henry's law constant at 20°C (calculated): 5×10^{-10} Pa × m³ ×·mol^{-1}		Krohn, J. 1996
Solubility in water	0.185 g/L at 20°C The solubility is not influenced by the pH in the range between pH 4 and pH 9.	Material 1, OECD 105 ≅ EU A.6.	Krohn, J. 1996
Solubility in organic solvents (at 20 °C, in g/L)	n-heptane < 0.1 g/L at 20°C xylene 0.30 g/L at 20°C 1-octanol 1.4 g/L at 20°C 2-propanol 3.0 g/L at 20°C ethyl acetate 9.4 g/L at 20°C polyethylen glycol (PEG) 42 g/L at 20°C acetonitrile 52 g/L at 20°C acetone 64 g/L at 20°C dichloromethane 160 g/L at 20°C dimethylsulfoxide 150 g/L at 20°C	Material 3, CIPAC MT 157, part 2	Krohn, J. 1996
Dissociation constant	Thiacloprid has no acidic or basic properties in aqueous solutions. It is not possible to specify dissociation constants of the active substance in water.	Material 1, OECD 112	Krohn, J. 1996
Partition coefficient n-octanol/ water	P_{OW} = 18 log P_{OW} = 1.26 at 20°C The effect of pH (4-9) was not investigated because there is no influence of pH on the water solubility.	Material 1, OECD 107 ≅ EU A.8	Krohn, J. 1996
Hydrolysis rate	Thiacloprid is stable at pH 5, 7 and 9. Under the experimental conditions the test substance was recovered from solution at content levels throughout the experiment (95-98% of applied). In the pH range tested formation of hydrolysis products was only observed at pH 9 at amounts less than 2% of the applied radioactivity. Considering the hydrolytic stability determined under environmental pH and temperature conditions it is not expected that hydrolytic processes will contribute to the degradation of thiacloprid in the environment.	Material 4, EPA 161-1	Brumhard, B. 1998

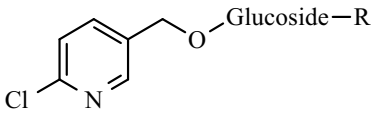
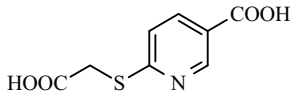
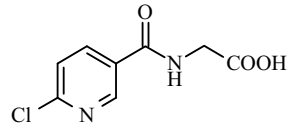
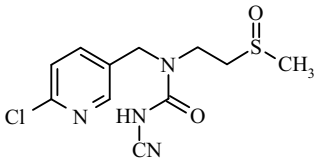
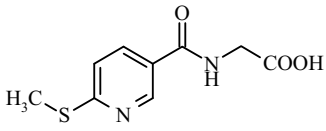
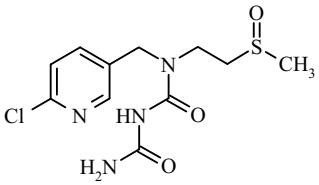
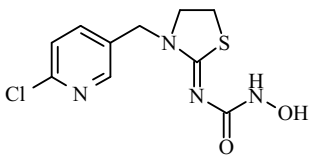
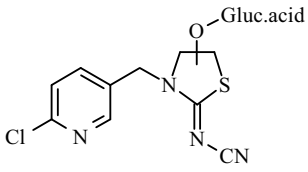
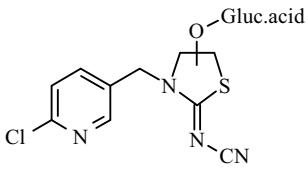
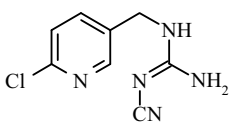
Property	Results	Test Material, Method	Reference
Photochemical degradation	Under the experimental conditions used thiacloprid degraded very slowly with an experimental half life of 79.7 days. Recovery ranged from 100.8 to 107.4% of the applied radioactivity. One main photoproduct (WAK 7259 A) was observed during the course of the experiment and accounted for a maximum of about 5% of the applied radioactivity. There was no degradation observed in the dark control samples. Considering the slow photolytic breakdown determined under environmental pH and temperature conditions it is expected that photolytic processes in aqueous solutions will contribute to the degradation of thiacloprid in the environment only to a very limited extent.	Material 4, EPA 161-2	Henneböle + Bornatsch, 1998

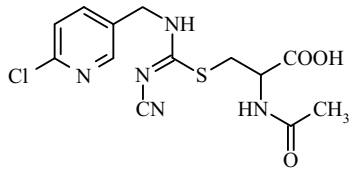
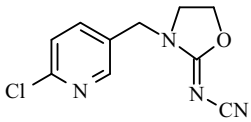
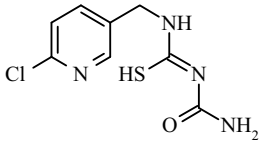
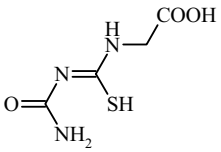
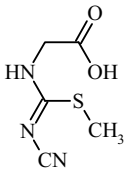
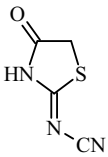
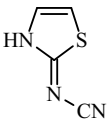
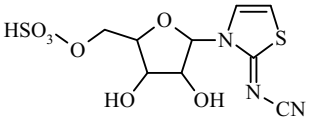
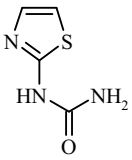
METABOLISM AND ENVIRONMENTAL FATE

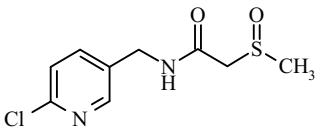
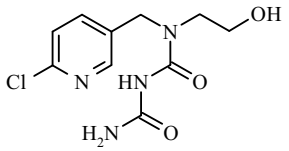
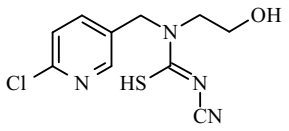
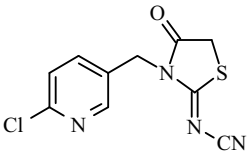
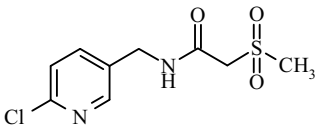
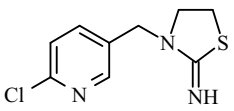
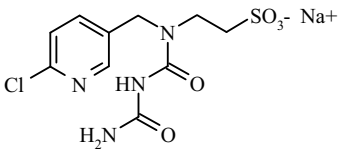
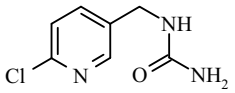
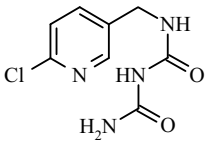
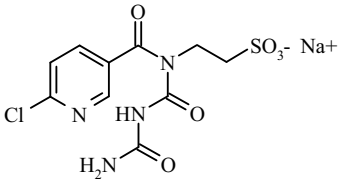
Chemical names, structures and code names of metabolites and degradation products of thiacloprid are shown below.

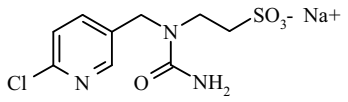
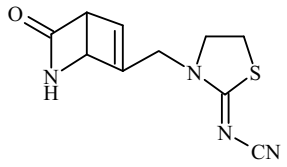
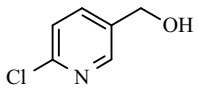
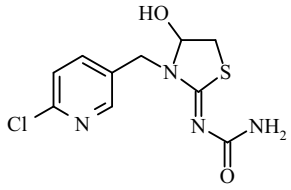
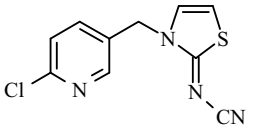
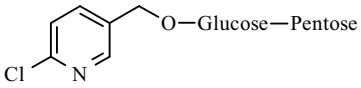
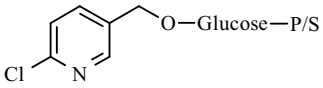
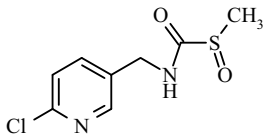
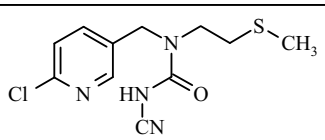
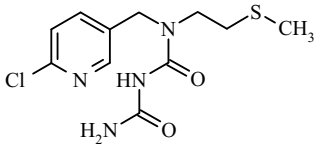
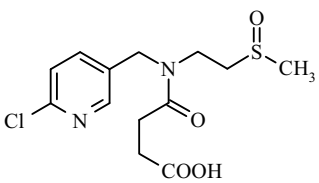
List of Metabolites – sorted by chemical structures

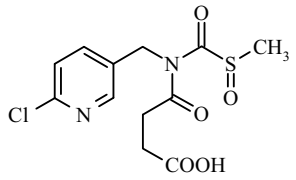
Structure	Name	Occurrence
	AS, Thiacloprid YRC 2894, PIZ 1264, ECW 10874, THS 4432, Ja752-J *{3-[(6-chloro-3-pyridinyl)methyl]-2-thiazolidinylidene}cyanamide*	Apple, tomato, cotton Goat, hen, rat Soil
	M01 4-hydroxy-thiacloprid, WAK 6856, PIZ 1265, KNO 1863, FHW 0106E, G1 *{3-[(6-chloro-3-pyridinyl)methyl]-4-hydroxy-2-thiazolidinylidene}cyanamide*	Apple, tomato, cotton Goat, hen, rat
	M02 thiacloprid-amide, KKO 2254, Ja752-A, FHW 0104D *{3-[(6-chloro-3-pyridinyl)methyl]-2-thiazolidinylidene}urea*	Apple, cotton Soil Rotational crops Soil (by photolysis)
	M03 6-CNA, BNF5518A, Ja752-K, G6 *6-chloro-3-pyridinecarboxylic acid*	Tomato, cotton Goat, hen, rat Soil Rotational crops
	M04 6-CPA-glucoside, G4 *Glucoside of 6-chloro-3-pyridine-methanol*	Tomato, cotton

Structure	Name	Occurrence
	M05 6-CPA-complex glucoside *Complex glucoside of 6-chloro-3-pyridi-ne-methanol*	Tomato
	M06 6-CMT-nicotinic acid *6-[(carboxymethyl)thio]-3-pyridine-carboxylic acid*	Rat
	M07 6-CN-glycine, WAK 3583 *N-[(6-chloro-3-pyridinyl)-carbonyl]glycine*	Goat, hen, rat
	M08 6-CP-urea sulfoxide, KNO 2672 *N-[(6-chloro-3-pyridinyl)methyl]-N'-cyano-N-[2-(methylsulfinyl)ethyl]urea*	Goat, hen, rat
	M09 KNO 1889 *N-{[6-(methylthio)-3-pyridinyl]-carbonyl}glycine*	Hen, rat
	M10 6-CP-biuret sulfoxide, KNO 1891, KNO 1873B N-[(6-chloro-3-pyridinyl)methyl]-N-N-[2-(methylsulfinyl)ethyl]iminodi carbonic diamide	Goat, hen, rat
	M11 thiacloprid-hydroxylamide, KNO 1893 N-{3-[(6-chloro-3-pyridinyl)methyl]-2-thiazolidinylidene}-N'-hydroxyurea	Goat, hen, rat
	M12 KNO 2621, PIZ 1270 Glucuronic acid conjugate of {3-[(6-chloro-3-pyridinyl)methyl]-4(or 5)-hydroxy-2-thiazolidinylidene}= cyanamide	Goat, rat
	M13 KNO 2665, PIZ 1271 *Glucuronic acid conjugate of {3-[(6-chloro-3-pyridinyl)methyl]-5(or 4)-hydroxy-2-thiazolidinylidene}= cyanamide*	Goat, rat
	M14 6-CP-cyanoguanidine, KNO 1872 N-[(6-chloro-3-pyridinyl)methyl]-N'-cyanoguanidine	Hen, rat

Structure	Name	Occurrence
	M15 S-(6-CP-cyanoamidino)-acetylcystein, KNO 2684 N-acetyl-3-{[N-[(6-chloro-3-pyridinyl) methyl]-N'-cyano]amidinothio}alanine	Goat, hen, rat
	M16 thiacloprid O-analogue, NTN 35078, PIZ 1266, KNO 1859 {3-[(6-chloro-3-pyridinyl)methyl]-2-oxazolidinylidene}cyanamid	Goat, hen, rat
	M17 6-CP-thiobiuret, KNO 1864 1-[(6-chloro-3-pyridinyl)methyl]-2-thiobiuret	Goat rat
	M18 1-CM-2-thiobiuret, PIZ 1241C N-{[(aminocarbonyl)amino]=thioxomethyl}glycine	Rat
	M19 PIZ 1252, PIZ 1250 N-[cyanimino(methylthio)methyl]=glycine	Rat
	M20 PIZ 1297B (4-oxo-2-thiazolidinylidene)=cyanamide	Rat
	M21 PIZ 1245 2-thiazolylcyanamide	Rat
	M22 PIZ 1243, PIZ 1244 [3-(5-O-sulfono-furanosyl)-2-thiazolyl]cyanamide	Rat
	M23 PIZ 1249 2-thiazolylurea	Rat

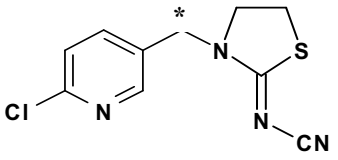
Structure	Name	Occurrence
	M24 PIZ 1297E N-[(6-chloro-3-pyridinyl)methyl]-2-(methylsulfinyl)acetamide	Rat
	M25 WAK 6935, PIZ 1297F N-[(6-chloro-3-pyridinyl)methyl]-N-(2-hydroxyethyl)imidodicarbonic diamide	Rat
	M26 PIZ 1253 N-[(6-chloro-3-pyridinyl)methyl]-N'-cyano-N-(2-hydroxyethyl)thiourea	Rat
	M27 PIZ 1297D {3-[(6-chloro-3-pyridinyl)methyl]-4-oxo-2-thiazolidinylidene}cyanamide	Rat
	M28 PIZ 1269X N-[(6-chloro-3-pyridinyl)methyl]-2-(methylsulfonyl)acetamide	Rat
	M29 thiacloprid thiazolidinimine, KTU 3072, LZR 7497, Ja752-C, NTN 36232, WAK 7376 3-[(6-Chloro-3-pyridinyl)methyl]-2-thiazolidinimine	Soil Rotational crops
	M30 thiacloprid sulfonic acid, sodium salt, thiacloprid sodium sulfonate, WAK 6999, Ja752-D, G9 Sodium 2-[[[(aminocarbonyl)amino]-carbonyl][(6-chloro-3-pyridinyl)-methyl]amino]ethanesulfonate	Soil Rotational crops
	M31 thiacloprid urea, DIJ 10739, Ja752-H [(6-chloro-3-pyridinyl)methyl]urea	Soil Rotational crops
	M32 thiacloprid diamide, WAK 7747, Ja752-I, De23, Z8 N-[(6-chloro-3-pyridinyl)methyl]-imidodicarbonic diamide	Soil
	M33 thiacloprid oxo-sodium sulfonate, Ja752-B Sodium 2-[[[(aminocarbonyl)amino]-carbonyl][(6-chloro-3-pyridinyl)-carbonyl]amino]ethanesulfonate	Soil

Structure	Name	Occurrence
	M34 YRC sulfonic acid amide, SAA, De24, Z1 Sodium 2-[(aminocarbonyl)[(6-chloro-3-pyridinyl)methyl]amino]ethane-sulfonate	Soil Rotational crops
	M35 thiacloprid dewar pyridone, WAK 7259A [3-[(3-oxo-2-azabicyclo[2.2.0]hex-5-en-6-yl)methyl]-2-thiazolidinylidene]-cyanamide	Water
	M36 6-CPA, G2 6-chloro-3-pyridinemethanol	Cotton Rotational crops
	M37 4-hydroxy-KKO 2254, FHW 0104B {3-[(6-chloro-3-pyridinyl)methyl]-4-hydroxy-2-thiazolidinylidene}urea	Cotton Rotational crops
	M38 thiacloprid-olefin, NTN 35099 {3-[(6-chloro-3-pyridinyl)methyl]-2-thiazolylidene}cyanamide	Cotton
	M39 6-CPA-glucosylpentoside, G7 Glucosylpentoside of 6-chloro-3-pyridine-methanol	Cotton
	M40 6-CPA-glucosylphosphate/sulfate, G10 Glucosylphosphate/sulfate of 6-chloro-3-pyridinemethanol	Cotton
	M41 KNO 2673 N-[(6-chloro-3-pyridinyl)methyl]-(methylsulfinyl)carboxamide	Goat, hen
	M42 ANC 1502 N-[(6-chloro-3-pyridinyl)methyl]-N'-cyano-N-[2-(methylthio)ethyl]urea	Goat
	M43 ANC 1503 N-[(6-chloro-3-pyridinyl)methyl]-N-[2-(methylthio)ethyl]imidodi-carbonic diamide	Goat
	M44 ANC 1508A 4-{[(6-chloro-3-pyridinyl)methyl] [2-(methylthio)ethyl]amino}-4-oxo-butanoic acid	Goat

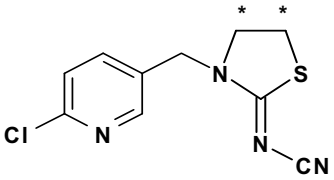
Structure	Name	Occurrence
	M45 ANC 1508B 4-{[(6-chloro-3-pyridinyl)methyl][(methylsulfinyl)carbonyl]amino}-4-oxo-butanoic acid	Goat

Studies of metabolism and degradation were carried out with [^{14}C]-thiacloprid labelled in the methylene-position and in the thiazolidine-position as shown below.

[Methylene-^{14}C]-thiacloprid



[Thiazolidine-^{14}C]-thiacloprid



★ = position of label

Animal metabolism

The metabolism of thiacloprid has been studied in laboratory rats, goats and hens, in compliance with GLP. Rat metabolism studies were evaluated by the WHO Core Assessment Group of the 2006 JMPR. A short summary of the rat metabolism in comparison with the goat and hen metabolism is given on the end of this section.

Lactating goat

The kinetic behaviour and the metabolism of [methylene-^{14}C]-thiacloprid were investigated in a lactating goat (Anderson, C.; Weber, H. and Bornatsch, W. 1998). A target dose of 10 mg/kg body weight was administered orally as a suspension in tragacanth to one lactating goat (34 kg bw at first dosing) on three consecutive days in time intervals of 24 hours.

Radioactivity was measured in the excreta, plasma and milk at different sampling intervals. The goat was sacrificed 6 hours after the final dosage, after which the edible tissues kidney, liver, muscle and fat were radio-assayed. Metabolites were extracted from milk and edible tissues and purified by applying chromatographic techniques (TLC and HPLC). Metabolite identification was based on co-chromatography with authentic references in two different chromatographic systems or on spectroscopic evidence (mass- and NMR-spectroscopy as well as hyphenated techniques). The quantification of the metabolites was conducted by integrating the ^{14}C-signals in the chromatograms of the tissue extracts.

The radioactivity concentrations in the plasma were followed after the first administration. A broad maximum 2 hours after dosage with a peak level of 4.17 µg/mL, corresponding to about 42% of the equidistribution concentration of 10 µg/mL could be observed. The radioactivity was eliminated from the plasma in two phases. The initial phase was described by a half-life of about 5 hours. Thereafter, the elimination process slowed down and was governed by a half-life of about 32 hours. At this time, the concentration in plasma had decreased to 1.47 µg/mL.

The recovery of radioactivity and the excretion behaviour of the lactating goat are presented in Table 2. The excretion amounted to about 53.7% of the total administered radioactivity until sacrifice. A portion of about 48.3% was eliminated with urine and 4.5% with faeces.

A small amount, 0.93% of the total dose, was secreted in milk. Milk was collected twice daily, just before application and again in the evening. An equivalent concentration of 2.43 µg/mL was measured in the milk 8 hours after the first dosage. The maximum concentration of 4.70 µg/mL was obtained at 32 hours.

Table 2. Percentages of the total radioactivity excreted/secreted with urine, faeces and milk.

Sample	Time after 1st dose (h)	Dose No.	% of the total dose
Urine (incl. urine funnel rinse)	0 24 48 54	1 2 3 (sacrifice)	-- 17.95 30.12 0.18
Subtotal			48.25
Faeces	0 24 48 54	1 2 3 (sacrifice)	-- 0.21 2.90 1.36
Subtotal			4.47
Milk	0 8 24 32 48 54	1 2 3 (sacrifice)	-- 0.14 0.17 0.27 0.14 0.21
Subtotal			0.93
Total excreted			53.65
Calculated/estimated residue in edible tissues			5.61
Recovery			59.25

Due to the short survival period after the last dosage, 40% of the dose was not recovered in the excreta.

At sacrifice 6 hours after the last administration, the highest equivalent concentration was measured in the kidney (24.78 mg/kg fresh weight), followed by that obtained for the liver (17.4 mg/kg). These concentrations corresponded to 0.21% and 1.25% of the total dose in the kidneys and liver, respectively. The residue concentrations of the other edible tissues are at least fourfold lower. The detailed data are shown in Table 3.

Table 3. Residue levels of thiacloprid equivalents in the edible tissues and organs of the lactating goat.

Organ	Residue levels (mg/kg)
Kidney	24.78
Liver	17.40
Muscle (flank)	4.18
Muscle (loin)	3.92
Muscle (round)	3.81
Fat (omental)	1.56
Fat (perirenal)	1.59
Fat (subcutaneous)	4.86
Milk (at sacrifice)	4.10

The radioactive residues were extracted from milk and edible tissues with high recoveries of 92 up to 99% using acetonitrile and mixtures of acetonitrile with 0.5% aqueous NaCl. In order to optimise the clean-up procedure and to provide sufficient sample material for metabolite identification, several series of extractions were conducted. After sample clean-up the extracts were analysed by HPLC for the quantitative determination of thiacloprid and its metabolites. The radioactive components were identified by co-chromatography with authentic reference compounds and by spectroscopic investigations.

More than about 96% of the TRR in fat and muscle was recovered by extraction. After sample clean-up, 94% of the TRR or more was subjected to quantitative analysis by HPLC. Unchanged thiacloprid was the pre-dominant component of the TRR accounting for at least 87% of the TRR in fat and about 90% in muscle. The corresponding concentrations were 1.6 mg/kg in fat and 3.5 mg/kg in muscle. Several metabolites were detected at levels near the LOQ. The total rate of identification was about 95% of the TRR in fat and at least 93% of that in muscle (Table 4).

In kidney about 90% or more of the TRR was recovered by extraction. Eighty nine percent of the TRR was subjected to quantitative analysis by HPLC after sample clean-up. Unchanged thiacloprid was the main component of the TRR accounting for at least 28.3% of the TRR in kidney, which corresponded to 7.0 mg/kg. The main metabolites in kidney were the glucuronides M12 and M13 as well as M08 with up to 10.7, 7.1 and 12.3% of the TRR each. A series of metabolites such as M01, M07, M10, M11, M16, M41 and M44/M45 accounted for about 2 to 5% of the TRR individually. The metabolite M15 and two peaks of unknown identity were near or below the LOQ of the TRR in kidney. The portion identified was about 81% of the TRR in kidney (Table 4).

About 94% or more of the TRR in liver was recovered by extraction. After sample clean-up at least 90% of the TRR was subjected to quantitative analysis by HPLC. Unchanged thiacloprid was the predominant component of the TRR accounting for at least 83% of the TRR in liver, corresponding to 14.4 mg/kg. Several metabolites were detected at levels near the LOQ. The total rate of identification was 88 to 92% of the TRR in liver (Table 4).

More than 92% of the TRR in milk was recovered by. For quantitative analysis by HPLC samples containing at least 87% of the TRR following sample clean-up were chosen. Unchanged thiacloprid was also the major radioactive component in milk accounting for at least 58% of the TRR, corresponding to 1.4 mg/kg. The main metabolite in milk was M08 at a level of up to 8.7%. Several metabolites such as M07, M16, M17, M42, M43 and M44/M45, were detected at levels below 5% of the TRR. The metabolite M43 could not be quantified due to interference of other minor unknown metabolites and the lack of an authentic reference compound. A few other metabolites were near the LOQ of 0.9% of the TRR. The portion identified was about 83% of the TRR in milk (Table 4).

Table 4. Quantitative distribution of metabolites in the edible tissues and in milk after administration of [methylene-^{14}C]thiacloprid to a lactating goat based on the first series of extractions.

Metabolite	Fat		Kidney		Liver		Muscle		Milk	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
M01	1.3	0.024	2.6	0.636	0.9	0.149	< 0.8	0.015	< 0.9	0.011
M07[1]			4.4	1.090					3.5	0.087
M08			12.3	3.055	0.9	0.151			8.7	0.213
M10	< 0.8	0.007	3.3	0.812	< 0.8	0.104	< 0.8	0.017	1.4	0.035
M11	< 0.8	0.013	4.4	1.088	< 0.8	0.105	1.0	0.036	< 0.9	0.009
M12	< 0.8	0.013	10.1	2.500	< 0.8	0.049				
M13	< 0.8	0.012	7.1	1.769	< 0.8	0.060				
M15	1.1	0.020	0.8	0.200	0.8	0.148	0.9	0.035	< 0.9	0.019
M16	< 0.8	0.012	4.2	1.039	< 0.8	0.060			1.7	0.042
M17									1.6	0.039
M41			1.1	0.277					< 0.9	0.006
M42									2.3	0.057
M44/M45			2.1	0.513					1.0	0.026
thiacloprid	89.8	1.607	28.3	7.012	83.1	14.45	92.0	3.535	61.0	1.494
Unknown			10.0	1.931	2.4	0.411	2.9	0.112	3.6	0.048
Losses	4.6		9.3		9.8		2.3		13.2	
Extraction yield	97.1		95.3		93.8		98.5		91.9	
Identification	95.4	1.71	80.7	19.99	87.8	15.28	94.7	3.64	83.1	2.04

1) Metabolites not containing the thiazolidine heterocycle

Laying hens

The kinetic behaviour and the metabolism of [methylene-^{14}C]-thiacloprid were studied in laying hens (Weber, H.; Printz, H. and Klempner, A. 1998). The test compound was administered to six hens in tragacanth suspension in three oral doses of 10 mg/kg bw (corresponding to 124 ppm in feed on dry weight basis), one dose per day, on three consecutive days.

Radioactivity was measured in the excreta, plasma and eggs at different intervals. The animals were sacrificed 6 hours after the final dose, after which the edible tissues kidney, liver, skin, muscle and fat were radioassayed. Metabolite analyses were performed with the eggs and the edible tissues except kidney. Metabolites were extracted from eggs and edible tissues with acetonitrile and mixtures of acetonitrile and methanol with a saline solution. This extraction procedure was followed by a microwave extraction step. Purification was conducted by chromatographic techniques (TLC and HPLC). Metabolite identification was based on co-chromatography with authentic references in two different chromatographic systems or on spectroscopic evidence (mass- and NMR-spectroscopy as well as hyphenated techniques). The quantification of the metabolites was conducted by integrating the ^{14}C-signals in the chromatograms of the tissue extracts.

The absorption was fast so that the concentration-time-course of radioactivity in the plasma did not allow a determination of the absorption rate. A concentration of 1.54 µg/mL was obtained at the first sampling point (0.25 hours after dosing). The mean plasma concentration peaked at 3 hours with a mean value of approx. 1.6 µg/mL. Related to the dose of 10 mg/kg body weight, this value corresponded only to 16% of the so-called equidistribution concentration. The radioactivity was monophasically eliminated from the plasma with a half-life of 6.8 hours. Twenty-four hours after a single dose, the mean plasma concentration had declined to 0.19 µg/mL.

Until sacrifice the excretion amounted on average to 75.4% of the radioactivity totally administered. About 29.4% and 29.6% of the radioactivity totally eliminated during the whole test period was excreted within 24 hours after the first and the second administration, respectively. Another portion of 16.4% was excreted after the last dose until sacrifice. On average, only 0.06% of the total dose was determined in the eggs. The recovery of radioactivity and the excretion behaviour of the laying hens, after administration of a daily dose of 10 mg per kg body weight on three consecutive days, are presented in Table 5.

Table 5. Percentages of the total radioactivity excreted/secreted with urine, faeces and eggs.

Sample	Time after 1st dose (h)	% of the total dose	
		Mean	CV (%)[1]
Excreta	24	29.37	11.31
	48	29.62	9.64
	54	16.36	32.52
Subtotal		75.35	10.22
Eggs; 0 - 54 h		0.06	53.18
Totally excreted		75.41	10.22
Estimated residue in tissues prepared		0.71	14.79
Recovery		76.12	10.16

1) CV: coefficient of variance

The highest equivalent concentrations were determined in the liver (3.061 mg/kg) and kidneys (2.404 mg/kg), respectively. The residue concentrations of the other edible tissues were at least fourfold lower. The average data are shown in Table 6.

Table 6. Residue levels of thiacloprid equivalents in the edible tissues and organs of laying hens.

Organ	Residue levels (mg/kg)
Liver	3.061
Kidney	2.404
Muscle (leg)	0.152
Muscle (breast)	0.128
Skin (without fat)	0.295
Fat (subcutaneous)	0.083
Eggs (prior to sacrifice)	0.424
Eggs (from oviduct)	0.652

The radioactivity was extracted with solvent followed by microwave extraction with recoveries above 93%. After purification the extracts were co-chromatographed in two different HPLC-systems with authentic ^{14}C-labelled reference compounds, which were previously isolated during the rat- and goat metabolism studies. All reference compounds were spectroscopically identified.

The unchanged parent compound was the major component in all extracts of edible tissues and eggs. Its concentration was higher in the more lipophilic matrices as compared to muscle or liver. Correspondingly, polar metabolites occurred at higher quantities in muscle and liver. In egg and fat extracts thiacloprid was found in quantities ranging from 48.2% to 71.8% of the TRR. In addition, up to four further polar metabolites, ranging from 1.3% to 8.9% of TRR were identified.

In muscle and liver extracts thiacloprid was found at quantities ranging from 17.3% to 19.4% of the TRR, while up to eight further polar metabolites, ranging from 1.1% to 5.1% of the TRR were identified. Table 7 gives a quantitative overview of the extraction yields and the amounts of identified compounds in the extracts of edible tissues and eggs.

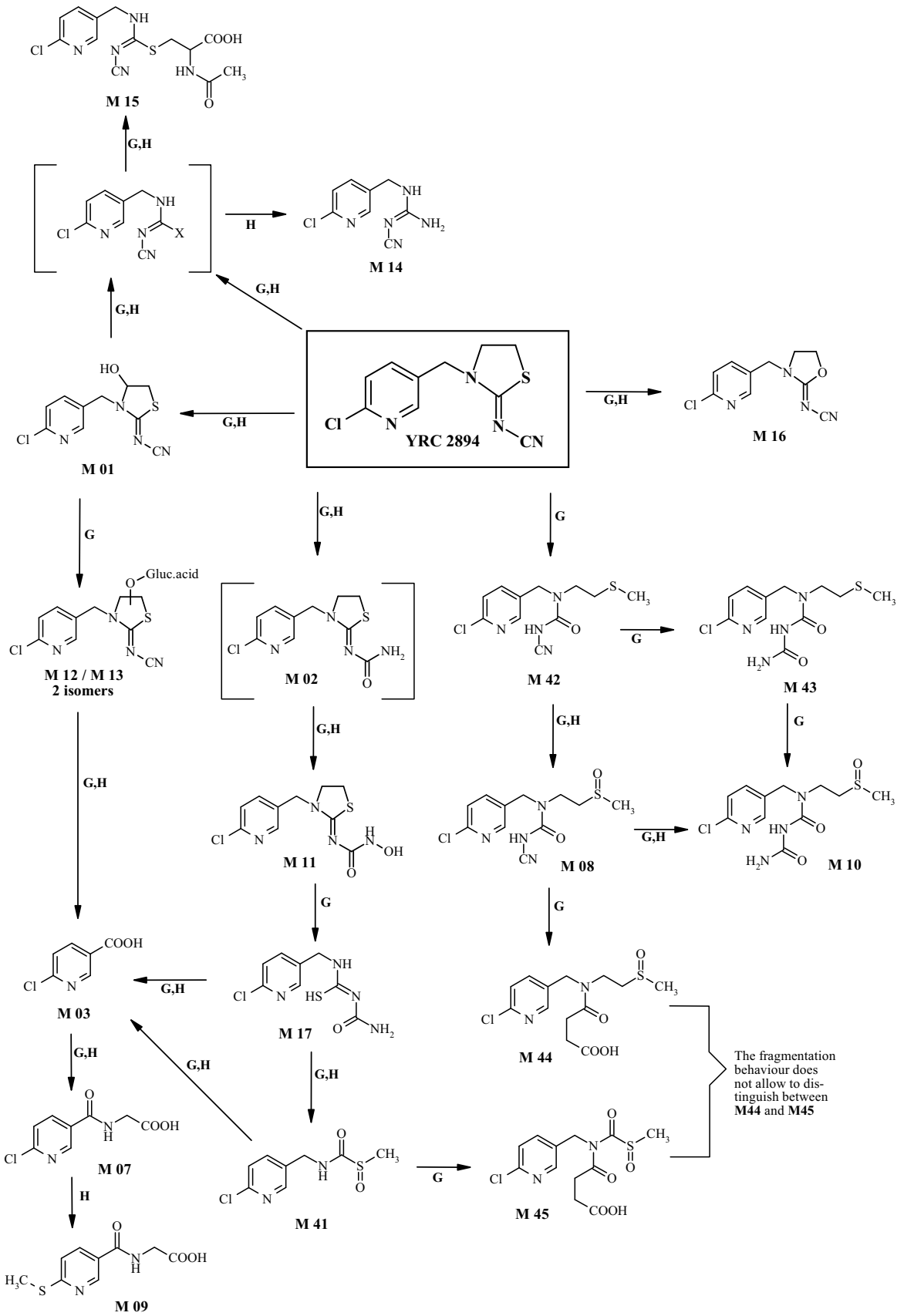
Table 7. Quantitative distribution of metabolites in the edible tissues and in eggs after administration of [methylene-^{14}C]thiacloprid to laying hens.

Metabolite	Eggs		Liver		Muscle		Fat	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
M01	4.6	0.006	1.7	0.054	3.8	0.006		
M03[1]	1.9	0.002			3.3	0.005		
M07[1]	6.4	0.008	1.1	0.034	1.5	0.002		
M08			4.1	0.128			8.9	0.010
M09[1]					1.4	0.002		
M10					5.1	0.007		
M11	1.3	0.002	4.6	0.144	10.9	0.016		
M14			4.6	0.142				
M15			1.5	0.047				
M16			2.8	0.088				
M41			2.4	0.076	4.8	0.007		
thiacloprid	48.2	0.059	17.3	0.537	19.4	0.028	71.8	0.077
Extraction yield	97.5		96.9		96.6		93.2	
Sum identified	62.5	0.077	40.2	1.250	50.2	0.073	80.8	0.087

1) Metabolites not containing the thiazolidine heterocycle ring

The metabolites found in the edible tissues and eggs of the laying hen were almost completely identical with those found in the edible tissues and milk of the lactating goat as well as those found in the rat metabolism study. Therefore, the proposed biotransformation pathway of thiacloprid shows the degradation in poultry and ruminants (Figure 1).

Based on the results of the livestock metabolism studies, the parent compound only is considered as relevant residue of concern for food commodities of animal origin.



G: Goat; H: Hen

Figure 1. Metabolic pathways of thiacloprid in goats and hens.

Plant metabolism

The metabolism of thiacloprid has been studied after spray application in apples, tomatoes, cotton and wheat in compliance with the GLP.

Apples

Apples of the variety James Grieve were treated twice with [methylene-^{14}C]-thiacloprid at an interval of 14 days (Clark, T. and Bornatsch, W. 1997). The last application was made 14 days prior to harvest. An aqueous suspension of the formulated product was applied uniformly to each of the apples using an Eppendorf syringe fitted with a tuft of hair at the tip. The following amounts were applied to each apple at both application dates: 104.8 µg 600 SC (50.6% ai), 53.0 µg ai, 0.22 MBq. The application rate was slightly exaggerated when compared to the annual recommended field rate of 300 g/ha thiacloprid. The apples were sampled 14 days after the second application (day 0).

In the scope of this study also a translocation experiment was conducted, in which [methylene-^{14}C]-thiacloprid was applied on the same days as for the metabolism experiment. The tests were conducted each with one apple and the adjacent leaves above and below. The same solutions and method of application was used as for the metabolism experiment, i.e., the leaves received the same total amount of radioactivity as each apple in the metabolism experiment.

The apples were extracted with methanol/water (1:1) and methanol. The radioactivity in the extract was measured by liquid scintillation. The solids were air dried and aliquots taken and combusted. The identification was achieved by co-chromatography (TLC and HPLC) with the authentic reference compounds as well as by ^{1}H-NMR and mass-spectroscopic methods.

The total radioactive residue (TRR) in apples amounted to 0.74 mg/kg parent compound equivalents. The vast majority of the TRR was removed by surface washing with dichloromethane (84.4% or 0.62 mg/kg), 12.9% (0.10 mg/kg) was detected in the extract and only 2.7% (0.02 mg/kg) remained unextracted in the solids, which were not investigated further.

Of the radioactivity present in the surface wash solution, extract and solids 90.8% (0.67 mg/kg) was identified as unchanged parent compound. Only two other metabolites were detected in any significant quantities and these were identified as the 4-hydroxy derivative of the parent compound (M01; 2.2%, 0.02 mg/kg) and the amide (M02; 1.3%, 0.01 mg/kg). A summary of the distribution of metabolites in the different fractions of apples is presented in Table 8.

The results of the translocation experiments showed that some of the applied radioactivity (ca. 25% on average) was lost, probably due to volatilisation. Virtually all the recovered radioactivity was found in the treated leaves while only traces of parent compound and metabolites (0.05% on average) were translocated from the leaves to the apples above and below the treated leaves.

Table 8. Distribution of metabolites in the different fractions of apples.

Compound/Metabolite	% TRR	mg/kg parent equivalents
Surface wash solution	84.4	0.62
Thiacloprid	82.4	0.61
M01	1.4	0.01
M02	0.6	< 0.01
Extract	12.9	0.1
Thiacloprid	8.4	0.06
M01	0.8	0.01
M02	0.7	0.01
Polar radioactivity	3.0	0.02
Solids	2.7	0.02
Total	100	0.74

Tomatoes - metabolism

In a greenhouse 10 bunches of tomatoes (82 tomatoes) were sprayed twice with [methylene-^{14}C]-thiacloprid at an interval of 14 days (Babczinski, P. 1997). The last application was made 14 days prior to the final harvest. An aqueous suspension of the formulated product was applied uniformly to each of the plants using a metre jet spray gun fitted with a flat-fan nozzle. Each bunch of tomatoes including the surrounding leaves and stalks was sprayed separately. In each application a total of 30 mL of formulation was applied which is equivalent to 7.9 mg ai or to 32.6 MBq of total radioactivity, respectively. This corresponded to an application rate of approximately 2 × 0.375 kg ai/ha.

The tomatoes were harvested as follows: Immediately after the second application (day 0) five tomatoes were harvested. Three of these were used to calculate the TRR and to analyse the quantitative distribution of metabolites. The remaining two tomatoes were used to determine the efficiency of surface washing with methanol.

At day 3, after the second application, 29 tomatoes were collected while the final harvest of 38 tomatoes was performed at day 14. Eight tomatoes were also harvested at this time and stored at -20°C without surface wash as a reserve sample for the validation of the residue method.

The tomatoes were surface washed and extracted with methanol. The radioactivity in the extract was measured by liquid scintillation. The solids were air dried and aliquots taken and combusted. The identification was achieved by co-chromatography (TLC and HPLC) with the authentic reference compounds as well as by ^{1}H-NMR and mass-spectroscopic methods.

The TRR in tomatoes at day 0 amounted to 0.76 mg/kg parent compound equivalents. The vast majority of the TRR was removed by surface washing with methanol (95.8% or 0.72 mg/kg), 4.0% (0.03 mg/kg) was detected in the extract and only 0.2% (< 0.01 mg/kg) remained unextracted in the solids.

Tomatoes harvested at day 3 and 14 yielded TRR values of 0.77 and 0.94 mg/kg, respectively. Again, the biggest part could be removed by surface washing and amounted to 87.8% (0.68 mg/kg) on day 3 and 84.3% (0.79 mg/kg) on day 14. The respective amounts in the methanol extract were 11.2% (0.09 mg/kg) on day 3 and 14.1% (0.13 mg/kg) on day 14 indicating a slight increase of the uptake of radioactivity during this time. Also the level of radioactivity in the solids increased slightly from day 0 to day 3 (1.0%, < 0.01 mg/kg) and further to day 14 (1.6%, 0.02 mg/kg). The solids were not investigated further.

Of the radioactivity present in the surface wash solution and the extract 94.4% (0.88 mg/kg) was identified as unchanged parent compound. Six further metabolites were detected in low quantities ranging from < 0.01 to 0.03 mg/kg. The main metabolite was identified as a complex 6-chloropicolyl alcohol glucoside (M05; 2.8%, 0.03 mg/kg). Three further glucosides were detected (together 0.6%, < 0.01 mg/kg), one of which was the 6-chloropicolyl alcohol glucoside (M04, 0.3%, < 0.01 mg/kg). The other two remained unidentified. Two more metabolites were identified as the 4-hydroxy derivative of the parent compound (M01; 0.4%, < 0.01 mg/kg) and 6-chloronicotinic acid (M03; 0.2%, < 0.01 mg/kg). A total of 98.1% (0.92 mg/kg) of the TRR in tomatoes was identified. The results are summarised in Table 9.

Table 9. Distribution of metabolites in different fractions of tomatoes (day 14).

Compound/Metabolite	% TRR	Mg/kg parent equivalents
Surface wash solution	84.3	0.79
Identified	84.3	0.79
Thiacloprid	84.3	0.79
Methanol extract	14.1	0.13
Identified	13.8	0.09
Thiacloprid	10.1	< 0.01
M01	0.4	< 0.01
M03	0.2	< 0.01
M04	0.3	0.03
M05	2.8	< 0.01

Compound/Metabolite	% TRR	Mg/kg parent equivalents
Characterised as glucose conjugates of M04	0.3	< 0.01
Non extractable residues	1.6	0.02
Subtotal identified	98.1	0.92
Subtotal identified/characterised	98.4	0.92
Total residue	100	0.94

Tomatoes – translocation

In a supplementary study to the above described metabolism study, the translocation in tomatoes was also investigated (Koester, J. 1997). [Methylene-^{14}C-methyl]-thiacloprid, formulated as a 600 SC, was sprayed twice to the soil surface of four container grown plants. The application was based on the assumption that under GAP conditions a certain fraction of the application solution would reach the soil during and after spraying. The application rate was 0.55 mg active substance at the first and 0.58 mg active substance at the second application. The total application rate was equivalent to 89.7 g ai/ha. The time interval between the applications was 14 days. The tomatoes of the first two plants were harvested 3 days after treatment and those of the remaining two plants 14 days after the second application as a mixture of green, reddish, and red fruits.

The TRR in the tomatoes was determined by adding the radioactivity in the extracts and the air-dried solids after extraction. In all cases, less than 0.1% of the radioactivity applied to the soil surfaces was detected in the tomato fruits. From the total amount recovered, an average of 94.1% was found in the extracts, the remainder was measured in the unextracted solids. The transformation of the total residue concentrations to parent compound equivalents yielded in all cases concentrations below 0.001 mg/kg. As a result, further analyses on the extracts were not conducted.

Cotton

[Methylene-^{14}C-methyl]thiacloprid was applied to cotton in three spray applications (Babczinski, P. 1998). The cumulative application rate was 375 g ai/ha. The time interval between each of the treatments was seven days. 120 days after the last application the cotton plants were harvested, i.e., at the time of natural maturity.

Leaves, petals, gin trash, lint, and seeds were collected and homogenised. The homogenised samples were extracted with acetonitrile and acetonitrile/water (1:1). The residue remained in the extracted gin trash was further extracted using acetone/water (1:1) in a microwave at 120°C, and the aqueous remainder partitioned against n-hexane and dichloromethane. The residues in extracted seeds were further extracted with n-hexane, dichloromethane and acetone followed by an extraction of the resulting residue using acetic acid and acetone/water (1:1). The latter extraction step was repeated in a micro-wave. The combined aqueous phases were subsequently partitioned against dichloromethane and ethyl acetate. Radioactivity was determined in the extracts and the extracted solids.

Metabolites were purified from the extracts by solid phase extraction methods and identified by comparative thin-layer chromatography and HPLC with authentic reference compounds using different chromatographic methods. Mass- and NMR-spectroscopy were also employed for structure elucidation.

The total radioactive residue (TRR) in cotton gin trash at harvest amounted to 3.21 mg/kg (ai equivalents), i.e., 97.2% was extracted. The main component was the parent compound (73.5%, 2.36 mg/kg). Fourteen metabolites were detected, twelve of these amounted to 14.8% (0.48 mg/kg) of the gin trash residue. These were: 6-chloronicotinic acid (M03) as the main gin trash metabolite (3.3%, 0.11 mg/kg), 4-hydroxy thiacloprid (M01; 2.7%, 0.08 mg/kg), 6-chloro-picolyl alcohol (M36; 1.5%, 0.05 mg/kg) and its glucoside (M04; 1.2%, 0.04 mg/kg), two complex 6-CPA glucosides (glucosyl-pentoside [M39] and glucosylphosphate or -sulfate [M40]; each 1.1%, 0.04 mg/kg), the sulfonic acid derivative (M30; 0.9%, 0.03 mg/kg) and as a minor component monohydroxylated thiacloprid amide (M37; 0.4%, 0.01 mg/kg). Two further metabolites were identified as thiacloprid amide (M02; 1.9%,

0.06 mg/kg) and the olefin derivative of the parent compound (M38; not quantified). In total, 85.7% (2.76 mg/kg) of the TRR in cotton gin trash was identified.

The total radioactive residue in cotton leaves (including petals) amounted to 30.35 mg/kg ai equivalent. In total, 98.1% (29.77 mg/kg) was extracted; the non-extractable residue amounted to 1.9% (0.58 mg/kg). As in gin trash, the main component was thiacloprid (83.9%, 25.46 mg/kg). Thirteen metabolites were detected, amounting to 12.3% (3.73 mg/kg) of the leaves residue. Nine of these were identified, eight of them were also found in gin trash. These were: Two complex 6-CPA glucosides (M39; glucosyl-pentoside as the main leaf metabolite: 2.7%, 0.82 mg/kg; M40; glucosyl-phosphate or -sulphate: 1.4%, 0.43 mg/kg), 6-chloropicolyl alcohol (M36; 0.5%, 0.15 mg/kg) and its glucoside (M04; 1.2%, 0.37 mg/kg), 6-chloronicotinic acid (M03; 1.1%, 0.33 mg/kg), 4-hydroxylated thiacloprid (M01; 0.8%, 0.24 mg/kg), 4-hydroxylated thiacloprid amide (M37; 1.2%, 0.36 mg/kg) and as a minor component the sulfonic acid derivative (M30; 0.3%, 0.09 mg/kg). A further metabolite was characterised as a complex conjugate of 6-chloronicotinic acid with glucose and a plant constituent (possibly protocatechuic acid). This metabolite is probably similar to one of the complex 6-chloronicotinic acid conjugates described for cotton seed. The four unidentified metabolites were polar in nature and each amounted to ≤ 1.2% (≤ 0.37 mg/kg). In total, 93.1% (28.25 mg/kg) of the TRR in cotton leaves was identified.

The TRR in cotton seed at harvest amounted to 1.12 mg/kg ai equivalents, 99.8% thereof was extracted. The main metabolite was free 6-chloronicotinic acid (M03) which accounted for 45.8% (0.51 mg/kg) of the TRR. Unchanged thiacloprid was only a minor component (0.6%, 0.01 mg/kg). Up to twenty further metabolites were detected totally accounting for 42.7% (0.48 mg/kg) of the TRR. That part of the seed residue which was neither free 6-chloronicotinic acid (M03) nor thiacloprid, 41.3% (0.46 mg/kg) was characterised after oxidation to comprise the 6-chloronicotinic acid-moiety by using permanganate oxidation as developed in total residue method for imidacloprid, a structurally related chloronicotinyl insecticide. Therefore, the total residue based on or identical with 6-chloronicotinic acid (including the parent compound) equalled 87.7% (0.98 mg/kg).

The distribution of metabolites in cotton is summarised in Table 10.

Table 10. Distribution of metabolites in different fractions of tomatoes (day 14).

Crop	Cotton leaves	Cotton seed
TRR = mg/kg	30.35	1.12
thiacloprid	83.9	0.6
M01	0.8	-
M03[1]	1.1	45.8
M04[1]	1.2	-
M30	0.3	-
M36[1]	0.5	-
M37	1.2	-
M39[1]	2.7	-
M40[1]	1.4	-
Complex glucosides of M36	-	0.3
Complex glucosides of M03	-	29.7
Unknown (%)	5.0	23.4
Not extracted (%)	1.9	0.2
Total (%)	100	100

1) Metabolites not containing the thiazolidine heterocycle

Wheat

The metabolism of thiacloprid was investigated in spring wheat following two applications with a spray interval of 14 days and a pre-harvest interval of 21days (Bongartz, R. and Neumann, B. 2001). The actual application conditions simulated normal practice: Radiolabelled [methylene-^{14}C]-thiacloprid was formulated as a 112.5 SE containing 100 g/L thiacloprid and 12.5 g/L of a mixing partner, which was replaced by water in the study. A computer controlled track sprayer with a flat-fan

nozzle was used for the two applications. In the first spray application 49.9 g ai/ha was applied to wheat at growth stage 75 of the BBCH code (medium milk stage). The second application of 44.8 g ai/ha followed 14 days later at growth stage 77 of the BBCH code (late milk stage). This resulted in a total application rate of 94.7 g ai/ha. Wheat hay was sampled seven days after the first application. Wheat straw and grain were harvested at maturity 21 days after the second application.

Hay, straw, and grain were homogenised and extracted with acetonitrile/water (1:1) and acetonitrile. The combined extracts for each sample material were partitioned with dichloromethane. All phases were chromatographed and quantitated by HPLC with radioactivity detection. The solid remained after the first extraction was extracted with acetonitrile/water (1:1) at 120°C using a microwave. After this, the residues remaining in straw were hydrolysed with dioxane/2N HCl (9:1). Metabolites were isolated by HPLC and identified by co-chromatography with authentic reference compounds or by mass spectroscopy.

The total radioactive residue (TRR) in hay, which received only one application, amounted to 2.04 mg/kg (parent compound equivalents), 94.6% was extracted by liquid-solid and additional 3% by microwave extraction. The main component was the parent compound (81.4%, 1.66 mg/kg). Many minor metabolites were detected, all amounting to ≤ 0.03 mg/kg each. Ten metabolites were identified: a conjugate of 6-chloronicotinic acid (1.7%, 0.03 mg/kg) and 6-chloronicotinic acid (M03, 1.2%, 0.03 mg/kg), 4-hydroxy-thiacloprid (M01, 1.6%, 0.03 mg/kg), the sulfonic acid derivative (M30, 1.2%, 0.03 mg/kg) and a conjugate thereof (0.4%, 0.01 mg/kg), thiacloprid diamide (M32, 0.5%, 0.01 mg/kg), 6-chloropicolyl alcohol (M36, 0.4%, 0.01 mg/kg), thiacloprid-olefin (M38, 0.4%, 0.01 mg/kg), thiacloprid-amide (M02, 0.2%, < 0.01 mg/kg) and 3-aminocarbonyl-1-(6-chloro-pyridin-3-ylmethyl)-1-(2-hydroxy-ethyl)-urea (M25, 0.1%, < 0.01 mg/kg). In total, 89.3% (1.82 mg/kg) of the TRR in hay was identified.

In straw the TRR amounted to 12.36 mg/kg (parent compound equivalents), 95.0% was extracted by liquid-solid and additional 3.1% by microwave extraction. The extraction residue was further treated with dioxane/HCl, which again released 1.2% of the TRR. The main component in straw was the parent compound (83.4%, 10.30 mg/kg). Ten metabolites were identified: 6-chloronicotinic acid (M03, 2.2%, 0.27 mg/kg) and a conjugate thereof (1.1%, 0.13 mg/kg), 4-hydroxy-thiacloprid (M01, 1.9%, 0.23 mg/kg), the sulfonic acid derivative (M30, 1.0%, 0.13 mg/kg) and a conjugate thereof (0.3%, 0.03 mg/kg), thiacloprid diamide (M32, 0.4%, 0.05 mg/kg), 6-chloropicolyl alcohol (M36, 0.3%, 0.04 mg/kg), thiacloprid-olefin (M38, 0.3%, 0.04 mg/kg), thiacloprid-amide (M02, 0.3%, 0.04 mg/kg) and 3-aminocarbonyl-1-(6-chloro-pyridin-3-ylmethyl)-1-(2-hydroxy-ethyl)-urea (M25, 0.1%, 0.01 mg/kg). In total, 91.3% (11.28 mg/kg) of the TRR in straw was identified.

The total radioactive residue (TRR) in grain amounted to 0.21 mg/kg (parent compound equivalents), 89.6% was extracted by liquid-solid and additional 4.8% by microwave extraction. The main component in grain was the parent compound (80.9%, 0.17 mg/kg). Only few minor metabolites were detected, all of them << 0.01 mg/kg. Two metabolites were assigned to the conjugate of 6-chloronicotinic acid (1.7%, < 0.01 mg/kg) and 4-hydroxy-thiacloprid (M01, 0.7%, < 0.01 mg/kg). In total, 83.3% (0.17 mg/kg) of the TRR in grain was identified. The distribution of metabolites in wheat is summarised in Table 11.

Table 11. Distribution of metabolites in different fractions of wheat.

Crop crop part	Wheat hay	Wheat straw	Wheat grain
TRR = mg/kg	2.04	12.36	0.21
thiacloprid	81.4	83.4	80.9
M01	1.6	1.9	0.7
M02	0.2	0.3	-
M03[1]	1.2	2.2	-
M25	0.1	0.1	-
M30	1.2	1.0	-
M32	0.5	0.4	-

Crop crop part	Wheat hay	Wheat straw	Wheat grain
M36[1]	0.4	0.3	-
M38	0.4	0.3	-
Conjugate of M03	1.7	1.1	1.7
Conjugate of M30	0.4	0.3	-
Unknown (%)	8.3	8.0	11.1
Not extracted (%)	2.4	0.7	5.6
Total (%)	100	100	100

1 Metabolites not containing the thiazolidine heterocycle

Environmental fate in soil

Hydrolysis

The test was performed to determine the rate of hydrolysis of thiacloprid in sterile aqueous solution at various pH values at 25°C and to obtain information on the identity and pattern of hydrolysis products (Brumhard, B. 1998). The hydrolysis of [methylene-^{14}C]-thiacloprid was investigated in the dark at pH values of 5, 7 and 9 at a concentration of 0.35 mg ai/L. Test duration was 30 days with sampling intervals of 0, 2, 7, 13, 20 and 27 days. After the 30 days storage period thiacloprid recoveries were 95–98% of the applied radioactivity in all samples. In the pH range tested formation of hydrolysis products was only observed at pH 9 at amounts less than 2% of the applied radioactivity.

Photolysis on soil surfaces

The photo-transformation of [methylene-^{14}C]-thiacloprid was studied (Brumhard, B. 1998) on thin layers of the sandy loam soil "Howe" (IN/USA; 65.5% sand; 26.3% silt; 8.2% clay; 1.09% org. C; pH in CaCl2: 7.1) which was also used in the aerobic soil metabolism study. The dose rate was 2.34 mg/kg soil (dry substance) corresponding to about 350 g ai/ha (calculated for a soil density of 1.5 g/cm^3 and 1 cm depth). The water content of the samples was adjusted to 75% of the 1/3 bar moisture of the soil. The soil thin layers were continuously irradiated with a Xenon lamp simulating natural sunlight. The spectrum was cut off at wavelengths below 290 nm and the light intensity was 9.3 mW/cm^2. The temperature of the testing system was maintained at 25 ± 1°C. Duplicate samples were taken for analysis 0, 4, 7, 13 and 18 days post-treatment. 'Dark' samples were taken 7 and 19 days post-treatment. Volatile radioactivity was trapped using soda lime and released for measurement by adding HCl.

Soils were exhaustively extracted by shaking with methanol immediately after sampling. Additionally, the soil was subjected to further extraction with methanol/water (50/50) at about 180°C using a Soxtec® high temperature extraction unit. The radioactivity was determined in all samples and the extracts analysed by AMD (automated multiple development)-TLC and HPLC-methods. Metabolites were identified by NMR- and mass-spectroscopy and by comparison with authentic reference compounds.

Under the experimental conditions thiacloprid degraded with an experimental half-life (DT_{50}) of 18.8 days in the irradiated samples. This corresponds to a calculated environmental half-life of 74 days during midday and midsummer at 40° of latitude (Phoenix, AZ, USA). It is expected that the half-life at sites with less radiation intensity or in spring, fall or winter would be longer. The amount of unextracted residues was below 10% of the applied radioactivity. Besides the parent compound, one main degradate (M02) was observed in the extracts of irradiated and dark soil samples. At any time of the study all other products, individually made up less than 5% of the applied radioactivity. One of these metabolites was identified as the so-called "Dewar-pyridone" (M35). The results of the distribution of thiacloprid and its degradation products are summarised in Figure 2. Metabolic pathways of thiacloprid in plants

Table 12.

The degradation observed in the dark samples (DT_{50} = 6.3 days) was about threefold faster as compared to the irradiated samples. It is therefore concluded from this study that under environmental conditions the solar reaction will contribute only to a very limited extent to the overall degradation of thiacloprid on soil surfaces.

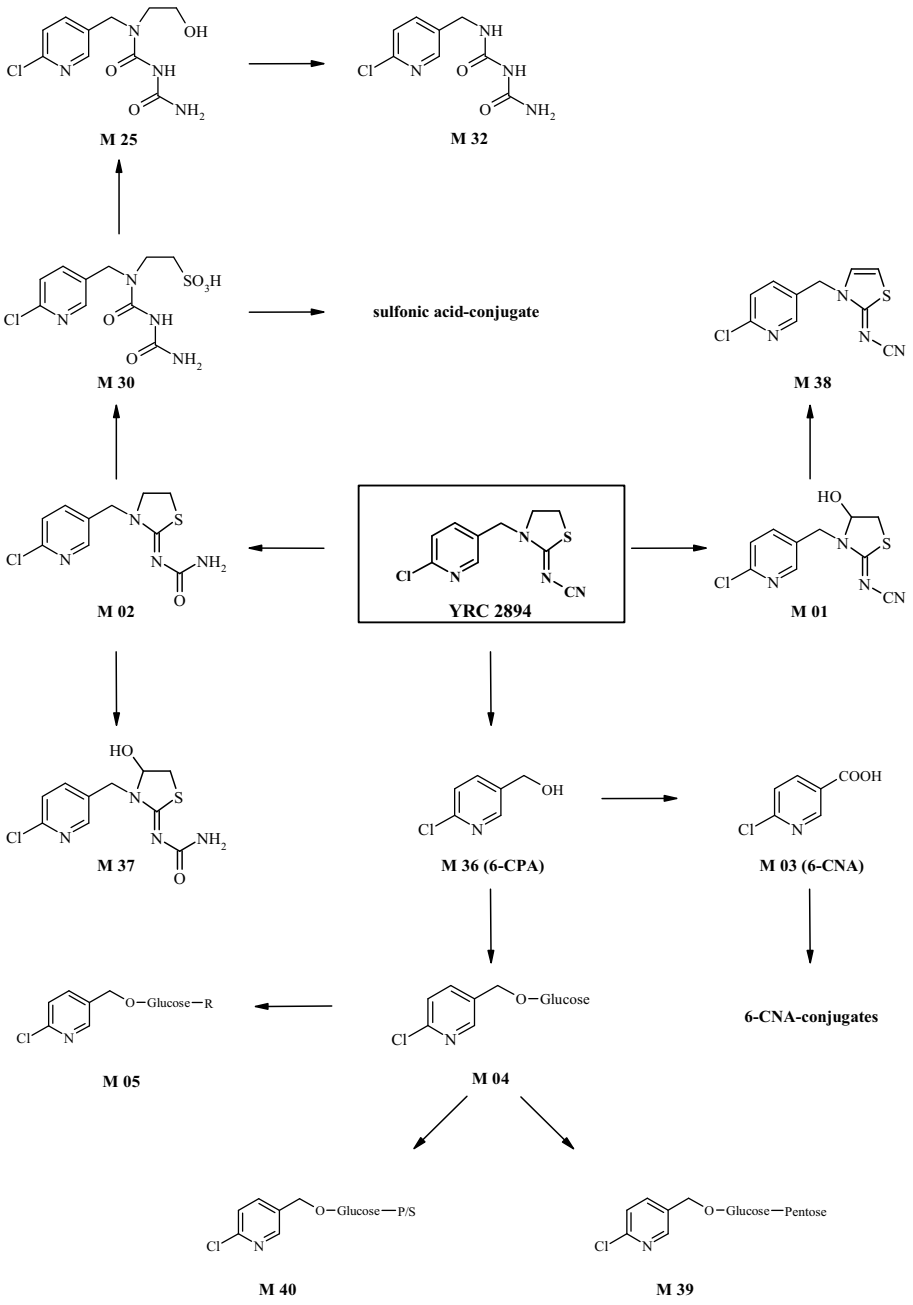


Figure 2. Metabolic pathways of thiacloprid in plants

Table 12. Recovery of radioactivity and distribution of the active substance and metabolites after application of [methylene-^{14}C]-thiacloprid to thin soil layers of sandy loam under artificial light conditions and in the dark (in % of the applied radioactivity) [mean of two values].

Study	Conditions	Exposure time (days)	A.S. %	M02 %	M35 %	CO_2 %	Unknown %	Extracted %	Not extracted %	Total %
		0	94.1	0.6		< 0.1	1.4	96.1	4.6	100.7

Study	Conditions	Exposure time (days)	A.S. %	M02 %	M35 %	CO_2 %	Unknown %	Extracted %	Not extracted %	Total %
		4	81.6	5.6		0.1	7.1	94.3	7.0	101.4
Photolysis	Irradiated	7	78.8	7.9		< 0.1	7.2	94.4	7.4	101.9
on soil		13	61.8	13.9	3.0	0.2	12.2	92.3	6.8	99.3
surfaces		18	47.5	23.8	3.8	0.2	14.2	89.6	9.5	99.3
	Dark	0	94.1	0.6		< 0.1	1.4	96.1	4.6	100.7
	control	7	35.5	49.6	0.6	0.1	7.5	93.2	7.2	100.4
		19	11.1	69.8	0.7	0.3	10.9	92.3	7.7	100.3

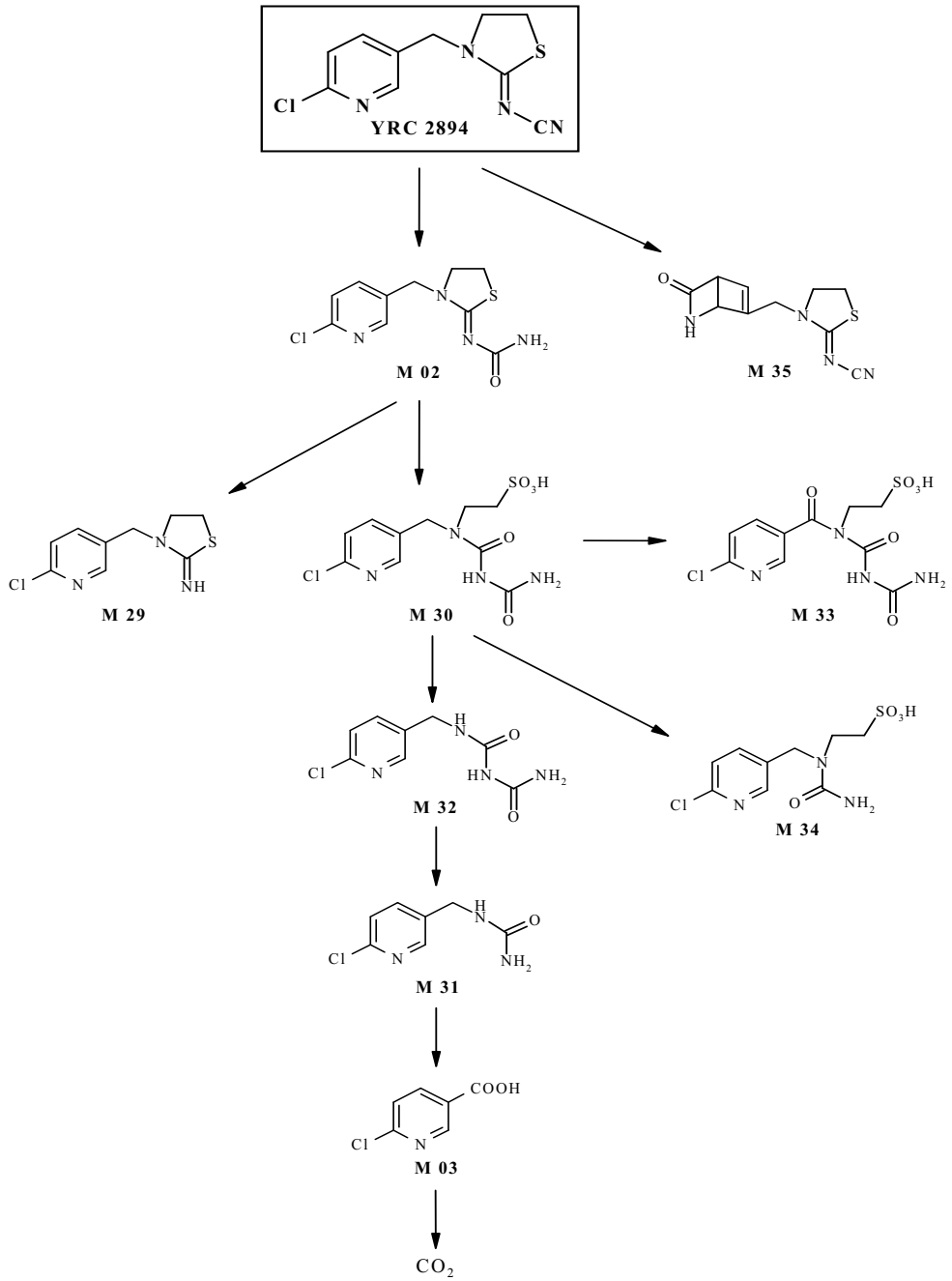


Figure 3. Proposed degradation pathway of thiacloprid in the soil, considering outdoor and photolysis on soil surfaces studies.

Residues in rotational crops

The metabolism of thiacloprid, formulated as a SC 480, was investigated (Clark, T. and Babczinski, P., 1998) in the following rotational crops spring wheat, lettuce and turnips planted into containers of soil treated with of [pyridinyl-^{14}C-methyl]-thiacloprid. The rate applied was approximately 10% above the highest rate used in the first season of residue trials (375 g ai/ha). This was to allow for any losses during application. The soil was aged for 30 days and tilled to a depth of 15 cm prior to planting the first set of rotational crops. Lettuce was transplanted in one quarter of the soil area,

turnips were sown in a second quarter and spring wheat was sown in the remaining half. Lettuce was harvested on day 63 (i.e., 63 days after application) and turnips on day 105. Wheat was sampled at three different intervals, immature (day 70), hay (day 128) and maturity (day 170). The roots of lettuce and wheat were not harvested and remained in the soil for all rotations. Following the harvest of wheat, the soil was tilled (as for the first interval) and a second set of rotational crops were sown/transplanted as described above on day 170. In the second cycle the crops were sown/transplanted into different sectors of the container. The harvest days were as follows, lettuce day 220, turnips day 259, immature wheat day 212, wheat hay 232 and wheat grain and straw day 261. The above was repeated for the third rotation at the one year plant back interval (day 354). The harvest days were as follows, lettuce day 387, turnips day 441, immature wheat day 395, wheat hay 455 and wheat grain and straw day 526.

At maturity the wheat was separated into grain and straw. The glumes and the remainder of the ears were added to the straw fraction. Mature turnips were separated into bulbs and tops. The following seven plant fractions were obtained at each of the three intervals: Lettuce, turnip tops, turnip bulbs, immature wheat, wheat hay, wheat straw, and wheat grain. The individual plant fractions were macerated and extracted with methanol/water (1:1). Exhaustive extraction was achieved by microwave extraction using mixtures of acetonitrile/water. The radioactive content was measured by LSC in all extracts and the extraction residues after combustion. The extracts were cleaned by solid phase extraction, where necessary, and analysed by TLC and HPLC. Metabolites were identified by co-chromatography with authentic reference compounds and by spectroscopic methods.

A significant decrease in the TRR was observed over the whole period of the study although in some cases an increase was seen between the 30 and 170 day rotational crops. The TRR ranged from 0.005 mg/kg in turnip bulbs from the 354 day rotation to 2.6 mg/kg in wheat straw from the 170 day rotation. Overall, good extractability was achieved by conventional means, generally over 80%, after which microwave extraction was performed on the solids of the 2nd rotation to extract further radioactivity. A maximum of 9.5% of the TRR (turnip bulbs) was additionally extracted from any of the crops. In all cases, except wheat hay and straw, the amounts additionally extracted by microwave were all below 0.001 mg/kg. Only in wheat straw was the residue of any significance (0.15 mg/kg). Furthermore, radio-TLC showed that the radioactivity extracted by microwave was distributed over many components and therefore no further work was carried out on these samples. Due to the fact that the residues were generally very low and distributed over a number of components, the individual components were not quantified and were thus not accounted for in the distribution of metabolites. The total radioactive residue (TRR) based on fresh weight for each crop in each rotation is given in Table 13.

Table 13. Total radioactive residue (TRR) in rotational crops based on fresh weight.

	Thiacloprid Equivalents (mg/kg)		
	30 day Replant	170 day Replant	354 day Replant
Turnip, tops	0.174	0.088	0.032
Turnip, bulbs	0.014	0.016	0.005
Lettuce	0.111	0.081	0.023
Wheat, immature	0.203	0.128	0.122
Wheat, hay	0.283	0.558	0.135
Wheat, grain	0.101	0.145	0.019
Wheat, straw	1.655	2.595	0.322

In general, all crops had a similar metabolic profile. Metabolites detected in the crops were the thiacloprid amide (M02), 4-OH-thiacloprid amide (M37), 6-chloronicotinic acid (M03), 6-chloropicolyl alcohol (M36), 6-chloropico-lyl urea (M31), the sulfonic acid (M30), the imine (M29) and the sulfonic acid amide (M34), the latter most likely being an artefact formed from the sulfonic acid in the presence of methanol/water. The quantitative distribution of the metabolites is summarised in Table 14.

Table 14. Quantitative distribution of metabolites in rotational crops (values are given in % of the total radioactivity at harvest).

	Replant (days)	M36	M37	M02	M31	M03	M30	M34	M29	Unknown	Sum of metab. not containing thiazolidine	Extracted
Lettuce	30		39.1							52.7		91.9
	170	10.3	23.4	6.1		6.9	6.5		5.4	25.9	17.2	90.5
	354		14.5				17.4		8.6	43.0		83.5
Turnip	30	8.8	20.5	31.5			12.6			5.6	8.8	79.0
bulbs	170	5.0	7.4	17.5		9.8	16.1	7.5	4.8		14.8	82.2
	354											82.0
Turnip	30	12.1	20.4	42.3			6.1			9.9	12.1	90.8
tops	170	11.5	23.2	18.1	6.9		6.5		4.9	16.1	11.5	91.2
	354		45.8	23.3						18.3		87.4
Wheat	30		19.7	31.3			31.4	8.0				90.4
forage	170		11.6	28.2			39.7	14.0				94.8
	354		10.7	13.4			41.7	20.1		7.5		93.4
Wheat	30		13.4	25.1			39.3	8.8				86.6
hay	170		18.0	25.6			23.6	11.9		7.9		91.4
	354		12.7	15.1			34.4	17.3		7.6		87.1
Wheat	30		8.1					31.2		40.3		79.6
grain	170		14.8					27.4		40.7		88.1
	354											49.9
Wheat	30		11.2	13.9			31.2	9.0		17.2		82.5
straw	170		15.6	18.2			32.7	19.1				91.3
	354		15.9	10.5			24.5	17.9		14.1		83.0

Since most of the metabolites detected in the plants were either soil metabolites or their derivatives, it was concluded that the residues in the rotational crops resulted from uptake of soil metabolites which remained stable in the plants or to some degree were further metabolised by the plant. This is summarised in the proposed degradation scheme shown in Figure 4.

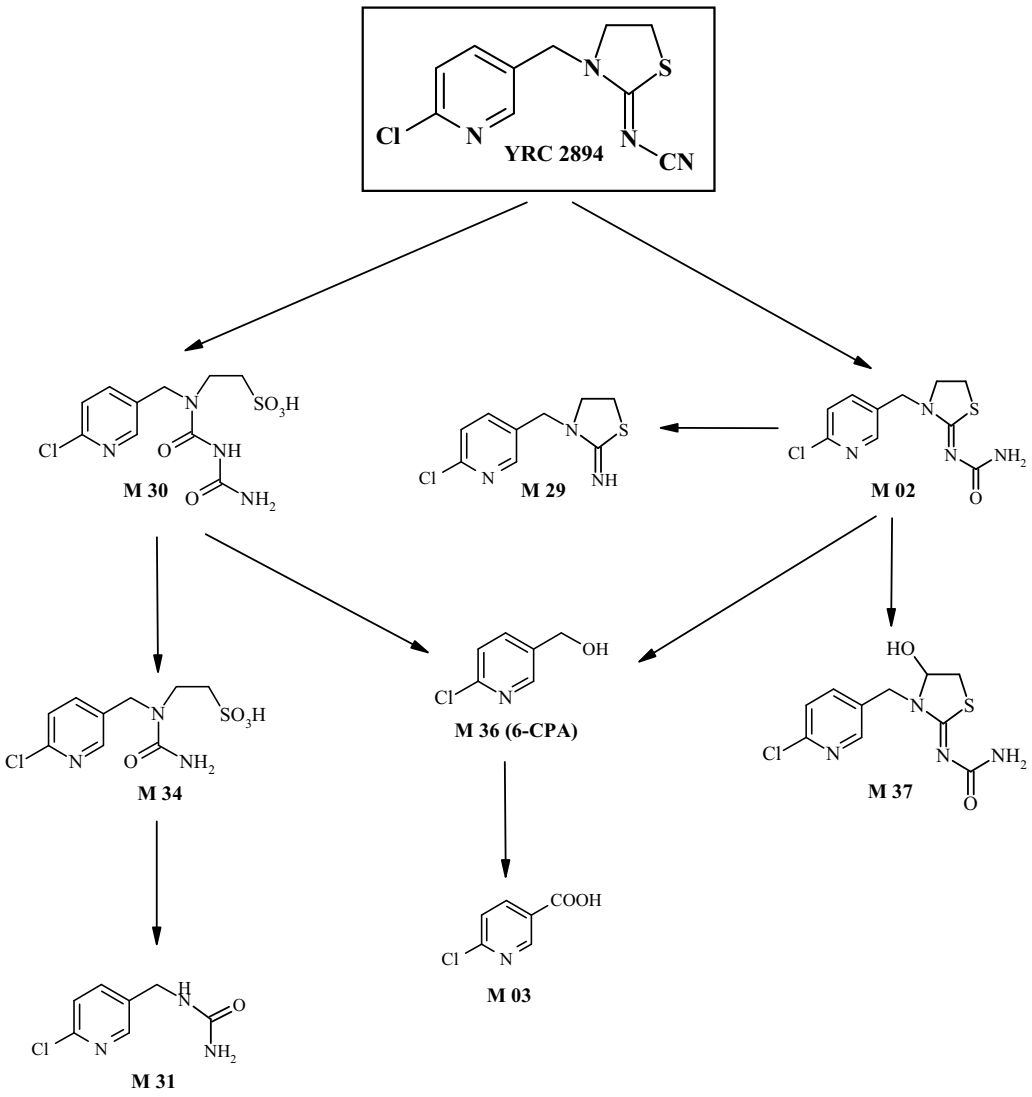


Figure 4. Metabolic pathway of thiacloprid in rotational crops

METHODS OF RESIDUE ANALYSIS

Analytical methods

Plant matrices-enforcement method

An analytical method for plant matrices based on HPLC-UV was reported (Report No.: MR-295/96 (Placke, F. J., 1996) and Report No.: 5438/1494225/T423 (Zyl, P. F. C. van, 2000)). Thiacloprid was extracted from 25 g of plant material with acetone/water (3:1; v:v). After vacuum filtration, an aliquot of the raw extract corresponding to a 5 g sample is concentrated and evaporated to the aqueous remainder. The residues are dissolved in water and partitioned against cyclohexane/ethyl acetate using a ChemElut column. Further clean-up is performed by column chromatography on Florisil and elution with acetonitrile. The residues of thiacloprid parent compound are quantified by reversed phase HPLC with UV detection at 242 nm.

Modifications for citrus: after elution, a partition clean-up step was included using a mixture of hexane and hexane-saturated acetonitrile. Florisil clean-up was not conducted.

The original method was validated by conducting recovery experiments with apple, cucumber, melon, red pepper, peach and tomato. Further recovery experiments were done using citrus matrices. Results obtained were within guideline requirements (recoveries: 70–110%; relative standard deviation (RSD) below 20%).

Control samples were spiked with thiacloprid at fortification levels of 0.02 and 0.2 mg/kg. Recoveries of thiacloprid ranged from 72 to 105% (overall mean: 95%, RSD: 5.8, n=97). Recoveries of thiacloprid for citrus matrices ranged from 77 to 101% (overall mean: 88%, RSD: 11%, n=6). The recoveries were not corrected for interferences. The results are summarised in Table 15 and Table 16.

Table 15. Recovery results from method 00419 for the determination of thiacloprid in plant matrices.

Matrix	Fortification level (mg/kg)	Recovery rate (%) mean	range	RSD (%)	Number of tests
Apple (fruit)	0.02*	101	98-103	1.9	5
	0.2	95	90-99	3.6	5
Apple (dried)	0.02*	82	72-88	10.6	3
	0.2	90	86-93	4.0	3
Apple (juice)	0.02*	98	94-100	3.3	3
Apple (pomace, dry)	0.02*	91	84-98	7.7	3
	0.2	86	97-97	0.0	3
Apple (sauce)	0.02*	98	95-100	2.6	3
	0.2	94	92-98	3.4	3
Cucumber (fruit)	0.02*	92	89-94	3.1	3
	0.2	90	83-96	7.4	3
Melon (peel)	0.02*	90	84-97	6.2	5
	0.2	98	92-101	3.5	5
Melon (pulp)	0.02*	95	92-97	2.7	3
	0.2	96	91-100	4.7	3
Red pepper (fruit)	0.02*	98	97-99	1.2	3
	0.2	95	92-99	3.7	3
Peach (fruit)	0.02*	98	91-103	6.4	3
	0.2	99	96-102	3.1	3
Peach (preserve)	0.02*	99	97-100	1.5	3
	0.2	95	93-96	1.6	3
Tomato (fruit)	0.02*	99	100-105	4.9	3
	0.2	100	97-104	3.6	3
Tomato (paste)	0.02*	98	97-99	1.0	3
	0.2	96	95-97	1.2	3
Tomato (juice)	0.02*	93	89-97	4.3	3
Tomato (preserve)	0.02*	94	90-98	4.3	3
	0.2	91	90-94	2.5	3

*: Limit of quantitation (LOQ), defined by the lowest validated fortification level

Table 16. Recovery results from report no. 5438/1494225/T423 for the determination of thiacloprid in plant matrices.

Matrix	Fortification level (mg/kg)	Recovery rate (%) mean	range	Number of tests
Citrus (peel)	0.04	--	101	1
Citrus (flesh)	0.04	--	98	1
	0.08	--	87	1
	0.16	--	82	1
Citrus (whole fruit)	0.08	--	82	1
	0.20	--	77	1

The chromatographic separation in combination with the preceding clean-up steps allows quantitation of the parent compound without significant matrix interferences. Blank values from control samples were well below 30% of the LOQ.

The limit of quantification (LOQ), defined as the lowest concentration at which an acceptable recovery is obtained, was 0.02 mg/kg of thiacloprid for all crop matrices mentioned above. Matrix interference was minimal as illustrated in the control sample chromatograms (< 10% LOQ).

Amendment E001 to method 00419 (Placke, F. J., 1998) was conducted to validate additional plant matrices for cotton, potato, pear, aubergine (eggplant), zucchini (courgette) and cherry. The principle of the method corresponds to the original method no. 00419 (Placke, F. J., 1996).

The method was validated by conducting recovery experiments with the additional commodities cotton, potato, pear, aubergine, zucchini and cherry. Results obtained were within guideline requirements (recoveries: 70–110%; RSD below 20%). Control samples were spiked with thiacloprid at fortification levels of 0.02 and 0.2 mg/kg. Individual recoveries of thiacloprid ranged from 85 to 103% (mean per crop matrix 90–97%, RSDs 1.5–4.4%, n=3–10). The recoveries were not corrected for interferences. The results obtained are summarised in Table 17.

Table 17. Recovery results for method 00419/E001 for the determination of thiacloprid in plant matrices.

Matrix	Fortification level (mg/kg)	Recovery rate (%) Mean	range	RSD (%)	Number of tests
Cotton (seed)	0.02*	93	92-96	1.7	5
	0.2	96	93-97	1.5	5
Potato (tuber)	0.02*	91	85-93	3.6	5
	0.2	93	91-95	1.6	5
Potato (French fries)	0.02*	94	92-96	1.9	3
	0.2	96	94-99	2.2	3
Cherry (juice)	0.02*	99	97-103	2.6	3
Pear (fruit)	0.02*	96	95-98	1.4	3
	0.2	98	97-99	0.8	3
Aubergine (fruit)	0.02*	89	86-91	2.2	5
	0.2	91	87-93	2.4	5
Zucchini (fruit)	0.02*	95	91-97	3.6	3
	0.2	99	97-100	1.3	3
Cherry (fruit)	0.02*	97	94-100	2.6	3
	0.2	93	91-94	1.6	3

*: LOQ, defined by the lowest validated fortification level

An independent laboratory validation of methods 00419 and 00419/E001 was conducted with the representative matrices apple fruit (high acid content), potato tuber (high water content) and cotton seed (high fat content) (Weber, H., 1998). Minor modifications included using chemicals (acetonitrile, cyclohexane and pure water) from a different manufacturer.

Control samples were spiked with thiacloprid at fortification levels of 0.02 and 0.2 mg/kg. Results obtained were within guideline requirements (recoveries: 70–110%; RSD below 20%, n=5). Individual recoveries of thiacloprid ranged from 79 to 110%. Mean recoveries for each crop ranged from 84 to 101%, with RSDs ranging from 3.2 to 8.8%. Blank values were not used for correcting recoveries. The results obtained are summarised in Table 18.

Table 18. Recovery results from the independent laboratory validation of method 00419 for the determination of thiacloprid in plant matrices (Weber, H., 1998)

Matrix	Fortification level (mg/kg)	Recovery rate (%) mean	range	RSD (%)	Number of tests
Apple (fruit)	0.02*	92	88-95	3.2	5
	0.20	92	83-97	6.1	5
Potato (tuber)	0.02*	84	79-88	4.0	5
	0.20	88	83-94	4.9	5
Cotton (seed)	0.02*	92	84-98	6.4	5
	0.20	94	85-102	7.3	5

*: LOQ, defined by the lowest validated fortification level

The enforcement method 00419 is suitable for the determination of residues of thiacloprid parent compound. The LOQ was 0.02 mg/kg in all analysed crop matrices.

Animal matrices-enforcement methods

Residue analysis of thiacloprid parent compound in animal matrices can be done by HPLC-UV according to Placke, F. J., 1998a (method 00519). The method is suitable as an enforcement method.

Thiacloprid is extracted from animal matrices (tissues, eggs and milk) using a mixture of acetonitrile/water or methanol. For milk samples, partitioning of the extracts against n-hexane is performed to remove fat. The extracts are evaporated to the aqueous remainder. For egg samples, clean-up with a polystyrene column (Chromabond HR-P) is performed. The aqueous remainder is partitioned against cyclohexane/ethyl acetate using a ChemElut column. Further clean-up is performed by column chromatography on Florisil and elution with acetonitrile.

The residues are quantified by reversed phase HPLC with UV-detection at 242 nm. The method was validated by conducting recovery tests with muscle, milk and eggs.

Five control samples were spiked with thiacloprid at fortification levels of 0.02 and 0.2 mg/kg for eggs and muscle, and 0.01 and 0.1 mg/kg for milk, respectively. Recoveries of thiacloprid ranged from 82 to 101% (mean: 93%, relative standard deviation (RSD): 5.0%, n=30). The recoveries were not corrected for interference. Blank values were not used for correcting recoveries. The results obtained are summarised in Table 19.

Table 19. Recovery results for method 00519 for the determination of thiacloprid in animal matrices.

Reference	Matrix	Fortification level (mg/kg)	Recovery rate (%) mean	range	RSD (%)	n
Placke, 1998 HPLC/UV, thiacloprid parent	Milk	0.01*	88	80-91	5.4	5
		0.1	95	90-100	4.2	5
	Muscle	0.02*	90	82-95	5.8	5
		0.2	94	92-101	4.1	5
	Egg	0.02	92	88-96	3.2	5
		0.2	94	93-96	1.2	5
HPLC/UV [confirmatory], thiacloprid parent	Milk	0.01*	87	78-95	8.6	5
		0.1	98	91-104	5.9	5
	Muscle	0.02*	90	80-94	6.2	5
		0.2	93	77-78	1.2	5
	Egg	0.02*	93	88-100	5.2	5
		0.2	94	93-95	0.8	5

*: LOQ, defined by the lowest validated fortification level

An independent laboratory validation of method 00519 was conducted with the representative animal materials milk, egg and meat (Weber, H., 1998a). Duplicates of control- and fortified samples (five each at 0.02 and 0.2 mg/kg, except for milk with five each at 0.01 and 0.1 mg/kg) were extracted and analysed. Minor modifications included the use of chemicals of a different specification.

The method was validated for both, the higher and the lower fortification level. Recoveries at the lower level were in the range of 76 to 89% (mean: 82%; RSD: 6.0%) for egg, 75 to 87% (mean: 80%; RSD: 6.3%) for meat, and 85 to 95% (mean: 90%; RSD: 4.5%) for milk. Recoveries at the higher level were in the range of 76 to 102% (mean: 84%; RSD: 12.0%) for egg, 84 to 90% (mean: 86%; RSD: 2.8%) for meat and 93 to 102% (mean: 98%; RSD: 3.4%) for milk. The overall recoveries were 83% (RSD: 9.2%, n=10) for egg, 83% (RSD: 5.9%, n=10) for meat and 94% (RSD: 5.6%, n=10) for milk. Blank values were not used for correcting recoveries. The results obtained are summarised in Table 20.

Table 20. Recovery results from the independent method validation of method 00519 for the determination of thiacloprid in animal matrices.

Reference	Matrix	Fortification level (mg/kg)	Recovery rate (%) mean	range	RSD (%)	n
Weber, 1998 HPLC/UV - ILV	Milk	0.01* 0.1	90 98	85-95 93-102	4.6 3.5	5 5
	Muscle	0.02* 0.2	80 86	75-87 84-90	6.3 2.8	5 5
	Egg	0.02* 0.2	82 84	76-89 76-102	6.0 12	5 5

*: LOQ, defined by the lowest validated fortification level

Enforcement method 00519 for the determination of residues of thiacloprid parent compound by HPLC-UV in a number of animal matrices was successfully validated by an independent laboratory. The LOQ is 0.01 mg/kg in milk and 0.02 mg/kg in muscle and egg.

Specialised methods- thiacloprid only

For thiacloprid additional specialised methods were presented. A summary of the validation data is given in Table 21.

Schoening (1998, 2001, 2002, 2005, 2005a), Billian, P. and Schoening (2003) and Sur, R. (2000) developed a HPLC-MS/MS method (method 00548) for the analysis of thiacloprid parent compound in various plant matrices. The plant material was extracted with a mixture of acetonitrile/water (1/1, v/v). The residues of thiacloprid parent compound are determined by reversed-phase HPLC on a C18-column using a triple-stage mass spectrometer (HPLC-MS/MS) with an electrospray interface (ESI: TurboIonSpray) operated in the positive ion mode under multiple-reaction monitoring (MRM) conditions.

Ballesteros, C. and Meilland - Berthier, I. (2004, 2005) modified method 00548. Modifications involve a change in the composition of the extraction solvent, and filtration and evaporation steps were replaced by centrifugation.

The original method 00548 was adapted by Clay, S. (2003) to produce a new method for the analysis of thiacloprid parent compound be HPLC-MS(SIM) in a number of plant matrices. The residues of thiacloprid were determined by reversed-phase HPLC on a C18-column using the positive SIM-mode after atmospheric pressure chemical ionisation (APCI). As quantification ion m/z = 253 and as qualifier ion m/z = 255 was used. Quantification was performed using matrix matched standards because signal suppression was observed in matrix standards versus solvent standards.

HPLC/UV based method was presented by Fukuda, T. (1998) for green tea. A comparable method for rice matrices relies on HPLC/UV also (Anon. 2002).

For walnuts Baravelli, P. L. (2003) used a method where the residues of thiacloprid parent compound are determined by reversed-phase HPLC using a triple-stage mass spectrometer (HPLC-MS/MS) with an electrospray interface (ESI: TurboIonSpray) operated in the positive ion mode under multiple-reaction monitoring (MRM) conditions for detection. For quantification the following parent and daughter ion were used: m/z = 230 and m/z = 126, respectively. The daughter ion is used for quantification. Quantitation was done using external standards.

A method for animal matrices was developed by Schoening, R. (1998a). Thiacloprid is extracted from animal tissues using a mixture of acetonitrile/water, and from milk samples with methanol and diluted sulphuric acid. The residues are quantified by reversed phase HPLC with electrospray MS/MS-detection using deuterated thiacloprid as internal standard.

Table 21. Validation data for special analytical methods for the determination of parent thiacloprid residues in food of plant and animal origin.

Reference	Sample	Fortified level, mg/kg	Average recovery [%]	RSD [%]	No. of analyses
Schoening, R., 1998	Apple (fruit)	0.02* 0.2	95 91	4.4 1.7	3 3
	Aubergine (fruit)	0.02* 0.2	86 93	3.3 1.6	3 3
	Cherry (fruit)	0.02* 0.2	88 82	4.7 3.2	3 3
	Cherry (juice)	0.02* 0.2	84 87	2.6 3.8	5 5
	Cotton (seed)	0.02* 0.2	79 81	5.7 9.5	5 5
	Cucumber (fruit)	0.02* 0.2	88 84	1.3 5.5	3 3
	Peach (fruit)	0.02* 0.2	86 89	6.5 3.9	3 3
	Pepper (fruit)	0.02* 0.2	88 85	4.6 5.3	3 3
	Potato (tuber)	0.02* 0.2	92 97	2.5 3.0	5 5
	Potato (green matter)	0.02* 0.2	88 98	2.4 8.5	3 3
	Potato (French fries)	0.02* 0.2	95 93	5.4 4.0	5 5
	Strawberry (fruit)	0.02* 0.2	87 90	3.0 3.2	5 5
	Strawberry (marmalade)	0.02* 0.2	86 90	1.5 2.5	5 5
	Tomato (fruit)	0.02* 0.2	99 95	4.2 4.8	3 3
	Tomato (juice)	0.02* 0.2	89 90	0.9 3.0	5 5
	Tomato (puree)	0.02* 0.2	93 92	1.6 2.6	5 5
	Tomato (paste)	0.02* 0.2	91 88	5.6 1.5	3 3
Schoening, R., 2001	Melon (pulp)	0.02* 0.2	98 94	2.6 1.1	3 3
	Melon (peel)	0.02* 0.2	90 93	12.5 4.1	3 3
	Currant (fruit)	0.02* 0.2	95 94	2.2 5.9	3 3
	Plum (fruit)	0.02* 0.2	96 95	5.1 3.0	3 3
	Sugar beet (leaves)	0.02* 0.2	90 93	13.3 0.6	3 3
	Sugar beet (body)	0.02* 0.2	96 93	2.2 3.3	3 3
	Wheat (grain)	0.02* 0.2	100 99	4.4 1.2	3 3
	Wheat (rest of plant)	0.02* 0.2	98 93	3.6 4.7	3 3

Reference	Sample	Fortified level, mg/kg	Average recovery [%]	RSD [%]	No. of analyses
	Wheat (straw)	0.02* 0.2	94 98	6.9 1.2	3 3
	Barley (grain)	0.02* 0.2	97 95	1.6 1.1	3 3
	Barley (rest of plant)	0.02* 0.2	98 95	2.6 3.7	3 3
	Barley (straw)	0.02* 0.2	96 92	1.2 4.5	3 3
	Pea with pod	0.02* 0.2	100 98	0.0 4.8	3 3
	Pea without pod	0.02* 0.2	97 97	3.3 1.8	3 3
	Pea (pod empty)	0.02* 0.2	101 96	2.1 2.2	3 3
Schoening, R., 2002	Rape (rest of plant, green material)	0.02* 0.2	92 91	3.1 0.6	3 3
	Rape (pod)	0.02* 0.2	96 94	1.2 2.8	3 3
	Rape (seed)	0.02* 0.2	96 92	1.0 0.6	3 3
	Rape (straw)	0.02* 0.2	87 84	4.1 2.5	3 3
	Raspberry (fruit)	0.02* 0.2	96 93	1.6 0.6	3 3
Schoening, R., 2005	Onion (whole plant)	0.01* 0.1	90 97	5.9 1.0	5 3
Schoening, R., 2005a	Leek (whole plant)	0.01* 0.1	94 93	2.7 2.3	3 4
	Zucchini (fruit)	0.02* 0.2	90 88	1.3 2.0	3 3
Sur, R., 2000	Strawberry (fruit)	0.02* 0.2	87 90	3.0 3.2	5 5
	Strawberry (marmalade)	0.02* 0.2	86 90	1.5 2.5	5 5
	Tomato (juice)	0.02* 0.2	89 90	0.9 3.0	5 5
	Tomato (puree)	0.02* 0.2	93 92	1.6 2.6	5 5
	Tomato (paste)	0.02* 0.2	91 88	5.6 1.5	5 5
Billian, P. and Schoening, R., 2003	Bean (bean with pod)	0.01* 0.02 0.20	98 97 98	5.8 1.6 2.9	3 3 3
	Olive (fruit)	0.01* 0.02 0.20	99 89 89	1.5 0.0 1.3	3 3 3
	Olive (pomace wet)	0.01* 0.02 0.20	101 88 87	2.0 0.7 0.7	3 3 3
	Olive (oil)	0.01* 0.20	104 98	1.4 3.4	5 5
	Broccoli (curd)	0.01* 0.02 0.20	97 97 97	2.2 2.0 2.4	5 5 5
	Cauliflower (curd)	0.01* 0.02 0.20	97 97 96	4.3 3.3 0.6	3 3 3
	Head Cabbage (head)	0.01* 0.02 0.20	96 90 96	1.2 1.3 0.6	3 3 3
	Brussels Sprouts	0.01* 0.02 0.20	100 95 94	0.6 0.6 1.1	3 3 3

Reference	Sample	Fortified level, mg/kg	Average recovery [%]	RSD [%]	No. of analyses
	Kohlrabi (leaf)	0.01* 0.20	102 94	2.8 0.6	3 3
	Kohlrabi (corm)	0.01* 0.20	99 94	1.0 1.1	3 3
	Corn (whole plant)	0.01* 0.20	98 98	1.0 2.6	3 3
	Corn (kernel)	0.01* 0.20	99 96	1.7 0.5	5 5
	Corn (cob without husks)	0.01* 0.20	99 97	3.0 0.0	3 3
	Artichoke (head)	0.01* 0.20	100 103	3.0 0.0	3 3
	Lettuce (head)	0.01* 0.20	97 96	1.6 2.6	3 3
	Hazelnut (nut)	0.01* 0.02 0.20	98 95 96	0.6 0.6 1.2	3 3 3
Ballesteros, C. and Meilland - Berthier, I., 2004	Zucchini (fruit)	0.01* 0.10	91 93	1.5 1.1	5 5
	Pepper (fruit)	0.01* 0.10	96 96	3.3 3.8	3 3
Ballesteros, C. and Meilland - Berthier, I., 2005	Sugar beet (body)	0.01* 0.1	99 103	1.5 1.5	3 3
	Sugar beet (leaves with root collar)	0.01* 0.1	93 88	1.6 2.4	3 3
	Tomato (fruit)	0.01* 0.1	104 97	3.6 1.6	3 3
	Field pea	0.01* 0.1	90 90	4.0 1.3	3 3
	Corn (kernel)	0.01* 0.1	84 84	4.1 4.8	3 3
	Corn (whole plant without roots)	0.05* 0.5	76 75	6.8 0.0	3 3
	Artichoke (head)	0.01* 0.1	83 88	1.2 1.3	3 3
	Watermelon (fruit)	0.01* 0.1	115 109	2.2 3.2	3 3
	Lettuce (head)	0.01* 0.1	98 100	4.3 2.6	3 3
	Wheat (grain)	0.01* 0.1	92 88	2.9 1.1	3 3
	Wheat (straw)	0.05* 0.5	89 86	6.9 3.7	3 3
	Barley (grain)	0.01* 0.1	93 93	4.4 3.8	3 3
	Barley (straw)	0.05* 0.5	100 93	2.0 4.4	3 3
Clay, S. 2003	Kiwi whole fruit	0.02* 0.20	87 91	8 8	7 7
	Peach fruit	0.02* 0.02* 0.20 0.20	84 81 93 90	9.0 7.9 5.3 3.6	8 5 8 5
	Sweetcorn	0.02* 0.20	74 82	5.8 4.6	4 5
	Lemon pulp	0.02* 0.20	78 83	6.8 5.7	5 5
	Nectarine fruit	0.02* 0.20	103 101	9 7	5 5

Reference	Sample	Fortified level, mg/kg	Average recovery [%]	RSD [%]	No. of analyses
Fukuda, T. 1998	Green tea (leaf)	0.4*	87	0	2
		0.4	84	0	2
Anon., 2002	Rice grain	5.0	90	1.5	3
		1.0	86	3.2	3
		0.5*	88	4.0	3
	Rice husk	5.0	88	2.0	3
		1.0	84	2.0	3
		0.5*	88	3.1	3
	Rice straw	5.0	91	2.6	3
		1.0	84	4.9	3
		0.5*	88	2.0	3
Baravelli, P. L., 2003	Walnut	0.005*	101.0	-	1
		0.010	86.5	-	1
		0.050	98.6	-	1
		0.100	97.8	-	1
		0.500	83.1	-	1
		1.000	88.0	-	1
		5.000	89.7	-	1
Schoening, R., 1998a	Milk	0.01*	95	1.2	5
		0.1	94	3.2	5
	Muscle	0.02*	98	2.0	5
		0.2	95	3.7	5
	Liver	0.02*	95	2.1	3
		0.2	90	2.9	3
	Kidney	0.02*	90	2.3	3
		0.2	93	2.8	3
	Fat	0.02*	98	2.7	3
		0.2	95	2.4	3

*: LOQ, defined by the lowest validated fortification level

Specialised methods- thiacloprid total residue

For thiacloprid additional methods for the determination of the total residue containing the 6-chloropicolyl moiety were presented. A summary of the validation data is given in Table 21.

Schoening, R. (1999) and Babczinski, P. (1997a) developed a method, where thiacloprid and its metabolites were extracted from plant matrices with an acidic methanol / water mixture. After the clean-up thiacloprid and all metabolites containing the 6-chloropicolyl moiety were oxidised with alkaline potassium permanganate solution to yield 6-chloronicotinic acid. This was followed by acidification and reduction of the excess permanganate and the developed manganese dioxide with sodium bisulfite. The 6-CNA was converted to the corresponding trimethylsilyl ester with MSTFA prior to quantitation by gas chromatography with mass selective detection in the single-ion monitoring mode (GC-MS).

In a comparable method presented by DeHaan, R. A. (1999) and Perez, R. (1999) thiacloprid total residues were extracted with a mixture of methanol and sulfuric acid. Residues were treated with alkaline potassium permanganate, which oxidised thiacloprid and all metabolites containing the 6-CNA. The 6-CNA was extracted from the oxidised mixture and derivatized. The derivative, trimethylsilyl 6-chloronicotinate, was measured by gas chromatography/mass spectroscopy selected ion monitoring (GC/MS-SIM).

Orosz, F. (2000 and 2000a) validated the method for the analysis of thiacloprid total residue in rape seeds and sunflower.

A thiacloprid total residue method was presented by Schoening, R. (1998b) using GC-MS after oxidisation to 6-CNA and derivatization with MSTFA.

Table 22. Validation data for special analytical methods for the determination of total thiacloprid residues in food of plant and animal origin.

Reference	Sample	Fortified level, mg/kg	Average recovery [%]	RSD [%]	No. of analyses
Schoening, R., 1999 and Babczinski, P., 1997a	Cotton (seed)	0.05*	81	4.1	5
		0.0507[1]	85	3.2	5
		0.5	100	4.4	5
	Potato (tuber)	0.05*	84	8.1	5
		0.0507[1]	86	2.9	5
		0.5	91	0.5	5
DeHaan, R. A., 1999	Apples (whole fruit)	Thiacloprid:			
		0.01*	88	7.3	9
		0.05	87	9.7	9
		0.3	90	1.7	3
		4-hydroxy-thiacloprid :			
		0.01*	61	7.7	13
		0.05	69	8.4	8
		0.3	61	2.3	3
	Apples (juice)	Thiacloprid:			
		0.01*	85	5.0	4
		0.25	84	8.4	3
		4-hydroxy-thiacloprid:			
		0.01*	68	4.2	3
		0.25	62	3.8	3
	Apples (wet pomace)	Thiacloprid:			
		0.01*	81	10.3	3
		0.6	89	4.6	3
		4-hydroxy-thiacloprid:			
		0.01*	58	5.7	3
		0.6	61	3.6	3
	Pears (whole fruit)	Thiacloprid:			
		0.01*	100	10.6	5
		0.05	87	13.3	7
		0.4	79	14.4	3
		4-hydroxy-thiacloprid:			
		0.01*	56	10.3	10
		0.05	62	15.8	6
		0.4	59	8.9	3
	Cotton (meal)	Thiacloprid:			
		0.05*	80	-	1
		6-CNA:			
		0.05*	98	-	1
		mix of equimolar amounts of thiacloprid and 6-CNA:			
		0.05*	88	-	1
	Cotton (hulls)	Thiacloprid:			
		0.05*	66	-	1
		0.2	65	-	1
		6-CNA:			
		0.05*	100	-	1
		0.2	87	-	1
		mix of equimolar amounts of thiacloprid and 6-CNA:			
		0.05*	66	-	1
		0.2	78	-	1

Reference	Sample	Fortified level, mg/kg	Average recovery [%]	RSD [%]	No. of analyses
	Cotton (refined oil)	Thiacloprid:			
		0.05*	94	-	1
		0.5	86	-	1
		6-CNA:			
		0.05*	100	-	1
		0.5	95	-	1
		mix of equimolar amounts of thiacloprid and 6-CNA:			
		0.05*	94	-	1
		0.5	91	-	1
	Cotton (undelinted seed)	Thiacloprid:			
		0.05*	83	15.1	4
		0.1	78	6.5	5
		0.5	104	-	1
		1.0	81	7.1	2
		6-CNA:			
		0.05*	98	-	1
		0.5	97	-	1
		1.0	88	-	1
		mix of equimolar amounts of thiacloprid and 6-CNA:			
		0.05*	82	-	1
		0.5	102	-	1
		1.0	78	-	1
	Cotton (gin trash)	Thiacloprid:			
		0.05*	94	16.9	2
		0.5	80	-	1
		5.0	110	-	1
		11	73	-	1
		15	97	-	1
		6-CNA:			
		0.05*	106	-	1
		11	61	-	1
		mix of equimolar amounts of thiacloprid and 6-CNA:			
		0.05*	86	-	1
		11	71	-	1
Perez, R., 1999	Cotton (seed)	Thiacloprid:			
		0.05*	83	9.9	2
		1.0	77	5.7	2
		6-CNA:			
		0.05*	89	5.7	2
		1.0	124	0.0	2
Orosz, F. 2000	Rape (seed)	0.02*	89	20.8	7
		0.1	88	17.3	4
		0.5	86	6.3	4
Orosz, F. 2000a	Sunflower (seed)	0.02*	92	15.3	7
		0.1	93	8.8	4
		0.5	83	6.2	4
Schoening, R., 1998b	Milk	Total residue:			
		0.01*	92	2.0	5
		0.1	85	3.1	5
		6-CP-urea sulfoxide:			
		0.01*	86	3.5	3
	Muscle	0.02*	82	6.6	5
		0.2	88	2.5	5
	Liver	0.02*	87	1.3	3

Reference	Sample	Fortified level, mg/kg	Average recovery [%]	RSD [%]	No. of analyses
		0.2	94	1.0	3
	Kidney	Total residue:			
		0.02*	85	4.2	3
		0.2	84	1.3	3
		6-CP-urea sulfoxide:			
		0.02*	64	3.5	3
	Fat	0.02*	102	3.7	3
		0.2	91	3.5	3

*: LOQ, defined by the lowest validated fortification level

1: Fortification with a mixture of amide-thiacloprid and 6-CNA at 0.02 mg/kg, each, corresponding to 0.0507 mg/kg thiacloprid

Specialised methods- thiacloprid parent and metabolites

An LC-MS/MS method for measuring thiacloprid, thiacloprid-amide, 4-hydroxy-thiacloprid-amide and thiacloprid-sodium sulfonate was developed by Moore, S. M., 2002 and Harbin, A. M., 2004. The analytes were extracted using methanol/water (3:1) followed by 18C solid phase extraction. The quantitation was based on comparison of daughter ion transitions between the analytes and their deuterated analogs, which were used as the internal standards.

Table 23. Validation data for special analytical methods for the determination of thiacloprid residues and its metabolites in food of plant and animal origin.

Reference	Sample	Fortified level, mg/kg	Average recovery [%]	RSD [%]	No. of analyses
Moore, S. M., 2002	Soybean seed	Thiacloprid:			
		0.01*	98	4.8	10
		0.1	95	0.58	3
		thiacloprid-amide:			
		0.01*	96	4.1	10
		0.1	80	2.9	3
		4-hydroxy-thiacloprid-amide:			
		0.01*	102	2.5	10
		0.1	91	2.1	3
		thiacloprid-sodium-sulfonate:			
		0.01*	108	2.2	10
		0.1	94	1.5	3
	Soybean forage	Thiacloprid:			
		0.01*	97	5.0	8
		0.1	102	1.5	3
		thiacloprid-amide:			
		0.01*	100	3.2	8
		0.1	100	1.0	3
		4-hydroxy-thiacloprid-amide:			
		0.01*	101	3.0	8
		0.1	95	2.0	3
		thiacloprid-sodium-sulfonate:			
		0.01*	111	2.9	8
		0.1	92	0.58	3

Reference	Sample	Fortified level, mg/kg	Average recovery [%]	RSD [%]	No. of analyses
	Soybean hay	Thiacloprid:			
		0.01*	94	3.1	8
		0.1	91	4.7	3
		thiacloprid-amide:			
		0.01*	103	6.2	8
		0.1	98	3.6	3
		4-hydroxy-thiacloprid-amide:			
		0.01*	100	1.8	8
		0.1	100	1.2	3
		thiacloprid-sodium-sulfonate:			
		0.01*	105	2.7	8
		0.1	91	1.5	3
	Wheat grain	Thiacloprid:			
		0.01*	93	2.8	9
		0.1	96	2.3	3
		thiacloprid-amide:			
		0.01*	94	3.5	9
		0.1	93	2.1	3
		4-hydroxy-thiacloprid-amide:			
		0.01*	92	4.4	9
		0.1	93	2.1	3
		thiacloprid-sodium-sulfonate:			
		0.01*	99	5.9	9
		0.1	93	1.2	3
	Wheat forage	Thiacloprid:			
		0.01*	94	2.2	9
		0.1	85	2.1	3
		thiacloprid-amide:			
		0.01*	94	2.9	9
		0.1	85	1.7	3
		4-hydroxy-thiacloprid-amide:			
		0.01*	94	3.8	9
		0.1	85	1.5	3
		thiacloprid-sodium-sulfonate:			
		0.01*	107	4.4	9
		0.1	92	3.5	3
	Wheat hay	Thiacloprid:			
		0.01*	94	4.3	9
		0.1	91	1.0	3
		thiacloprid-amide:			
		0.01*	93	4.5	9
		0.1	91	2.1	3
		4-hydroxy-thiacloprid-amide:			
		0.01*	95	4.3	9
		0.1	87	0.58	3
		thiacloprid-sodium-sulfonate:			
		0.01*	102	6.5	9
		0.1	91	1.0	3

Reference	Sample	Fortified level, mg/kg	Average recovery [%]	RSD [%]	No. of analyses
Harbin, A. M., 2004	Pecan nutmeat	Thiacloprid: 0.01*	97	3.2	7
		thiacloprid-amide: 0.01*	97	4.3	7
		4-hydroxy-thiacloprid-amide: 0.01*	98	3.2	7
	Almond nutmeat	Thiacloprid: 0.01*	96	1.9	6
		thiacloprid-amide: 0.01*	96	6.3	6
		4-hydroxy-thiacloprid-amide: 0.01*	97	2.3	6
	Almond hulls	Thiacloprid: 0.01*	79	7.2	9
		thiacloprid-amide: 0.01*	86	3.0	9
		4-hydroxy-thiacloprid-amide: 0.01*	89	5.8	9

Stability of residues in stored analytical samples

Storage stability was examined in three different water-containing crop commodities (apple fruit, tomato fruit, melon peel), and in cotton seed and potato tuber, representing oil-containing and starch-containing matrices, up to a period of 18 months. In a follow-up study, freezer storage stability was demonstrated for the extended period of 24 months in the additional crops tobacco, wheat, rape, pea, currant and potato. A summary of the results is presented in Table 24.

In the study by Placke, F.J. (1997), samples of apple, tomatoes and melons were fortified with 0.2 mg/kg thiacloprid each. Immediately after fortification, a sample from each matrix was taken to determine the initial residues (fortification level). The remaining fortified samples were deep frozen (approx. -20°C) and analysed after nominal storage intervals of 1, 3, 6, 12 and 18 months.

Schoening, R. (2000 & 2005a) examined the storage stability of thiacloprid using samples of potato tubers, cotton seed, wheat straw, rape seed, peas with pods, currants and tobacco leaves. Tobacco leaf was fortified with a level of 2 mg/kg, all other samples with 0.2 mg/kg. The remaining fortified samples were deep frozen (approximately -20°C) and analysed after nominal storage intervals of 1, 3, 6, 12, 18 and 24 months.

Table 24. Stability of residues in stored analytical samples.

Reference	Commodity and fortification level	Storage Interval (days)	Procedural recovery (%)	Thiacloprid in stored sample, uncorrected (%)
Placke, F.J. 1997	Tomato, fruit 0.2 mg/kg	0	-	90.5
		33	76.5	78.7
		90	96.9	95.6
		180	92.3	95.2
		365	95.8	96.2
		540	96.2	97.6
	Apple, fruit 0.2 mg/kg	0	-	89.7
		32	95.0	99.5
		89	98.9	98.9
		180	92.1	87.7
		364	94.2	95.0
		539	96.1	97.2

Reference	Commodity and fortification level	Storage Interval (days)	Procedural recovery (%)	Thiacloprid in stored sample, uncorrected (%)
	Melon, peel 0.2 mg/kg	0	-	83.8
		29	87.1	88.4
		90	94.4	97.7
		182	89.3	91.2
		365	92.3	91.8
		540	95.4	94.6
Schoening, R. 2000	Potato, tuber 0.2 mg/kg	0	-	98.6
		75	99.9	101.2
		152	93.3	94.5
		300	94.5	95.7
		540	100.3	98.9
	Cotton, seed 0.2 mg/kg	0	-	93.9
		75	103.2	94.5
		152	99.3	97.0
		300	95.0	100.0
		540	99.1	93.4
Schoening, R. 2005a	Potato, tuber 0.2 mg/kg	0	-	105
		30	110	108
		90	100	101
		180	99	98
		360	95	96
		540	102	98
		730	96	92
	Wheat, straw 0.2 mg/kg	0	-	104
		30	106	100
		90	97	97
		180	100	96
		360	102	96
		540	91	91
		730	93	91
	Tobacco, leafs dry 2 mg/kg	0	-	104
		30	103	102
		90	92	97
		180	102	102
		360	105	106
		540	93	92
		730	95	96
	Peas with pod 0.2 mg/kg	0	-	107
		30	108	106
		90	104	101
		180	105	101
		360	102	99
		540	100	92
		730	93	102
	Rape seed 0.2 mg/kg	0	-	105
		30	107	106
		90	101	96
		180	100	95
		360	101	103
		540	95	91
		730	95	97
	Currant, fruits 0.2 mg/kg	0	-	106
		30	108	108
		90	102	99
		180	101	94
		360	102	93
		540	95	85
		730	98	90

USE PATTERNS

Thiacloprid is registered globally as an insecticide and is used for foliar treatment on a wide variety of crops. The information available to the Meeting on registered uses relevant to the supervised field data is summarised in Table 25. It is based on the labels or translation of labels provided by the manufacture.

Additional uses were also submitted by the Queensland Government Department of Primary Industries and Fisheries, Australia.

Table 25. Registered uses of thiacloprid.

Crop	Country	Formulation, ai %	Application rate		No. Per season	PHI (days)
			kg ai/ha	kg ai/hL		
Almond	United Kingdom	SC, 48	0.18	0.018	2	
American upland	Israel	SC, 48	0.19	0.095 - 0.19	1	21
Apple	Argentina	SC, 48	0.072 – 0.084 kg ai/m canopy height	0.004 - 0.006 per m canopy height	2	14
Apple	Belgium	SC, 48	0.06 per m canopy height	0.004 - 0.006 per m canopy height	2	14
Apple	Chile	SC, 48		0.0096	2	1
Apple	Croatia	SC, 48		0.0096	2	14
Apple	Cyprus	SC, 48		0.0096 - 0.014	2	14
Apple	Czech Republic	SC, 48	0.04 - 0.06 per m canopy height	0.004 - 0.006 per m canopy height	2	14
Apple	Estonia	SC, 48	0.072 - 0.096	0.00960 - 0.019	1	14
Apple	Georgia	SC, 48	0.096 - 0.14			
Apple	Greece	SC, 48	0.096 - 0.18	0.0096 - 0.012	2	14
Apple	Hungary	SC, 48		0.014	3	14
Apple	Israel	SC, 48	0.2	0.01	2	3
Apple	Italy	SC, 48	0.18	0.012	2	14
Apple	Japan	WG, 30	(1.05)	0.015	3	7
Apple	Latvia	SC, 48		0.072 - 0.096	2	14
Apple	Lithuania	SC, 48		0.048 - 0.096	3	14
Apple	Mexico	SC, 48	Min. 0.14	0.014		30
Apple	Morocco	SC, 48	(0.014)	0.0067		14
Apple	Netherlands	SC, 48	0.12	0.012	2	14
Apple	New Zealand	SC, 48	0.17	0.029	2	42
Apple	Poland	SC, 48	0.096	0.013 - 0.019	1	14
Apple	Portugal	SC. 48	0.096	0.0096	2	14
Apple	Romania	SC, 48	0.144	0.0096		
Apple	Russia	SC, 48		0.014	2	28
Apple	Slovakia	SC, 48	0.096	(0.0096)	2	14
Apple	Slovenia	SC, 48	0.144	0.0096	2	14
Apple	South Africa	SC, 48		0.0072	4	14
Apple	South Korea	SC, 10		0.0005	5	21
Apple	Spain	SC, 48	(0.18)	0.0096	2	14
Apple	Tunisia	SC, 48		0.0096	3	30
Apple	Turkey	SC, 48		0.0096		14
Apple	United Kingdom	SC, 48	0.18	0.012 - 0.018	2	14
Apricot	Australia	SC, 48	0.27	0.018	3	14
Apricot	Cyprus	SC, 48		0.0096	2	14
Apricot	Germany	SC, 48	0.048 per m canopy height	0.0032	1	21
Apricot	Italy	SC, 48	0.18	0.012		14
Apricot	Slovenia	SC, 48	0.18	0.012	2	14
Apricot	Spain	SC, 48		0.014	2	14
Aubergine	Brazil	SC, 48	0.096	(0.0096)		7
Aubergine	Cyprus	SC, 48		0.014	3	3
Aubergine	Greece	SC, 48	0.216	0.014	2	3
Aubergine	Italy	SC, 48	0.14	0.014		3

Crop	Country	Formulation, ai %	Application rate		No. Per season	PHI (days)
			kg ai/ha	kg ai/hL		
Aubergine	Japan	WG, 30	0.23	0.0075	3	1
Aubergine	Kenya	SC, 48	0.19	(0.019)	4	3
Aubergine	Netherlands (greenhouse use)	SC, 48		0.012	4	1
Aubergine	Netherlands (greenhouse use, drip irrigation)	SC, 48	0.12		1	3
Aubergine	Slovenia	SC, 48	0.21	0.014	2	3
Aubergine	South Korea	SC, 10		0.00025	3	3
Aubergine	Spain	SC, 48		0.014	3	3
Aubergine	United Kingdom	SC, 48	0.22	max. 0.022	3	3
Barley	Romania	SC, 48	0.048	0.012 - 0.016	1	
Bell Pepper	Tunisia	SC, 48		0.014	2	3
Berries	Switzerland	SC, 48	0.096	0.0096	2	21
Bilberry	Netherlands	SC, 48	0.14	0.012	2	3
Bilberry	United Kingdom	SC, 48	0.12	0.024	3	3
Black currants	Latvia	SC, 48	0.072	(0.019)	2	14
Blackberry	Germany	SC, 48	0.096	0.0096	2	14
Blackberry	Netherlands	SC, 48	0.14	0.012	2	3
Blackberry	Poland	SC, 48	0.072	0.0096	2	5
Blackberry	United Kingdom	SC, 48	0.12	0.024	3	3
Blueberry	Germany	SC, 48	0.096	0.0096	2	14
Blueberry	Netherlands	SC, 48	0.14	0.012	2	3
Blueberry	United Kingdom	SC, 48	0.12	0.024	3	3
Cherry	Australia	SC, 48	0.27	0.018	3	14
Cherry	Croatia	SC, 48	0.096	0.0048	2	14
Cherry	Cyprus	SC, 48		0.0096	2	14
Cherry	Czech Republic	SC, 48	0.096	0.0096	2	14
Cherry	Germany	SC, 48	0.048 per m canopy height	0.0032	2	14
Cherry	Hungary	SC, 48		0.0067	3	14
Cherry	Japan	WG, 30	(1.05)	0.015	2	1
Cherry	Netherlands	SC, 48	0.12	0.012	2	14
Cherry	Poland	SC, 48	0.096	0.013 - 0.019	1	14
Cherry	Poland	SC, 49	0.048	0.007 - 0.01	1	14
Cherry	Romania	SC, 48	0.096	0.0096	2	14
Cherry	Slovakia	SC, 48	0.096			14
Cherry	Slovenia	SC, 48	0.18	0.012	2	14
Cherry	Turkey	SC, 48		0.0096		14
Cherry	United Kingdom	SC, 48	0.15	0.015	2	14
Chestnut	United Kingdom	SC, 48	0.18	0.018	2	
Chili	Belize	SC, 10	0.1	(0.02)		21
Chili	Costa Rica	SC, 10	0.1	(0.02)		21
Chili	Dom. Republic	SC, 10	0.1	(0.02)		21
Chili	El Salvador	SC, 10	0.1	(0.02)		21
Chili	Guatemala	SC, 10	0.1	(0.02)		21
Chili	Honduras	SC, 10	0.1	(0.02)		21
Chili	Kenya	SC, 48	0.19	(0.019)	4	3
Chili	Netherlands (greenhouse use)	SC, 48		0.012	4	1
Chili	Netherlands (greenhouse use, drip irrigation)	SC, 48	0.12		1	3
Chili	Nicaragua	SC, 10	0.1	(0.02)		21
Chili	Panama	SC, 10	0.1	(0.02)		21
Citrus	Brazil	SC, 48		0.0048		21
Citrus	Georgia	SC, 48	0.19 – 0.24			
Citrus	South Africa	SC. 48		0.0067		
Citrus	South Korea	SC, 10		0.0024	3	14
Cotton	Argentina	SC, 48	0.096		1	
Cotton	Belize	SC, 10	0.1	(0.02)		21
Cotton	Brazil	SC, 48	0.096	0.096	1	28

Crop	Country	Formulation, ai %	Application rate		No. Per season	PHI (days)
			kg ai/ha	kg ai/hL		
Cotton	Costa Rica	SC, 10	0.1	(0.02)		21
Cotton	Dom. Republic	SC, 10	0.1	(0.02)		21
Cotton	El Salvador	SC, 10	0.1	(0.02)		21
Cotton	Greece	SC, 48	0.072	0.01	2	21
Cotton	Guatemala	SC, 10	0.1	(0.02)		21
Cotton	Honduras	SC, 10	0.1	(0.02)		21
Cotton	India	SC, 24	0.14	0.024		52
Cotton	Israel	SC, 48	0.19	0.095 - 0.19	1	21
Cotton	Mexico	SC, 48	0.072		1	14
Cotton	Nicaragua	SC, 10	0.1	(0.02)		21
Cotton	Panama	SC, 10	0.1	(0.02)		21
Cotton	Peru	SC, 48	(0.096)	0.024		
Cotton	Spain	SC, 48	0.096		3	21
Cotton	Turkey	SC, 48	0.12			28
Cotton	USA	WG, 70	0.1		6	14
Cotton	USA	SC, 48	0.089		6	14
Courgette	Greece	SC, 48	0.21	0.014	2	3
Courgette	Italy	SC, 48	0.14	0.014	2	3
Courgette	Kenya	SC, 48	0.19	0.019	3	3
Courgette	Netherlands	SC, 48		0.012	4	1
Courgette	Slovenia	SC 48	0.21	0.014	2	3
Courgette	Spain	SC, 48		0.014	3	3
Courgette	United Kingdom	SC, 48	0.22	min. 0.022	3	3
Cranberry	Netherlands	SC, 48	0.14	0.012	2	3
Cranberry	United Kingdom	SC, 48	0.12	0.024	3	3
Cucumber	Belize	SC, 10	0.1	(0.02)		21
Cucumber	Brazil	SC, 48	0.096	0.0048		7
Cucumber	Costa Rica	SC, 10	0.1	(0.02)		21
Cucumber	Croatia	SC, 48	0.14		2	4
Cucumber	Cyprus	SC, 48		0.014	3	3
Cucumber	Dom. Republic	SC, 10	0.1	(0.02)		21
Cucumber	El Salvador	SC, 10	0.1	(0.02)		21
Cucumber	Georgia	SC, 48	0.15			
Cucumber	Greece	SC, 48	0.14	0.0096	2	3
Cucumber	Guatemala	SC, 10	0.1	(0.02)		21
Cucumber	Honduras	SC, 10	0.1	(0.02)		21
Cucumber	Italy	SC, 48	0.14	0.014	2	3
Cucumber	Kenya	SC, 48	0.19	0.019	3	3
Cucumber	Netherlands	SC, 48	0.12	0.012	4	1
Cucumber	Nicaragua	SC, 10	0.1	(0.02)		21
Cucumber	Panama	SC, 10	0.1	(0.02)		21
Cucumber	Slovakia	SC, 48		0.014		3
Cucumber	Slovenia	SC, 48	0.21	0.014	2	3
Cucumber	South Korea	SC, 10		0.0005	3	3
Cucumber	Spain	SC, 48		0.014	3	3
Cucumber	United Kingdom	SC, 48	0.22	min. 0.022	3	3
Currants	Germany	SC, 48	0.096	0.0096	1	21
Currants	Netherlands	SC, 48	0.14	0.012	2	3
Currants	United Kingdom	SC, 48	0.12	0.024	3	3
Garlic	Belize	SC, 10	0.1	(0.02)		21
Garlic	Brazil	SC, 48	(0.077)	0.0096		21
Garlic	Costa Rica	SC, 10	0.1	(0.02)		21
Garlic	Dom. Republic	SC, 10	0.1	(0.02)		21
Garlic	El Salvador	SC, 10	0.1	(0.02)		21
Garlic	Guatemala	SC, 10	0.1	(0.02)		21
Garlic	Honduras	SC, 10	0.1	(0.02)		21
Garlic	Nicaragua	SC, 10	0.1	(0.02)		21
Garlic	Panama	SC, 10	0.1	(0.02)		21
Gherkin	Greece	SC, 48	0.21	0.014	2	3
Gherkin	Netherlands	SC, 48		0.012	4	1
Gooseberry	Germany	SC, 48	0.096	0.0096	1	21
Gooseberry	Latvia	SC, 48	0.072	(0.019)	2	14

Crop	Country	Formulation, ai %	Application rate		No. Per season	PHI (days)
			kg ai/ha	kg ai/hL		
Gooseberry	Netherlands	SC, 48	0.14	0.012	2	3
Gooseberry	United Kingdom	SC, 48	0.12	0.024	3	3
Grains	Latvia	SC, 48	0.1		2	14
Grape	Japan	WG, 30	0.53	0.0075	2	21
Green Pepper	Japan	WG, 30	0.23	0.0075	3	1
Hazelnut	Germany	SC, 48	0.096	0.0096	2	
Hazelnut	Turkey	SC, 48		0.012		21
Hazelnut	United Kingdom	SC, 48	0.18	0.018	2	
Japan Apricot	Japan	WG, 30	0.53	0.0075	2	7
Japan pear	Japan	WG, 30	(1.05)	0.015	3	7
Kiwi	New Zealand	SC, 48		0.0096	2	pre-flowering app.
Loganberry	Netherlands	SC, 48	0.14	0.012	2	3
Maize	Belize	SE, 11	0.1	(0.02)		21
Maize	Costa Rica	SE, 11	0.1	(0.02)		21
Maize	Dom. Republic	SE, 11	0.1	(0.02)		21
Maize	El Salvador	SE, 11	0.1	(0.02)		21
Maize	Guatemala	SE, 11	0.1	(0.02)		21
Maize	Honduras	SE, 11	0.1	(0.02)		21
Maize	Nicaragua	SE, 11	0.1	(0.02)		21
Maize	Panama	SE, 11	0.1	(0.02)		21
Maize	Romania	SC, 48	0.048	0.012 - 0.016	1	
Mandarin	Croatia	SC, 48		0.038	2	21
Mandarin	Peru	SC, 48		0.014 – 0.024	1	
Marrow	Spain	SC, 48		0.0067	3	3
Melon	Belize	SC, 10	0.1	(0.02)		21
Melon	Brazil	SC, 48	0.096	0.0048		7
Melon	Costa Rica	SC, 10	0.1	(0.02)		21
Melon	Croatia	SC, 48	0.14		2	4
Melon	Dom. Republic	SC, 10	0.1	(0.02)		21
Melon	El Salvador	SC, 10	0.1	(0.02)		21
Melon	Greece	SC, 48	0.21	0.014	2	3
Melon	Guatemala	SC, 10	0.1	(0.02)		21
Melon	Honduras	SC, 10	0.1	(0.02)		21
Melon	Israel	SC, 48	0.19	0.026 - 0.128	2	10
Melon	Italy	SC, 48	0.14	0.014	2	3
Melon	Japan	WG, 30	0.45	0.015	3	1
Melon	Kenya	SC, 48	0.19	0.019	3	3
Melon	Nicaragua	SC, 10	0.1	(0.02)		21
Melon	Panama	SC, 10	0.1	(0.02)		21
Melon	Spain	SC, 48	0.14	0.0067	3	3
Mirabelle	United Kingdom	SC, 48	0.15	0.015	2	14
Nectarine	Argentina	SC, 48		0.0096	2	14
Nectarine	Australia	SC, 48	0.27	0.018	3	14
Nectarine	Chile	SC, 48		0.0096	2	1
Nectarine	Cyprus	SC, 48		0.0096	2	14
Nectarine	Greece	SC, 48	0.108	0.0072	2	14
Nectarine	Israel	SC, 48	0.2	0.01	1	30
Nectarine	South Africa	SC, 48		0.0034	3	60
Nectarine	Spain	SC, 48		0.014	2	14
Oats	Romania	SC, 48	0.048	0.012 - 0.016	1	
Oilseed rape	Czech Republic	SC, 48	0.096	0.016 - 0.048	2	
Oilseed rape	Hungary	SC, 48	0.048	0.012 - 0.018		30
Oilseed rape	Romania	SC, 48	0.048	0.012 - 0.018	1	
Oilseed rape	Slovakia	SC, 48	0.096			
Oilseed rape	Switzerland	SC, 48	0.096		2	21
Oilseed rape	United Kingdom	OD, 24	0.14	0.035 - 0.07	1	30
Onion	Belize	SC, 10	0.1	(0.02)		21
Onion	Brazil	SC, 48	(0.077)	0.0096		21
Onion	Costa Rica	SC, 10	0.1	(0.02)		21
Onion	Dom. Republic	SC, 10	0.1	(0.02)		21
Onion	El Salvador	SC, 10	0.1	(0.02)		21

Crop	Country	Formulation, ai %	Application rate		No. Per season	PHI (days)
			kg ai/ha	kg ai/hL		
Onion	Guatemala	SC, 10	0.1	(0.02)		21
Onion	Honduras	SC, 10	0.1	(0.02)		21
Onion	Nicaragua	SC, 10	0.1	(0.02)		21
Onion	Panama	SC, 10	0.1	(0.02)		21
Orange	Croatia	SC, 48		0.038	2	21
Paddy rice	India	SC, 24	0.12	0.024		30
Peach	Argentina	SC, 48		0.0096	2	14
Peach	Australia	SC, 48	0.27	0.018	3	21
Peach	Chile	SC, 48		0.0096	2	1
Peach	Croatia	SC, 48	0.096	0.0048	2	21
Peach	Cyprus	SC, 48		0.0096	2	14
Peach	Germany	SC, 48	0.048 per m canopy height	0.0032	1	21
Peach	Greece	SC, 48	0.108	0.0072	2	14
Peach	Israel	SC, 48	0.2	0.01	1	30
Peach	Italy	SC, 48	0.18	0.012		14
Peach	Japan	WG, 30	(1.05)	0.015	3	7
Peach	Slovenia	SC, 48	0.18	0.012	2	14
Peach	South Africa	SC, 48		0.0034	3	60
Peach	South Korea	SC, 10		0.0005	3	14
Peach	Spain	SC, 48		0.014	2	14
Peach	Turkey	SC, 48		0.0096		14
Pear	Argentina	SC, 48	0.072 – 0.084 kg ai/m canopy height	0.004 - 0.006 per m canopy height	2	14
Pear	Belgium	SC, 48	0.06 per m canopy height	0.004 - 0.006 per m canopy height	2	14
Pear	Chile	SC, 48		0.0096	2	1
Pear	Estonia	SC, 48	0.072 - 0.096	0.00960 - 0.019	1	14
Pear	Greece	SC, 48	0.096 - 0.18	0.0096 - 0.012	2	14
Pear	Italy	SC, 48	0.18	0.012	2	14
Pear	Japan	WG, 30	1.05	0.015	3	7
Pear	Latvia	SC, 48		0.072 - 0.096	2	14
Pear	Netherlands	SC, 48	0.12	0.12	2	14
Pear	Portugal	SC. 48	0.096	0.0096	2	14
Pear	South Africa	SC, 48		0.0072	4	14
Pear	South Korea	SC, 10		0.0005	5	21
Pear	Spain	SC, 48	(0.18)	0.0096	2	14
Pepper	Belize	SC, 10	0.1	(0.02)		21
Pepper	Brazil	SC, 48	0.096	0.0096		7
Pepper	Costa Rica	SC, 10	0.1	(0.02)		21
Pepper	Croatia	SC, 48	0.14	0.014	2	7
Pepper	Cyprus	SC, 48		0.014	3	3
Pepper	Dom. Republic	SC, 10	0.1	(0.02)		21
Pepper	El Salvador	SC, 10	0.1	(0.02)		21
Pepper	Greece	SC, 48	0.216	0.014	2	3
Pepper	Guatemala	SC, 10	0.1	(0.02)		21
Pepper	Honduras	SC, 10	0.1	(0.02)		21
Pepper	Italy	SC, 48	0.14	0.014		3
Pepper	Kenya	SC, 48	0.19	0.019	3	3
Pepper	Netherlands (greenhouse use)	SC, 48	0.12	0.012	4	1
Pepper	Netherlands (greenhouse use, drip irrigation)	SC, 48	0.0096 kg ai per 1000 plants		1	3
Pepper	Nicaragua	SC, 10	0.1	(0.02)		21
Pepper	Panama	SC, 10	0.1	(0.02)		21
Pepper	Slovakia	Sc, 48		0.014		3
Pepper	Slovenia	SC, 48	0.21	0.014	2	3
Pepper	Spain	SC, 48		0.014	3	3
Pepper	United Kingdom	SC, 48	0.22	max. 0.022	3	3
Pistachio	Turkey	SC, 48		0.012		21

Crop	Country	Formulation, ai %	Application rate		No. Per season	PHI (days)
			kg ai/ha	kg ai/hL		
Plum	Argentina	SC, 48		0.0096	2	14
Plum	Australia	SC, 48	0.27	0.018	3	14
Plum	Chile	SC, 48		0.0096	2	1
Plum	Croatia	SC, 48	0.096	0.0048	2	14
Plum	Czech Republic	SC, 48	0.12	0.012	2	14
Plum	Germany	SC, 48	0.048 per m canopy height	0.0032	2	14
Plum	Japan	WG, 30	0.53	0.0075	3	7
Plum	Netherlands	SC, 48	0.12	0.012	2	14
Plum	Poland	SC, 48	0.096	0.013 - 0.019	1	14
Plum	Romania	SC, 48	0.096	0.0096	2	14
Plum	Slovakia	SC, 48	0.096			14
Plum	United Kingdom	SC, 48	0.144	0.014	3	14
Pome fruit	Australia	SC, 48	0.27	0.018	3	14
Pome fruit	Austria	SC, 48		0.01 – 0.012	1-2	14
Pome fruit	Germany	SC, 48	0.048 per m canopy height		2	14
Pome fruit	Switzerland	SC, 48	(0.19)	0.0192	2	21
Pome fruit	USA	SC, 40	0.28	0.01	6	30
Potato	Argentina	SC, 48	0.072		min. 1	7
Potato	Austria	SC, 48	0.096	0.019 - 0.048	2	21
Potato	Belize	SC, 10	0.1	(0.02)		21
Potato	Brazil	SC, 48	0.048			21
Potato	Costa Rica	SC, 10	0.1	(0.02)		21
Potato	Croatia	SC, 48	0.048		2	14
Potato	Cyprus	SC, 48		0.0096	1	21
Potato	Czech Republic	SC, 48	0.048	0.0096 - 0.016	2	21
Potato	Dom. Republic	SC, 10	0.1	(0.02)		21
Potato	El Salvador	SC, 10	0.1	(0.02)		21
Potato	Estonia	SC, 48	0.048	0.096 - 0.024	1	21
Potato	Georgia	SC, 48	0.04			
Potato	Greece	SC, 48	0.072	0.005 - 0.014	2	21
Potato	Guatemala	SC, 10	0.1	0(0.02)		21
Potato	Honduras	SC, 10	0.1	(0.02)		21
Potato	Hungary	SC, 48	0.048	0.012		7
Potato	Israel	SC, 48	0.096	0.0048 - 0.0096	1	21
Potato	Japan	WG, 30	(0.23)	0.0075	3	7
Potato	Latvia	SC, 48	0.048		3	21
Potato	Lithuania	SC, 48	0.024 – 0.048	0.006 – 0.024	3	21
Potato	Mexico	SC, 48	0.096			7
Potato	Netherlands	SC, 48	0.12		3	14
Potato	Nicaragua	SC, 10	0.1	(0.02)		21
Potato	Panama	SC, 10	0.1	(0.02)		21
Potato	Poland	SC, 48	0.048	0.012	1	3
Potato	Portugal	SC, 48	0.072	0.009	2	21
Potato	Romania	SC, 48	0.077		2	
Potato	Slovakia	SC, 48	0.048	0.012	2	14
Potato	Slovenia	SC, 48	0.048	(0.006 - 0.0096)	2	21
Potato	South Korea	SC, 10		0.00075	3	21
Potato	Spain	SC, 48	0.096		3	21
Potato	Switzerland	SC, 48	0.048		3	21
Potato (seed)	United Kingdom	OD, 24	0.096	0.024 – 0.048	2	14
Potato (ware)	United Kingdom	OD, 24	0.096	0.024 – 0.048	1	14
Raspberry	Germany	SC, 48	0.096	0.0096	2	14
Raspberry	Netherlands	SC, 48	0.14	0.012	2	3
Raspberry and rubus hybrids	United Kingdom	SC, 48	0.12	0.024	3	3
Red currants	Latvia	SC, 48	0.072	(0.019)	2	14
Red Pepper	South Korea	SC, 10		0.0005	3	3
Rice	Belize	SE, 11	0.1	(0.02)		21
Rice	Costa Rica	SE, 11	0.1	(0.02)		21
Rice	Dom. Republic	SE, 11	0.1	(0.02)		21

Crop	Country	Formulation, ai %	Application rate		No. Per season	PHI (days)
			kg ai/ha	kg ai/hL		
Rice	El Salvador	SE, 11	0.1	(0.02)		21
Rice	Guatemala	SE, 11	0.1	(0.02)		21
Rice	Honduras	SE, 11	0.1	(0.02)		21
Rice	Indonesia	SC, 24	0.14	0.028		
Rice	Nicaragua	SE, 11	0.1	(0.02)		21
Rice	Panama	SE, 11	0.1	(0.02)		21
Rice	South Korea	GR, 1	0.5 g/box		1	
Rice	Thailand	SC, 24		0.018		14
Rice (nursery box)	Japan	GR, 1,5	50 g/box = 0.15 kg ai/ha		1	
Squash	Cyprus (field and greenhouse use)	SC, 48		0.014	3	3
Squash	Netherlands	SC, 48		0.012	4	1
Stone fruit	Argentina	SC, 48		0.0096	2	14
Stone fruit	Australia	SC, 48	0.27	0.018	3	14
Stone fruit	Chile	SC, 48		0.0096	2	1
Stone fruit	Czech Republic	SC, 48	0.096	0.0096	2	14
Stone fruit	Switzerland	SC, 48		0.0058	2	21
Stone fruit	Tunisia	SC, 48		0.0096		30
Strawberry	Japan	WG, 30	0.23	0.0075	3	1
Strawberry	Latvia	SC, 48	0.096	(0.024)	2	14
Strawberry	Netherlands	SC, 48	0.14	0.012	2	3
Strawberry	Netherlands (greenhouse use)	SC, 48	0.14	0.012	2	1
Strawberry	United Kingdom	Sc, 48	0.12	0.024	2	3
Strawberry	United Kingdom (greenhouse use)	Sc, 48	0.12	0.024	2	3
Sunflower	Hungary	SC, 48	0.036	0.008 - 0.012		30
Sunflower	Slovakia	SC, 48	0.048			
Sweet Corn	Israel	SC, 48	0.096	0.013 - 0.064	2	3
Sweet Pepper	Japan	WG, 30	0.23	0.0075	3	1
Sweet Pepper	Tunisia	SC, 48		0.014	2	3
Tayberry	Netherlands	SC, 48	0.14	0.012	2	3
Tea	Japan	WG, 30	0.6	0.015	1	7
Tomato	Argentina	SC, 48	0.144		min. 1	3
Tomato	Belize	SC, 10	0.1	(0.02)		21
Tomato	Brazil	SC, 48	0.096	(0.0096)		7
Tomato	Costa Rica	SC, 10	0.1	(0.02)		21
Tomato	Croatia	SC, 48	0.14	0.014	2	4
Tomato	Cyprus	SC, 48		0.014	3	3
Tomato	Dom. Republic	SC, 10	0.1	(0.02)		21
Tomato	El Salvador	SC, 10	0.1	(0.02)		21
Tomato	Greece	SC, 48	0.216	0.014	2	3
Tomato	Guatemala	SC, 10	0.1	(0.02)		21
Tomato	Honduras	SC, 10	0.1	(0.02)		21
Tomato	Israel	SC, 48	0.192			3
Tomato	Italy	SC, 48	0.14	0.014		3
Tomato	Japan	WG, 30	0.23	0.015	3	1
Tomato	Kenya	SC, 48	0.19	(0.019)	4	3
Tomato	Morocco	SC, 48		0.014		3
Tomato	Netherlands (greenhouse use)	SC, 48	0.12	0.012	4	1
Tomato	Netherlands (greenhouse use, drip irrigation)	SC, 48	0.0096 kg ai per 1000 plants		1	3
Tomato	Nicaragua	SC, 10	0.1	(0.02)		21
Tomato	Panama	SC, 10	0.1	(0.02)		21
Tomato	Peru	SC, 48		0.036		3
Tomato	Slovakia	SC, 48		0.014		3
Tomato	Slovenia	SC, 48	0.21	0.014	2	3
Tomato	Spain	SC, 48		0.014	3	3
Tomato	Tunisia	SC, 48		0.014	2	3

Crop	Country	Formulation, ai %	Application rate		No. Per season	PHI (days)
			kg ai/ha	kg ai/hL		
Tomato	Turkey	SC, 48		0.0096		3
Tomato	United Kingdom	SC, 48	0.22	max. 0.022	3	3
Tree nuts	Italy	SC, 48	0.18	0.012		14
Walnut	Argentina	SC, 48		0.0096	2	21
Walnut	Chile	SC, 48		0.0096	2	1
Walnut	Italy	SC, 48	0.18	0.012		14
Walnut	United Kingdom	SC, 48	0.18	0.018	2	
Watermelon	Belize	SC, 10	0.1	(0.02)		21
Watermelon	Brazil	Sc, 48	0.096			21
Watermelon	Costa Rica	SC, 10	0.1	(0.02)		21
Watermelon	Cyprus	SC, 48		0.014	3	3
Watermelon	Dom. Republic	SC, 10	0.1	(0.02)		21
Watermelon	El Salvador	SC, 10	0.1	(0.02)		21
Watermelon	Guatemala	SC, 10	0.1	(0.02)		21
Watermelon	Honduras	SC, 10	0.1	(0.02)		21
Watermelon	Japan	WG, 30	0.45	0.0075	3	1
Watermelon	Kenya	SC, 48	0.19	0.019	3	3
Watermelon	Nicaragua	SC, 10	0.1	(0.02)		21
Watermelon	Panama	SC, 10	0.1	(0.02)		21
Watermelon	South Korea	SC, 10		0.0005	3	3
Watermelon	Spain	SC, 48	0.14	0.0067	3	3
Wheat	Romania	SC, 48	0.048	0.012 - 0.016	1	
White mustard	Czech Republic	SC, 48	0.096	0.016 - 0.048	2	
Winter wheat	Lithuania	SC, 48	0.034		3	21
Wintersquash	Israel	SC, 48	0.19	0.026 - 0.128	2	10

Values in parenthesis are calculated from the spray volume

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on thiacloprid supervised trials on the following crops (Table 26).

Trials were well documented with laboratory and field reports. The former included method validation including procedural recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analysis or duration of sample storage were also provided. Although trials included control plots, no control data are recorded in the Tables because no residues in control samples exceeded the LOQ. Residues are unadjusted for recoveries.

When residues were not detected they are shown as below the LOQ (e.g. < 0.01 mg/kg). Residues, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residues from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. These results are double underlined.

Periods of freezer storage between sampling and analysis were recorded for all trials and were covered by the periods of the freezer storage stability studies.

Table 26. Overview of supervised residue trials.

Commodity	Application	Country	Table no.
Citrus	Foliar	Brazil, New Zealand, South Africa	Table 27
Apple	Foliar	Australia, Belgium, France, Germany, Italy, Japan, Netherlands, South Africa, Spain, United Kingdom, USA	Table 28
Pear	Foliar	Australia, Japan, South Africa, USA	Table 29
Apricot, Japanese	Foliar	Japan	Table 30
Peach	Foliar	France, Italy, Japan, Spain	Table 31
Cherry	Foliar	Belgium, France, Germany, Italy, Japan, Spain, USA	Table 32
Plum	Foliar	France, Germany, Japan, Spain, USA	Table 33

Grapes	Foliar (glasshouse use)	Japan	Table 34
Strawberries	Foliar (field use)	Belgium, France, Germany, United Kingdom	Table 35
Strawberries	Foliar (greenhouse use)	France, Germany, Italy, Japan, Netherlands, Spain	Table 36
Currants	Foliar	Belgium, Germany, United Kingdom	Table 37
Raspberries	Foliar	Germany, United Kingdom	Table 38
Kiwi fruits	Foliar	New Zealand	Table 39
Onions	Foliar	Brazil. Germany	Table 40
Garlic	Foliar	Brazil	Table 41
Eggplants	Foliar (glasshouse use)	Japan	Table 42
Cucumbers	Foliar (field use)	Germany, Italy, Spain	Table 43
Cucumbers	Foliar (greenhouse use)	Belgium, France, Germany, Greece, Italy, Netherlands, Spain	Table 44
Melons	Foliar (field use)	France, Greece, Italy	Table 45
Melons	Foliar (glasshouse use)	Japan	Table 46
Watermelons	Foliar (field use)	Greece, Spain	Table 47
Watermelons	Foliar (glasshouse use)	Japan	Table 48
Tomato	Foliar (field use)	France, Italy	Table 49
Tomato	Foliar (greenhouse use)	France, Germany, Japan, Spain	Table 50
Tomato	Drip application (greenhouse use)	Belgium, Netherlands	Table 51
Pepper	Foliar (field use)	France, Italy, Spain	Table 52
Pepper	Foliar (greenhouse use)	France, Netherlands, Japan, Spain	Table 53
Pepper	Drip application (greenhouse use)	Belgium, Netherlands	Table 54
Potato	Foliar	Belgium, Brazil, France, Germany, Italy, Japan, Spain, United Kingdom	Table 55
Wheat	Foliar	France, Germany	Table 56
Barley	Foliar	France, Germany	Table 57
Rice	Foliar	India	Table 58
Rice	Granular	Japan	Table 59
Maize	Foliar	France, Germany, Greece, Italy, Spain	Table 60
Walnuts	Foliar	Italy	Table 61
Almonds	Foliar	USA	Table 62
Pecan	Foliar	USA	Table 63
Oilseed rape	Foliar	France, Germany, Hungary, Spain, Sweden	Table 64
Cotton	Foliar	Greece, Spain, USA	Table 65[1]
Sunflowers	Foliar	Hungary	Table 66
Green tea	Foliar	Japan	Table 67

[1] Single underlined values were used for the evaluation of rape forage. Double underlined values were used for the evaluation of rape seed and white mustard.

Citrus fruits

Table 27. Thiacloprid residues resulting from foliar application to citrus.

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Brazil, Riberao Preto/SP (Pera Rio)	1998	480 SC	0.096	0.0048	3	Lemon, peel Lemon, pulp	21 21	0.02 < 0.02	Lancas, F. M. 1998, 1998a M-005338-01-2 and M-005340-01-2
Brazil, Riberao Preto/SP (Pera Rio)	1998	480 SC	0.19	0.0096	3	Lemon, peel Lemon, pulp	21 21	0.06 0.04	Lancas, F. M. 1998, 1998a M-005338-01-2 and M-005340-01-2

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
New Zealand Kerikeri 2002 (Yen Ben)	480 SC	0.19	0.0096	1	Lemon, whole fruit	1 3 5 7 10 14	0.19 0.2 0.1 0.14 0.15 0.07	Clay, S. 2002 M-261169-01-1
South Africa Vaalwater, Northern Province 1999 (Navel)	480 SC	0.00043	0.014	1	Orange, whole fruit Orange, peel Orange, pulp	44 93 123 151 190 93 123 151 190	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Zyl, P. F. C. van 2000a M-048686-02-1
South Africa Vaalwater, Northern Province 1999 (Navel)	480 SC	0.00086	0.029	1	Orange, whole fruit Orange, peel Orange, pulp	44 93 123 151 190 93 123 151 190	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Zyl, P. F. C. van 2000a M-048686-02-1

Pome fruits

Table 28. Thiacloprid residues resulting from foliar application to apples.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
United Kingdom Thurston 1995 (Gloster 69)	480 SC	0.14 – 0.15	0.0096	2	Fruit	0[1] 0 7 10 14 22	< 0.02 0.03 0.05 0.03 <u>0.04</u> 0.04	Placke, F. J. 1997a RA-2062/95
Belgium Kortenaken 1995 (Jonagold)	480 SC	0.14 – 0.15	0.0096	2	Fruit	0[1] 0 7 14 21	0.04 0.18 0.17 <u>0.12</u> 0.09	Placke, F. J. 1997a RA-2062/95
Germany Burscheid 1995 (Jonagold)	480 SC	0.14	0.0096	2	Fruit	0[1] 0 7 10 14 21	0.04 0.16 0.09 0.06 <u>0.07</u> 0.04	Placke, F. J. 1997a RA-2062/95
Germany Monheim 1995 (Golden Delicious)	480 SC	0.14	0.029	2	Fruit	0[1] 0 7 10 14 21	0.04 0.25 < 0.02 0.09 0.07 0.05	Placke, F. J. 1997a RA-2062/95
United Kingdom Bury St. Edmunds 1996 (Golden Delicious)	480 SC	0.18	0.012	2	Fruit	0 14	0.26, 0.28 (0.27) 0.15, 0.16 (<u>0.16</u>)	Placke, F. J. 1997b RA-2114/96

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Netherlands Kruisland 1996 (Jonagold)	480 SC	0.18	0.012	2	Fruit	0 14	0.11 0.05	Placke, F. J. 1997b RA-2114/96
Germany Burscheid 1996 (Elstar)	480 SC	0.18	0.012	2	Fruit	0 13	0.23 0.11	Placke, F. J. 1997b RA-2114/96
Germany Monheim 1996 (Golden Delicious)	480 SC	0.14	0.036	2	Fruit	0 14	0.23 0.11	Placke, F. J. 1997b RA-2114/96
France Bossay 2000 (Reine des Reinettes)	480 SC	0.18	0.012	2	Fruit	0 14	0.19 0.21	Schoening, R. 2001a M-080245-01-1
France Azay le Rideau 2000 (Golden)	480 SC	0.18	0.012	2	Fruit	0 14	0.11 0.1	Schoening, R. 2001a M-080245-01-1
Spain Pere Pescador 1995 (Starking)	480 SC	0.14	0.0096	2	Fruit	0[1] 0 7 10 14 21	0.05 0.15 0.11 0.09 0.08 0.07	Placke, F. J. 1997c M-000919-01-1
Italy Laives 1995 (Golden Delicious)	480 SC	0.14	0.0096	2	Fruit	0[1] 0 7 10 14 21	0.03 0.15 0.03 0.03 0.02 0.02	Placke, F. J. 1997c M-000919-01-1
Italy Laives 1995 (Granny Smith)	480 SC	0.14	0.0096	2	Fruit	0[1] 0 7 10 14 21	0.02 0.12 0.13 0.15 0.1 0.08	Placke, F. J. 1997c M-000919-01-1
Spain Pere Pescador 1995 (Golden)	480 SC	0.14	0.0096	2	Fruit	0[1] 0 7 10 14 21	0.05 0.15 0.13 0.13 0.11 0.1	Placke, F. J. 1997c M-000919-01-1
Spain Pere Pescador 1995 (Suprema)	480 SC	0.18	0.012	2	Fruit	0 14	0.19 0.14	Placke, F. J. 1997d M-000913-01-1
Italy Pineta di Laives 1996 (Stark Delicious)	480 SC	0.18	0.012	2	Fruit	0 14	0.14 0.1	Placke, F. J. 1997d M-000913-01-1
Italy Montemarzino 1996 (Golden)	480 SC	0.18	0.012	2	Fruit	0 14	0.48 0.36	Placke, F. J. 1997d M-000913-01-1
France Roches-Prémarie-Andillé 1996 (Golden)	480 SC	0.18	0.012	2	Fruit	0 14	0.22 0.04	Placke, F. J. 1997d M-000913-01-1

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
France Mas Grenier 2000 (Golden)	480 SC	0.18	0.012	2	Fruit	0 14	0.3 0.13	Schoening, R. 2001b M-075119-01-1
USA Pennsylvania 1998 (Starkrimson Red Delicious)	480 SC	0.28	0.046	2	Fruit	29 46	0.06 0.07	Harbin, A.M. 1999 M-009903-01-1
USA Pennsylvania 1998 (Starkrimson Red Delicious)	480 SC	0.28	0.0098	2	Fruit	29 46	0.11 0.08	Harbin, A.M. 1999 M-009903-01-1
USA New York 1998 (Red Delicious)	480 SC	0.28	0.037	2	Fruit	30 44	0.11 0.05	Harbin, A.M. 1999 M-009903-01-1
USA New York 1998 (Red Delicious)	480 SC	0.28	0.0075	2	Fruit	30 44	0.096 0.05	Harbin, A.M. 1999 M-009903-01-1
USA Pennsylvania 1998 (Law Rome/MM111)	480 SC	0.28	0.048	2	Fruit	30 45	0.03 0.02	Harbin, A.M. 1999 M-009903-01-1
USA Pennsylvania 1998 (Law Rome/MM111)	480 SC	0.28	0.0085	2	Fruit	30 45	0.04 0.03	Harbin, A.M. 1999 M-009903-01-1
USA North Carolina 1998 (Red Delicious)	480 SC	0.28	0.06	2	Fruit	31 45	0.02 0.02	Harbin, A.M. 1999 M-009903-01-1
USA North Carolina 1998 (Red Delicious)	480 SC	0.28	0.0081	2	Fruit	31 45	0.06 0.04	Harbin, A.M. 1999 M-009903-01-1
USA Illinois 1998 (Jonathon)	480 SC	0.28	0.043	2	Fruit	30 45	0.06 0.06	Harbin, A.M. 1999 M-009903-01-1
USA Illinois 1998 (Jonathon)	480 SC	0.28	0.0078	2	Fruit	30 45	0.07 0.03	Harbin, A.M. 1999 M-009903-01-1
USA Utah 1998 (Red Delicious)	480 SC	0.28	0.047	2	Fruit	29 47	0.04 0.01	Harbin, A.M. 1999 M-009903-01-1
USA Utah 1998 (Red Delicious)	480 SC	0.28	0.0074	2	Fruit	29 47	0.02 0.01	Harbin, A.M. 1999 M-009903-01-1
USA Idaho 1998 (Law Spur Rome)	480 SC	0.28	0.034	2	Fruit	30 45	0.03 0.02	Harbin, A.M. 1999 M-009903-01-1

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
USA Idaho 1998 (Law Spur Rome)	480 SC	0.28	0.0078	2	Fruit	30 45	0.06 0.04	Harbin, A.M. 1999 M-009903-01-1
USA Oregon 1998 (Jonagold)	480 SC	0.28	0.033	2	Fruit	30 46	0.07 0.02	Harbin, A.M. 1999 M-009903-01-1
USA Oregon 1998 (Jonagold)	480 SC	0.28	0.0073	2	Fruit	30 46	0.28 0.04	Harbin, A.M. 1999 M-009903-01-1
USA Washington 1998 (Red Delicious)	480 SC	0.28	0.041	2	Fruit	30 45	0.13 0.04	Harbin, A.M. 1999 M-009903-01-1
USA Washington 1998 (Red Delicious)	480 SC	0.28	0.0077	2	Fruit	30 45	0.14 0.05	Harbin, A.M. 1999 M-009903-01-1
USA Oregon 1998 (Fuji Apple)	480 SC	0.28	0.04	2	Fruit	23 30 37 44 52	0.05 0.03 0.03 0.02 0.03	Harbin, A.M. 1999 M-009903-01-1
USA Oregon 1998 (Fuji Apple)	480 SC	0.28	0.007	2	Fruit	23 30 37 44 52	0.09 0.09 0.03 0.03 0.07	Harbin, A.M. 1999 M-009903-01-1
USA California 1998 (Rome Beauty)	480 SC	0.28	0.038	2	Fruit	25 32 39 46 53	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	Harbin, A.M. 1999 M-009903-01-1
USA California 1998 (Rome Beauty)	480 SC	0.28	0.007	2	Fruit	25 32 39 46 53	0.02 0.02 0.06 0.02 0.05	Harbin, A.M. 1999 M-009903-01-1
USA Kansas 1998 (Golden Delicious)	480 SC	0.28	0.048	2	Fruit	41	< 0.01	Harbin, A.M. 1999 M-009903-01-1
USA Kansas 1998 (Golden Delicious)	480 SC	0.28	0.0082	2	Fruit	41	0.05	Harbin, A.M. 1999 M-009903-01-1
USA Oregon 1998 (Fuji Apples)	70 WG	0.28	0.04	2	Fruit	23 30 37 44 52	0.08 0.04 0.02 0.05 0.03	Harbin, A.M. 1999 M-009903-01-1
USA Oregon 1998 (Fuji Apples)	70 WG	0.28	0.007	2	Fruit	23 30 37 44 52	0.097 0.07 0.02 0.03 0.03	Harbin, A.M. 1999 M-009903-01-1

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
USA California 1998 (Rome Beauty)	70 WG	0.08	0.038	2	Fruit	25 32 39 46 53	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	Harbin, A.M. 1999 M-009903-01-1
USA California 1998 (Rome Beauty)	70 WG	0.08	0.007	2	Fruit	25 32 39 46 53	0.02 0.03 0.02 0.01 0.05	Harbin, A.M. 1999 M-009903-01-1
USA Pennsylvania 1998 (Starkrimson Red Delicious)	70 WG	0.28	0.046	2	Fruit	29 46	0.07 0.08	Harbin, A.M. 1999 M-009903-01-1
USA Pennsylvania 1998 (Starkrimson Red Delicious)	70 WG	0.28	0.0098	2	Fruit	29 46	0.09 0.09	Harbin, A.M. 1999 M-009903-01-1
South Africa Villiersdorp 2000 (Golden Delicious)	480 SC	0.24	0.0096	4	Fruit	0[1] 0 14	0.32, 0.31 (0.32) 0.68, 0.67 (0.68) 0.54, 0.54 (0.54)	Zyl, P. F. C. van 2000b 5438/1493281/T392
South Africa Villiersdorp 2000 (Granny Smith)	480 SC	0.24	0.0096	4	Fruit	0[1] 0 14	0.23, 0.26 (0.25) 0.73, 0.76 (0.75) 0.43, 0.43 (0.43)	Zyl, P. F. C. van 2000c 5438/1493257/T389
South Africa Villiersdorp 2000 (Royal Galaxy)	480 SC	0.19	0.0096	4	Fruit	0[1] 0 14	0.06, 0.06 (0.06) 0.48, 0.45 (0.47) 0.15, 0.14 (0.15)	Garbers, H. V. 2000 5438/1493265/T390
South Africa Villiersdorp 2000 (Royal Galaxy)	480 SC	0.38	0.019	4	Fruit	0[1] 0 14	0.12, 0.13 (0.13) 0.51, 0.71 (0.61) 0.60, 0.51 (0.56)	Garbers, H. V. 2000 5438/1493265/T390
Australia Applethorpe 1998 (Granny Smith)	480 SC	0.24	0.018	10	Fruit Replicate trial Fruit	0 7 14 21 0 7 14 21	0.28, 0.55 (0.42) 0.21, 0.21 (0.21) 0.50, 0.23 (0.37) 0.30, 0.31 (0.31) 0.70, 0.44 (0.57) 0.44, 0.38 (0.41) 0.25, 0.31 (0.28) 0.25, 0.32 (0.29)	Tancred, S. 1999 EMH453/99

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Japan, Iwate	1996 (Fuji)	WG, 30	0.6	0.015	3	Thiacloprid: Whole fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 1997, 1997a, 1997b, 1997c
							7	0.30, 0.28, 0.30[2], 0.30[2] (<u>0.30</u>)	
							15	0.33, 0.29, 0.27[2], 0.26[2] (0.29)	
							22	0.26, 0.25, 0.28[2], 0.27[2] (0.27)	
						Thiacloprid/amide: Whole Fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							7	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							15	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							22	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
Japan, Nagano	1996 (Tugaru)	WG, 30	0.6	0.015	3	Thiacloprid: Whole fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 1997, 1997a, 1997b, 1997c
							7	0.09, 0.09, 0.13[2], 0.13[2] (<u>0.11</u>)	
							15	0.05, 0.04, 0.05[2], 0.05[2] (0.05)	
							22	0.06, 0.06, 0.06[2], 0.06[2] (0.06)	
						Thiacloprid-amide: Whole Fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							7	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							15	< 0.005,	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
						22	< 0.005, < 0.005[2], < 0.005[2] (< 0.005) < 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	

1) sampling before last application

2) replicate analysis

Table 29. Thiacloprid residues resulting from foliar application to pears.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
USA Pennsylvania 1998 (Bartlett)	480 SC	0.28	0.039	2	Fruit	31 45	0.06 0.08	Harbin, A.M. 1999 M-009903-01-1
USA Pennsylvania 1998 (Bartlett)	480 SC	0.28	0.0097	2	Fruit	31 45	0.13 0.14	Harbin, A.M. 1999 M-009903-01-1
USA California 1998 (Bartlett)	480 SC	0.28	0.031	2	Fruit	29 44	0.25 0.21	Harbin, A.M. 1999 M-009903-01-1
USA California 1998 (Bartlett)	480 SC	0.28	0.0099	2	Fruit	29 44	0.23 0.13	Harbin, A.M. 1999 M-009903-01-1
USA California 1998 (Bosc)	480 SC	0.28	0.032	2	Fruit	30 44	0.21 0.26	Harbin, A.M. 1999 M-009903-01-1
USA California 1998 (Bosc)	480 SC	0.28	0.0094	2	Fruit	30 44	0.27 0.12	Harbin, A.M. 1999 M-009903-01-1
USA Idaho 1998 (Max Red Bartlett)	480 SC	0.28	0.033	2	Fruit	22 29 36 43 50	0.06 0.05 0.05 0.03 0.03	Harbin, A.M. 1999 M-009903-01-1
USA Idaho 1998 (Max Red Bartlett)	480 SC	0.28	0.01	2	Fruit	22 29 36 43 50	0.08 0.06 0.06 0.04 0.05	Harbin, A.M. 1999 M-009903-01-1
USA Oregon 1998 (Red Clapp)	480 SC	0.28	0.033	2	Fruit	30 45	0.04 0.03	Harbin, A.M. 1999 M-009903-01-1
USA Oregon 1998 (Red Clapp)	480 SC	0.28	0.01	2	Fruit	30 45	0.05 0.04	Harbin, A.M. 1999 M-009903-01-1

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
USA Washington 1998 (Red Bartlett)	480 SC	0.28	0.033	2	Fruit	30 45	0.18 0.14	Harbin, A.M. 1999 M-009903-01-1
USA Washington 1998 (Red Bartlett)	480 SC	0.28	0.01	2	Fruit	30 45	0.24 0.15	Harbin, A.M. 1999 M-009903-01-1
USA Sacramento 1998 (Bartlett)	70 WG	0.28	0.031	2	Fruit	29 44	0.22 0.17	Harbin, A.M. 1999 M-009903-01-1
USA Sacramento 1998 (Bartlett)	70 WG	0.28	0.0099	2	Fruit	29 44	0.14 0.13	Harbin, A.M. 1999 M-009903-01-1
USA Idaho 1998 (Max Red Bartlett)	70 WG	0.28	0.033	2	Fruit	22 29 36 43 50	0.06 0.05 0.08 0.02 0.02	Harbin, A.M. 1999 M-009903-01-1
USA Idaho 1998 (Max Red Bartlett)	70 WG	0.28	0.01	2	Fruit	22 29 36 43 50	0.11 0.08 0.08 0.10 0.06	Harbin, A.M. 1999 M-009903-01-1
South Africa Groot Drakenstein 2000 (Forelle)	480 SC	0.24	0.0096	4	Fruit	0[1] 0 14	0.19, 0.21 (0.20) 0.55, 0.48 (0.52) 0.22, 0.21 (0.22)	Zyl, P. F. C. van 2000d 5438/1502352/T465
South Africa Groot Drakenstein 2000 (Forelle)	480 SC	0.31	0.0096	4	Fruit	0[1] 0 14	0.51, 0.49 (0.50) 0.98, 1.1 (1.04) 0.51, 0.48 (0.50)	Zyl, P. F. C. van 2000e 5438/1502361/T466
South Africa Groot Drakenstein 2000 (Forelle)	480 SC	0.61	0.0192	4	Fruit	0[1] 0 14	0.85, 0.82 (0.84) 1.4, 1.5 (1.5) 1.1, 1.1 (1.1)	Zyl, P. F. C. van 2000e 5438/1502361/T466
Australia Cottenvale 1999 (Packham)	480 SC	0.35	0.018	8	Fruit	0[1] 0 7 14 21 28	0.43 0.66 0.30 0.37 0.34 0.29	Tancred, S. 2001 ADM 172/01
Australia Cottenvale 1999 (Packham)	480 SC	0.35	0.018	4	Fruit	0[1] 0 7 14 21 28	0.41 0.62 0.33 0.26 0.28 0.38	Tancred, S. 2001 ADM 172/01
Japan, Fukushima 1997 (Kousui)	30 WG	0.6	0.015	3	Thiacloprid: Whole fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 1997l, 1997m, 1997n, 1997o

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
							7	0.67, 0.64, 0.57[2], 0.57[2] (0.61)	
							14	0.45, 0.45, 0.34[2], 0.33 (0.39)	
							21	0.31, 0.31, 0.19[2], 0.18[2] (0.25)	
						Thiacloprid/amide: Whole Fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							7	0.02, 0.01, 0.01[2], 0.01[2] (0.01)	
							14	0.01, 0.01, 0.01[2], 0.01[2] (0.01)	
							21	0.01, 0.01, 0.01[2], 0.01[2] (0.01)	

1 sampling before last application

2 replicate analysis

Stone fruits

Table 30. Thiacloprid residues resulting from foliar application to Japanese apricots.

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Japan, Fukui	1997 (Benisashi)	WG, 30	0.6	0.015	2	Thiacloprid: Whole fruit w/o stone	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 2001a, 2001b
							7	1.7, 1.6, 1.3[2], 1.3[2] (1.5)	
							14	0.91, 0.81, 0.81[2], 0.77[2] (0.83)	
							21	0.94, 0.83, 0.69[2], 0.66[2] (0.78)	
						Thiacloprid/amide: Whole Fruit w/o stone	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							7	0.09, 0.08,	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
							0.06[2], 0.06[2] (0.07)	
						14	0.05, 0.05, 0.03[2], 0.02[2] (0.04)	
						21	0.04, 0.04, 0.02[2], 0.02[2] (0.03)	
Japan, Wakayama 1997 (Nanko)	WG, 30	0.6	0.015	2	Thiacloprid: Whole fruit w/o stone	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 2001a, 2001b
						7	1.3, 1.3, 0.96[2], 0.94[2] (1.1)	
						14	1.4, 1.4, 0.80[2], 0.79[2] (1.1)	
						21	0.44, 0.43, 0.28[2], 0.27[2] (0.36)	
					Thiacloprid/amide: Whole Fruit w/o stone	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
						7	0.08, 0.07, 0.05[2], 0.05[2] (0.06)	
						14	0.07, 0.05, 0.04[2], 0.04[2] (0.05)	
						21	0.03, 0.03, 0.02[2], 0.02[2] (0.03)	

1 sampling before last application

2 replicate analysis

Table 31. Thiacloprid residues resulting from foliar application to peaches.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
France Eyragues 2000 (Meryl Gen Free)	480 SC	0.11	0.0096	2	Fruit, whole	0 14	0.13 0.03	Schoening, R. 2001c RA-2113/00
Spain La Fortesa 1995 (Fire Red)	480 SC	0.14	0.0096	2	Fruit, whole	0[1] 0 8 10 14 21	0.13 0.23 0.24 0.17 0.13 0.07	Placke, F. J. 1997e RA-2064/95

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Spain Gualta 1995 (Baby Gold 5)	480 SC	0.14	0.0096	2	Fruit, whole	0[1] 0 7 10 14 21	0.05 0.15 0.09 0.10 0.08 0.05	Placke, F. J. 1997e RA-2064/95
Italy Ravenna 1995 (Red Heaven)	480 SC	0.14	0.0096	2	Fruit, whole	0[1] 0 7 10 14 21	0.02 0.08 0.06 0.04 0.03 < 0.02	Placke, F. J. 1997e RA-2064/95
Italy Fondi 1995 (O'Henry)	480 SC	0.14	0.0096	2	Fruit, whole	0[1] 0 7 10 14 21	0.06 0.24 0.17 0.11 0.06 0.05	Placke, F. J. 1997e RA-2064/95
Spain La Fortesa 1996 (Baby Gold 9)	480 SC	0.14	0.0096	2	Fruit, whole	0 14	0.19 0.09	Placke, F. J. 1997f RA-2121/96
Spain Can Rosell-Subiratt 1996 (Springcrest)	480 SC	0.14	0.0096	2	Fruit, whole	0 14	0.55 0.19	Placke, F. J. 1997f RA-2121/96
Italy Ravenna 1996 (Red Heaven)	480 SC	0.14	0.0096	2	Fruit, whole	0 14	0.10 0.03	Placke, F. J. 1997f RA-2121/96
France Eyragues 1996 (Meryl Gen Free)	480 SC	0.14	0.0096	2	Fruit, whole	0 14	0.18 0.13	Placke, F. J. 1997f RA-2121/96
Japan, Fukushima 1997 (Kawanakajimahakutou)	30 WG	0.6	0.015	3	Thiacloprid: Whole fruit w/o stone Thiacloprid-amide: Whole Fruit w/o stone	0[1] 7 14 21 0[1] 7 14	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005) 0.48, 0.48, 0.24[2], 0.24[2] (0.36) 0.42, 0.40, 0.33[2], 0.33[2] (0.37) 0.43, 0.42, 0.38[2], 0.36[2] (0.40) < 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005) 0.01, 0.01, < 0.005[2], < 0.005[2] (0.01) 0.01, 0.01, < 0.005[2], < 0.005[2]	Anon. 1997h, 1997i, 1997j, 1997k

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
							(0.01)	
						21	0.01, 0.01, < 0.005[2], < 0.005[2] (0.01)	
Japan, Wakayama 1997 (Takeihakuhou)	30 WG	0.6	0.015	3	Thiacloprid: Whole fruit w/o stone	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 1997h, 1997i, 1997j, 1997k
						7	0.31, 0.30, 0.23[2], 0.22[2] (0.27)	
						14	0.1, 0.1, 0.14[2], 0.14[2] (0.12)	
						21	0.07, 0.06, 0.09[2], 0.09[2] (0.08)	
					Thiacloprid-amide: Whole Fruit w/o stone	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
						7	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
						14	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
						21	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	

1 sampling before last application

2 replicate analysis

Table 32. Thiacloprid residues resulting from foliar application to cherries.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Belgium Engelmanshoven 1998 (Cherry, sour Kollaris)	480 SC	0.18	0.012	2	Fruit, whole	0[1]	0.03	Schoening, R. 1999a RA-2070/98
						0	0.33	
						7	0.10	
						14	0.04	
						21	< 0.02	
Germany Wachtberg – Niederbachem 1998 (Cherry, sour Schattenmorelle)	480 SC	0.18	0.012	2	Fruit, whole	0[1]	< 0.02	Schoening, R. 1999a RA-2070/98
						0	0.35	
						7	0.06	
						14	0.02	
						21	0.02	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Germany Toenisvorst-Tackheide 1998 (Cherry, sweet Castor)	480 SC	0.18	0.012	2	Fruit, whole	0[1] 0 7 14	0.08 0.23 0.17 0.15	Schoening, R. 1999a RA-2070/98
France Jussy 1998 (Cherry, sweet Gemersdorf)	480 SC	0.15	0.012	2	Fruit, whole	0[1] 0 7 14 21	0.02 0.22 0.13 0.10 0.05	Schoening, R. 1999a RA-2070/98
Germany Wachtberg – Niederbachem 1999 (Cherry, sour Schattenmorelle)	480 SC	0.12	0.012	2	Fruit, whole	0 14	0.24 < 0.02	Sur, R. and Schoening, R. 2000 RA-2070/99
Germany Burscheid 1999 (Cherry, sour Schattenmorelle)	480 SC	0.12	0.012	2	Fruit, whole	0 14	0.47 0.03	Sur, R. and Schoening, R. 2000 RA-2070/99
Germany Muehlheim-Kehrlich 2002 (Cherry, sweet Regina)	480 SC	0.18	0.012	2	Fruit, whole	0 14	0.30 0.11	Schoening, R. and Billian, P. 2003 RA-2030/02
France Pouligny St. Pierre 2002 (Cherry, sweet Garnet)	480 SC	0.12	0.012	2	Fruit, whole	0 14	0.25 0.06	Schoening, R. and Billian, P. 2003 RA-2030/02
Italy Bisceglie 1998 (Cherry, sweet New Star)	480 SC	0.14	0.012	2	Fruit, whole	0[1] 0 7 14 21	0.02 0.12 0.12 0.06 0.04	Schoening, R. 1999b RA-2071/98
Italy Bisceglie 1998 (Cherry, sweet Lapins)	480 SC	0.18	0.012	2	Fruit, whole	0[1] 0 7 14 21	< 0.02 0.35 0.05 0.02 < 0.02	Schoening, R. 1999b RA-2071/98
Italy Bisceglie 1999 (Cherry, sweet Lapins)	480 SC	0.15	0.012	2	Fruit, whole	0 14	0.17 0.08	Heinemann, O. and Schoening, R. 2000 RA-2071/99
Spain Begues 1999 (Cherry, sweet Starking)	480 SC	0.16	0.012	2	Fruit, whole	0 14	0.23 0.07	Heinemann, O. and Schoening, R. 2000 RA-2071/99
USA Porterville, California 2001 (Cherry, sweet Brooks)	480 SC	0.28	0.0083	2	Fruit, w/o stone	13 25	0.17, 0.18 (0.18) 0.13, 0.11 (0.12)	Dorschner, K. W. 2002 200540

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
USA Visalia, California 2001 (Cherry, sweet Brooks)	480 SC	0.28	0.044	2	Fruit, w/o stone	14 28	< 0.02, < 0.02 (< 0.02) < 0.02 < 0.02 (< 0.02)	Dorschner, K. W. 2002 200540
USA Idaho 2001 (Cherry, sweet Brooks)	480 SC	0.28	0.012	2	Fruit, w/o stone	14 26	0.22, 0.25 (0.24) 0.15, 0.13 (0.14)	Dorschner, K. W. 2002 200540
USA Michigan 2001 (Cherry, sweet Emperor Francis)	480 SC	0.29	0.0498	2	Fruit, w/o stone	14 28	0.22, 0.26 (0.24) 0.13, 0.11 (0.12)	Dorschner, K. W. 2002 200540
USA Michigan 2001 (Cherry, sweet Cavalier)	480 SC	0.29	0.013	2	Fruit, w/o stone	13 27	0.12, 0.13 (0.13) 0.05, 0.05 (0.05)	Dorschner, K. W. 2002 200540
USA Oregon 2001 (Cherry, sweet Bing)	480 SC	0.28	0.011	2	Fruit, w/o stone	14 26	0.18, 0.19 (0.19) 0.10, 0.09 (0.10)	Dorschner, K. W. 2002 200540
USA Washington 2001 (Cherry, sweet Bing)	480 SC	0.295	0.047	2	Fruit, w/o stone	13 26	0.16, 0.17 (0.17) 0.05, 0.05 (0.05)	Dorschner, K. W. 2002 200540
Japan, Alita 2003 (Satonishiki)	WG, 30	0.75	0.015	1	Thiacloprid: Whole fruit w/o stone Thiacloprid-amide: Whole Fruit w/o stone	 1 3 7 14 1 3 7 14	 1.4, 1.4 (<u>1.4</u>) 0.8, 0.7 (0.8) 1.1, 1.0 (1.1) 0.6, 0.5 (0.6) < 0.2, < 0.2 (< 0.2) < 0.2, < 0.2 (< 0.2) < 0.2, < 0.2 (< 0.2) < 0.2, < 0.2 (< 0.2)	Anon. 2003

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Japan, Fukushima 2003 (Satonishiki)	WG, 30	0.75	0.015	1	Thiacloprid: Whole fruit w/o stone	1 3 7 14	2.4, 2.3 (2.4) 2.3, 2.1 (2.2) 2.4, 2.3 (2.4) 1.7, 1.7 (1.7)	Anon. 2003
					Thiacloprid-amide: Whole Fruit w/o stone	1 3 7 14	< 0.2, < 0.2 (< 0.2) < 0.2, < 0.2 (< 0.2) < 0.2, < 0.2 (< 0.2) < 0.2, < 0.2 (< 0.2)	

1 sampling before last application

Table 33. Thiacloprid residues resulting from foliar application to plums.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
France Billy 2002 (Mirabelle de Nancy)	480 SC	0.1	0.0097	2	Fruit, whole	0 14	0.08 0.02	Schoening, R. and Billian, P. 2003a RA-2080/02
France Thillot 2003 (Mirabelle de Nancy)	480 SC	0.096	0.0096	2	Fruit, whole	0[1] 0 4 10 14 18	0.03. 0.07 0.03 0.02 0.03 0.03	Schoening, R. and Billian, P. 2003a RA-2080/02
Germany Freinsheim 2000 (Auerbacher)	480 SC	0.11	0.0096	2	Fruit, whole	0[1] 0 4 10 14 18	< 0.02 0.04 0.03 < 0.02 < 0.02 < 0.02	Schoening, R. 2001d RA-2126/00
France Saint Maurice 2000 (Dark Red Plum)	480 SC	0.16	0.015	2	Fruit, whole	0[1] 0 4 10 13 17	0.02 0.08 0.03 0.02 0.02 < 0.02	Schoening, R. 2001d RA-2126/00
Germany Freinsheim 2000 (Cydimer)	480 SC	0.11	0.0096	2	Fruit, whole	0 14	0.02 < 0.02	Schoening, R. 2001d RA-2126/00
France Vieville 2000 (Dark Red Plum)	480 SC	0.14	0.014	2	Fruit, whole	0 13	0.11 0.05	Schoening, R. 2001d RA-2126/00
Germany Freinsheim 2001 (Cydimer)	480 SC	0.12	0.0096	2	Fruit, whole	0 14	0.03 < 0.02	Schoening, R. 2002a RA-2120/01

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Germany Freinsheim 2001 (Auerbacher)	480 SC	0.12	0.0096	2	Fruit, whole	0[1] 0 4 10 14 18	< 0.02 0.05 0.04 0.02 0.03 < 0.02	Schoening, R. 2002a RA-2120/01
France Moissac 2001 (Prune d'Ente	480 SC	0.14	0.0096	2	Fruit, whole	0 15	0.03 0.02	Schoening, R. 2002b RA-212101
France Montauban 2001 (President)	480 SC	0.096	0.0096	2	Fruit, whole	0[1] 0 4 10 14 18	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Schoening, R. 2002b RA-2121/01
Spain St. Feliu de Llobregat 2000 (Black Goal)	480 SC	0.14	0.0096	2	Fruit, whole	0 4 11 14 19	0.06 0.04 0.03 0.02 < 0.02	Schoening, R. 2002c RA-2127/00
Spain St. Vincent dels Horts 2000 (Golden Japan)	480 SC	0.14	0.0096	2	Fruit, whole	0 14	0.04 0.02	Schoening, R. 2002c RA-2127/00
France Montauban 2000 (President)	480 SC	0.096	0.0096	2	Fruit, whole	0[1] 0 4 10 14 18	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Schoening, R. 2002c RA-2127/00
France Orgueil 2000 (President)	480 SC	0.096	0.0096	2	Fruit, whole	0 14	0.02 < 0.02	Schoening, R. 2002c RA-2127/00
USA California 2001 (Casselman)	480 SC	0.29	0.046	2	Fruit, w/o stone Fruit, dried w/o stone	15 29 15 29	0.03, < 0.02 (0.03) < 0.02, < 0.02 (< 0.02) 0.05, 0.05 (0.05) 0.05, 0.08 (0.07)	Dorschner, K. W. 2002a 200509
USA California 2001 (Black Beaut)	480 SC	0.29	0.012	2	Fruit, w/o stone	14 28	< 0.02, < 0.02 (< 0.02) < 0.02, < 0.02 (< 0.02)	Dorschner, K. W. 2002a 200509
USA California 2001 (Angeleno)	480 SC	0.28	0.012	2	Fruit, w/o stone	14 28	< 0.02, < 0.02 (< 0.02) < 0.02, < 0.02 (< 0.02)	Dorschner, K. W. 2002a 200509
USA California 2001 (French)	480 SC	0.28	0.014	2	Fruit, w/o stone	14 28	0.03, 0.03 (0.03) 0.03, 0.03 (0.03)	Dorschner, K. W. 2002a 200509

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
USA Michigan 2001 (Early Golden)	480 SC	0.28	0.060	2	Fruit, w/o stone	0 7 14 28 33	0.06, 0.04 (0.05) 0.08, 0.06 (0.07) < 0.02, 0.03 (0.03) 0.03, 0.03 (0.03) < 0.02, 0.02 (0.02)	Dorschner, K. W. 2002a 200509
USA Oregon 2001 (Brooks)	480 SC	0.28	0.069	2	Fruit, w/o stone	15 26	< 0.02, < 0.02 (< 0.02) < 0.02, < 0.02 (< 0.02)	Dorschner, K. W. 2002a 200509
Japan, Nagano 2001 (Ohishiwase)	30 WG	0.6	0.015	3	Thiacloprid: Whole fruit w/o stone	0[1] 7 14 21 0[1] 7 14 21	< 0.01, < 0.01, < 0.005[2], < 0.005[2] (< 0.005) 0.06, 0.05, 0.03[2], 0.03[2] (0.04) 0.02, 0.02, 0.02[2], 0.02[2] (0.02) 0.06, 0.06, 0.02[2], 0.02[2] (0.04) < 0.01, < 0.01 < 0.01[2], < 0.01[2] (< 0.01) < 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01) < 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01) < 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01)	Anon. 1997ab, 1997ac, 1997ad, 1997ae
					Thiacloprid/amide: Whole Fruit w/o stone			

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Japan, Wakayama 2001 (Ohishiwase)	30 WG	0.6	0.015	3	Thiacloprid: Whole fruit	0[1]	< 0.01, < 0.01, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 1997ab, 1997ac, 1997ad, 1997ae
						7	0.11, 0.10, 0.07[2], 0.06[2] (0.09)	
						14	0.05, 0.05, 0.02[2], 0.02[2] (0.04)	
						21	0.06, 0.06, 0.04[2], 0.04[2] (0.05)	
					Thiacloprid/amide: Whole Fruit	0[1]	< 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01)	
						7	< 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01)	
						14	< 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01)	
						21	< 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01)	

1 sampling before last application

2 replicate analysis

Berries and other small fruits

Table 34. Thiacloprid residues resulting from foliar application to grapes (glasshouse use).

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Japan, Ishikawa 2002 (Delaware)	WG, 30	0.6	0.015	2	Thiacloprid: Berries	0[1]	< 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	Anon. 2002c, 2002d
						21	0.96, 0.94, 0.64[2], 0.64[2] (0.80)	

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
							28	0.67, 0.66, 0.53[2], 0.53[2] (0.60)	
							42	0.90, 0.88, 0.56[2], 0.54[2] (0.72)	
						Thiacloprid-amide: Berries	0[1]	< 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	
							21	< 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	
							28	< 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	
							42	< 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	
Japan, Osaka	2002 (Delaware)	WG, 30	0.6	0.015	2	Thiacloprid: Berries	0[1]	< 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	Anon. 2002c, 2002d
							21	1.9, 1.9, 1.4[2], 1.3[2] (1.6)	
							28	1.8, 1.8, 1.5[2], 1.4[2] (1.6)	
							49	1.3, 1.3, 0.85[2], 0.83[2] (1.1)	
						Thiacloprid-amide: Berries	0[1]	< 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	
							21	< 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	
							28	< 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	
							42	< 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Japan, Nagano 2002 (Kyoho)	WG, 30	0.6	0.015	2	Thiacloprid: Berries	 0[1]	 < 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	Anon. 2002a, 2002b
						21	0.46, 0.43, 0.27[2], 0.26[2] (0.36)	
						28	0.45, 0.44, 0.45[2], 0.43[2] (<u>0.44</u>)	
						42	0.37, 0.37, 0.26[2], 0.26[2] (0.32)	
					Thiacloprid-amide: Berries	 0[1]	 < 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	
						21	< 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	
						28	< 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	
						42	< 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	
Japan, Kyoto 2002 (Pione)	WG, 30	0.6	0.015	2	Thiacloprid: Berries	 0[1]	 < 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	Anon. 2002a, 2002b
						21	0.11, 0.11, 0.07[2], 0.07[2] (0.09)	
						29	0.06, 0.06, 0.05[2], 0.05[2] (0.06)	
						42	0.12, 0.12, 0.13[2], 0.12[2] (<u>0.12</u>)	
					Thiacloprid-amide: Berries	 0[1]	 < 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	
						21	< 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	
						29	< 0.02,	

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
							42	< 0.02, < 0.02[2], < 0.02[2] (< 0.02) < 0.02, < 0.02, < 0.02[2], < 0.02[2] (< 0.02)	

1 sampling before last application

2 replicate analysis

Table 35. Thiacloprid residues resulting from foliar application to strawberries (field use).

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Belgium Riemst (Elsanta)	2000	480 SC	0.12	0.02	2	Fruit	0 3	0.1 0.07	Schoening, R. and Nuesslein, F. 2001 RA-2053/00
France Ecquevilly (Majeral)	2000	480 SC	0.12	0.02	2	Fruit	0 3	0.02 0.02	Schoening, R. and Nuesslein, F. 2001 RA-2053/00
United Kingdom Thurston (Cambridge Favourite)	2000	480 SC	0.12	0.02	2	Fruit	0 3	0.06 0.09	Schoening, R. and Nuesslein, F. 2001 RA-2053/00
Germany Monheim (Elsanta)	2000	480 SC	0.12	0.02	2	Fruit	0 3	0.07 0.08	Schoening, R. and Nuesslein, F. 2001 RA-2053/00
Germany Leverkusen (Elsanta)	1999	480 SC	0.12	0.02	2	Fruit	0[1] 0 1 3 7 14	0.03 0.13 0.09 0.08 0.05 0.03	Schoening, R. 2000a RA-2006/99
Germany Monheim (Symphonie)	1999	480 SC	0.12	0.02	2	Fruit	0[1] 0 1 3 8 14	< 0.02 0.06 0.04 0.03 0.02 < 0.02	Schoening, R. 2000a RA-2006/99
France Glisolles (Pandora)	1999	480 SC	0.12	0.02	2	Fruit	0[1] 0 1 3 7 14	0.05 0.10 0.05 0.04 0.04 0.02	Schoening, R. 2000a RA-2006/99
United Kingdom Thurston (Cambridge Favourite)	1999	480 SC	0.12	0.02	2	Fruit	0[1] 0 1 3 7 14	< 0.02 0.05 0.06 0.07 0.06 0.04	Schoening, R. 2000a RA-2006/99

1 sampling before last application

Table 36. Thiacloprid residues resulting from foliar application to strawberries (greenhouse use).

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Germany Telgte 2002 (Elsanta)	480 SC	0.12	0.012	2	Fruit	0 1 3	0.34 0.31 0.25	Schoening, R. and Billian, P. 2003b RA-2081/02
Germany Bocholt 2002 (Elsanta)	480 SC	0.12	0.012	2	Fruit	0 1 3	0.34 0.33 0.21	Schoening, R. and Billian, P. 2003b RA-2081/02
Netherlands Wognum 2002 (Elsanta)	480 SC	0.12	0.012	2	Fruit	0[1] 0 1 3 7	0.07 0.13 0.12 0.12 0.13	Schoening, R. and Billian, P. 2003b RA-2081/02
Netherlands Wognum 2002 (Polka)	480 SC	0.12	0.012	2	Fruit	0[1] 0 1 3 7	0.13 0.26 0.31 0.27 0.31	Schoening, R. and Billian, P. 2003b RA-2081/02
Italy Francolino 2002 (Marmolada)	480 SC	0.12	0.012	2	Fruit	0 1 3	0.05 < 0.02 0.04	Schoening, R. and Billian, P. 2003b RA-2081/02
Spain Sant Pol de Mar 2002 (Aromas)	480 SC	0.12	0.012	2	Fruit	0 1 3	0.20 0.19 0.22	Schoening, R. and Billian, P. 2003b RA-2081/02
Spain Calella 2002 (Diamante)	480 SC	0.12	0.012	2	Fruit	0[1] 0 1 3 7	0.12 0.39 0.31 0.23 0.25	Schoening, R. and Billian, P. 2003b RA-2081/02
France Reynies 2002 (Gariguette)	480 SC	0.12	0.012	2	Fruit	0[1] 0 1 3 7	0.04 0.06 0.05 0.05 0.04	Schoening, R. and Billian, P. 2003b RA-2081/02
Japan, Saitama 1997 (Nyohou)	30 WG	0.15	0.0075	2	Thiacloprid: Whole fruit Thiacloprid/amide: Whole Fruit	 0[1] 1 3 7 0[1]	 < 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005) 0.41, 0.41, 0.34[2], 0.34[2] (0.38) 0.38, 0.35, 0.31[2], 0.28[2] (0.33) 0.24, 0.23, 0.19[2], 0.18[2] (0.21) < 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 1996d, 1996e, 1996f, 1996g

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
							1	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							3	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							7	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
Japan, Mie	1997 (Nyohou)	30 WG	0.15	0.0075	2	Thiacloprid: Whole fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 1996d, 1996e, 1996f, 1996g
							1	0.81, 0.80, 0.69[2], 0.68[2] (0.75)	
							3	0.59, 0.52, 0.72[2], 0.69[2] (0.63)	
							7	0.53, 0.50, 0.47[2], 0.47[2] (0.49)	
						Thiacloprid/amide: Whole Fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							1	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							3	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							7	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	

1 sampling before last application

2 replicate analysis

Table 37. Thiacloprid residues resulting from foliar application to currants.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Germany Burscheid 2000 (Rofet)	480 SC	0.12	0.012	3	Fruit	0[1] 0 1 3 7 14	0.10 0.28 0.23 <u>0.21</u> 0.13 0.08	Schoening, R. and Nuesslein, F. 2001a RA-2054/00
Belgium Pringen 2000 (Tsema)	480 SC	0.12	0.012	3	Fruit	0[1] 0 1 3 7 14	0.21 0.38 0.39 <u>0.35</u> 0.32 0.32	Schoening, R. and Nuesslein, F. 2001a RA-2054/00
Germany Monheim 2000 (Red Lake)	480 SC	0.12	0.012	3	Fruit	0[1] 0 1 3 7 13	0.35 0.91 0.73 <u>0.59</u> 0.39 0.32	Schoening, R. and Nuesslein, F. 2001a RA-2054/00
United Kingdom Thurston 2000 (Ben Tirran)	480 SC	0.12	0.012	3	Fruit	0[1] 0 1 3 7 14	0.06 0.13 0.12 0.19 <u>0.28</u> 0.13	Schoening, R. and Nuesslein, F. 2001a RA-2054/00
Belgium Meeffe 2001 (Rovada)	480 SC	0.12	0.012	3	Fruit	0 3	0.19 <u>0.16</u>	Schoening, R. 2002d RA-2111/01
United Kingdom Thurston 2001 (Ben Tirran)	480 SC	0.12	0.012	3	Fruit	0 3	0.43 <u>0.37</u>	Schoening, R. 2002d RA-2111/01
Germany Burscheid 2001 (Rovada)	480 SC	0.12	0.012	3	Fruit	0 3	0.24 <u>0.21</u>	Schoening, R. 2002d RA-2111/01
Germany Burscheid 2001 (Titania)	480 SC	0.12	0.012	3	Fruit	0 3	0.19 <u>0.08</u>	Schoening, R. 2002d RA-2111/01

1 sampling before last application

Table 38. Thiacloprid residues resulting from foliar application to raspberries.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Germany Wachtberg-Niederbachem 2001 (Resa)	480 SC	0.12	0.012	3	Fruit	0[1] 0 1 3 7 14	< 0.02 0.34 0.37 <u>0.15</u> 0.09 0.05	Heinemann, O. and Schoening, R. 2002 RA-2133/01
Germany Monheim 2001 (Schoenemann)	480 SC	0.12	0.012	3	Fruit	0[1] 0 1 3 7 14	0.12 0.81 0.88 <u>0.10</u> 0.08 0.06	Heinemann, O. and Schoening, R. 2002 RA-2133/01

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
United Kingdom Thurston 2001 (Malling Jewel)	480 SC	0.12 0.12 0.16	0.012 0.012 0.016	3	Fruit	0[1] 0 1 3 7 14	0.10 0.55 0.50 0.31 0.17 0.08	Heinemann, O. and Schoening, R. 2002 RA-2133/01
United Kingdom East Malling 2001 (Ample)	480 SC	0.12	0.012	3	Fruit	0[1] 0 1 3 7 14	0.04 0.29 0.31 0.27 0.19 0.09	Heinemann, O. and Schoening, R. 2002 RA-2133/01
Germany Burscheid 2002 (Winklers Sämling)	480 SC	0.12	0.012	3	Fruit	0 3	0.76 0.62	Schoening, R. and Billian, P. 2003c RA-2082/02
Germany Monheim 2002 (Schoenemann)	480 SC	0.12	0.012	3	Fruit	0 3	0.36 0.34	Schoening, R. and Billian, P. 2003c RA-2082/02
United Kingdom Thurston 2002 (Malling Jewel)	480 SC	0.12	0.012	3	Fruit	0 3	0.49 0.34	Schoening, R. and Billian, P. 2003c RA-2082/02
United Kingdom East Malling 2002 (Ample)	480 SC	0.12	0.012	3	Fruit	0 3	0.27 0.15	Schoening, R. and Billian, P. 2003c RA-2082/02

1 sampling before last application

Tropical fruits – inedible peel

Table 39. Thiacloprid residues resulting from foliar application to kiwi fruits.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
New Zealand Ramarama 2002 (Hayward)	480 SC	0.096	0.0048	1	Fruit	56 84 112 140 184	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Clay, S. 2003a LabGLP75
New Zealand Ramarama 2002 (Hayward)	480 SC	0.096	0.0048	2	Fruit	56 84 112 140 184	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Clay, S. 2003a LabGLP75
New Zealand Ramarama 2002 (Hayward)	480 SC	0.14	0.0072	1	Fruit	56 84 112 140 184	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Clay, S. 2003a LabGLP75

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
New Zealand Silvervale 2002 (Hayward)	480 SC	0.1	0.0096	2	Fruit	56 63 70 79 117	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Clay, S. 2003a LabGLP75
New Zealand Silvervale 2002 (Hayward)	480 SC	0.15	0.014	2	Fruit	56 63 70 79 177	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Clay, S. 2003a LabGLP75
New Zealand Silvervale 2002 (Hayward)	480 SC	0.1	0.0096	1	Fruit	56 63 70 79 177	0.04 0.03 < 0.02 < 0.02 < 0.02	Clay, S. 2003a LabGLP75
New Zealand Brightwater 2002 (Hayward)	480 SC	0.096	0.0096	1	Fruit	56 63 70 78 84 173	0.03 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Clay, S. 2003a LabGLP75
New Zealand Brightwater 2002 (Hayward)	480 SC	0.096	0.0096	2	Fruit	56 63 70 78 84 173	0.06 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Clay, S. 2003a LabGLP75
New Zealand T.McCormack 2003 (Hayward)	480 SC	0.19 0.19	0.0096 0.013	2	Fruit	49 57 64 70 77 83	0.10 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Clay, S. 2004 LabGLP104
New Zealand Te Puke 2003 (Hayward)	480 SC	0.19 0.19	0.0096 0.019	2	Fruit	49 56 63 70 84	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Clay, S. 2004 LabGLP104
New Zealand Katikati 2003 (Hayward)	480 SC	0.19 0.19	0.0096 0.013	2	Fruit	48 57 63 70 76 85	0.03 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Clay, S. 2004 LabGLP104
New Zealand Gisborne 2003 (Hayward)	480 SC	0.19	0.0096	2	Fruit	49 56 63 70 77 84	0.10 0.05 < 0.02 < 0.02 < 0.02 < 0.02	Clay, S. 2004 LabGLP104
New Zealand Makatu 2003 (Zespri Gold)	480 SC	0.096 0.12	0.0096	2	Fruit	63 70 77 84 91	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Clay, S. 2004 LabGLP104
New Zealand Makatu 2003 (Zespri Gold)	480 SC	0.14 0.17	0.014	2	Fruit	63 70 77 84 91	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Clay, S. 2004 LabGLP104

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
New Zealand Appleby 2003 (Hayward)	480 SC	0.19	0.0096	2	Fruit	70 77 84 91 98 105	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Clay, S. 2004 LabGLP104
New Zealand Appleby 2003 (Hayward)	480 SC	0.19	0.0096	2	Fruit	49 56 63 70 77 84	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Clay, S. 2004 LabGLP104

Bulb vegetables

Table 40. Thiacloprid residues resulting from foliar application to onions.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Brazil Sta. Inacia 1998 (Taqui / Superex)	480 SC	0.096	0.024	4	Bulb	21	< 0.02	Lancas, F. M. 1998b M-005335-01-2
Brazil Sta. Inacia 1998 (Taqui / Superex)	480 SC	0.19	0.048	4	Bulb	21	0.04	Lancas, F. M. 1998b M-005335-01-2
Germany Schifferstadt 2004 (Elody)	480 SC	0.11	0.24	3	bulb	21 28	< 0.01 < 0.01	Schoening, R. 2005 MR-068/05
Germany Schifferstadt 2004 (Elody)	480 SC	0.1 0.096 0.096	0.016	3	Bulb	0 7 10 14	0.55 0.06 0.03 0.02	Schoening, R. 2005 MR-068/05
Germany Schifferstadt 2004 (Elody)	480 SC	0.096	0.016	3	Bulb	0 7 10 14	0.69 0.05 0.03 0.03	Schoening, R. 2005 MR-068/05
Germany Aholming 2004 (BGS 194)	480 SC	0.096	0.024	3	bulb	21 28	< 0.01 < 0.01	Schoening, R. 2005 MR-068/05

Table 41. Thiacloprid residues resulting from foliar application to garlic.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Brazil Fazenda Riberao 1998 (Lavinia)	480 SC	0.14	0.036	3	Bulb	21	0.12	Lancas, F. M. 1998c M-005328-01-2
Brazil Fazenda Riberao 1998 (Lavinia)	480 SC	0.29	0.072	3	Bulb	21	0.2	Lancas, F. M. 1998c M-005328-01-2

Fruiting vegetables

Table 42. Thiacloprid residues resulting from foliar application to eggplants (glasshouse use).

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Japan, Kochi 2001 (Ryuma)	WG, 30	0.3	0.015	3	Thiacloprid: Whole fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 2001, 2001a
						1	0.44, 0.42, 0.34[2], 0.33[2] (<u>0.38</u>)	
						3	0.24, 0.22, 0.30[2], 0.30[2] (0.27)	
						7	0.1, 0.06, 0.1[2], 0.1[2] (0.09)	
					Thiacloprid-amide: Whole Fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
						1	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
						3	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
						7	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
Japan, Miyazaki 2001 (Kokuyou)	WG, 30	0.3	0.015	3	Thiacloprid: Whole fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 2001, 2001a
						1	0.27, 0.26, 0.29[2], 0.28[2] (<u>0.28</u>)	
						3	0.24, 0.23, 0.22[2], 0.21[2] (0.23)	
						7	0.15, 0.14, 0.16[2], 0.16[2] (0.15)	
					Thiacloprid-amide: Whole Fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2]	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
						1 3 7 	(< 0.005) < 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005) < 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005) < 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	

1 sampling before last application

2 replicate analysis

Table 43. Thiacloprid residues resulting from foliar application to cucumbers (field use).

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Germany Vetschau 2004 (Dolomit)	480 SC	0.10 0.098	0.033	2	Fruit	3	0.03	Schoening, R. 2005a MR-067/05
Germany Vetschau 2004 (Dolomit)	480 SC	0.10 0.11	0.035	2	Fruit	3	0.02	Schoening, R. 2005a MR-067/05
Germany Aholming 2004 (Melody)	480 SC	0.096	0.024	2	Fruit	0 3 5	0.03 0.02 0.02	Schoening, R. 2005a MR-067/05
Germany Niederhausen 2004 (Dirigent)	480 SC	0.096	0.024	2	Fruit	0 3 5	0.02 0.02 0.01	Schoening, R. 2005a MR-067/05
Spain Cabrera de Mar 1995 (Dasher II)	480 SC	0.12	0.012	3	Fruit	0[1] 0 3 7 9	0.02 0.12 <u>0.03</u> 0.02 < 0.02	Placke, F. J. 1997g RA-2066/95
Italy Borgo Piave 1995 (Hyeld)	480 SC	0.12	0.012	3	Fruit	0[1] 0 3 7 10	< 0.02 0.05 <u>0.03</u> < 0.02 < 0.02	Placke, F. J. 1997g RA-2066/95
Italy Comiso 1995 (Galo F1)	480 SC	0.12	0.012	3	Fruit	0[1] 0 3 7 10	0.02 0.16 <u>0.10</u> 0.03 < 0.02	Placke, F. J. 1997g RA-2066/95
Spain Viladecans 1995 (Dasher II)	480 SC	0.12	0.012	3	Fruit	0[1] 0 3 7 9	0.02 0.05 <u>0.02</u> 0.02 < 0.02	Placke, F. J. 1997g RA-2066/95

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Spain Cabrera de Mar 1996 (Dasher)	480 SC	0.14	0.014	3	Fruit	0[1] 0 1 3 7	0.02 0.08 <u>0.11</u> 0.08 < 0.02	Placke, F. J. 1997h RA-2116/96
Italy Fondi 1996 (Hyeld)	480 SC	0.14	0.014	3	Fruit	0[1] 0 1 3 7	< 0.02 0.04 <u>0.04</u> 0.02 < 0.02	Placke, F. J. 1997h RA-2116/96
Italy Borgo Piave 1996 (Hyeld)	480 SC	0.14	0.014	3	Fruit	0[1] 0 1 3 7	< 0.02 0.03 <u>0.03</u> 0.02 < 0.02	Placke, F. J. 1997h RA-2116/96
Spain Gava 1996 (Dasher)	480 SC	0.14	0.014	3	Fruit	0[1] 0 1 3 7	0.04 0.12 <u>0.14</u> 0.08 0.03	Placke, F. J. 1997h RA-2116/96

1 sampling before last application

Table 44. Thiacloprid residues resulting from foliar application to cucumbers (greenhouse use).

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Germany Leichlingen 1999 (Indira)	480 SC	0.22	0.014	3	Fruit	0[1] 0 1 3 7	0.04 0.13 0.12 <u>0.08</u> 0.04	Schoening, R. 2000a RA-2134/99
Germany Leichlingen 1999 (Indira)	480 SC	0.22	0.014	3	Fruit	0[1] 0 1 3 7	0.03 0.08 0.10 <u>0.07</u> 0.04	Schoening, R. 2000a RA-2134/99
Belgium Brecht 1999 (Korinda)	480 SC	0.22	0.014	3	Fruit	0[1] 0 1 3 7	0.02 0.14 0.12 <u>0.08</u> 0.03	Schoening, R. 2000a RA-2134/99
Netherlands HS Tholen 1999 (Flamingo)	480 SC	0.22 0.20 0.22	0.014	3	Fruit	0[1] 0 1 3 7	0.02 0.05 0.07 <u>0.04</u> 0.02	Schoening, R. 2000a RA-2134/99
Greece Vasilika 1996 (Venus)	480 SC	0.22	0.014	3	Fruit	0[1] 0 1 3 7	0.06 0.18 0.17 <u>0.12</u> 0.05	Placke, F. J. 1997i RA-2117/96
Italy Pozzo Ribaudo 1996 (Sprint)	480 SC	0.22	0.014	3	Fruit	0[1] 0 1 3 7	0.04 0.13 0.11 <u>0.08</u> 0.04	Placke, F. J. 1997i RA-2117/96
Italy Fondi 1996 (Hyeld)	480 SC	0.22	0.014	3	Fruit	0[1] 0 1 3 7	0.03 0.12 0.10 <u>0.07</u> 0.03	Placke, F. J. 1997i RA-2117/96

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Spain Ruescas 1996 (Alaska)	480 SC	0.22	0.014	3	Fruit	0[1] 0 1 3 7	0.07 0.25 0.18 0.15 0.09	Placke, F. J. 1997i RA-2117/96
France Avignon 1996 (Aramon)	480 SC	0.22	0.014	3	Fruit	0[1] 0 1 3 7	0.02 0.08 0.08 0.04 0.05	Placke, F. J. 1997i RA-2117/96
Italy Bosco Braccetto 1996 (Sprint)	480 SC	0.22	0.014	3	Fruit	0[1] 0 1 3 7	0.05 0.08 0.09 0.08 0.05	Placke, F. J. 1997i RA-2117/96
Spain Ruescas 1996 (Alaska)	480 SC	0.22	0.014	3	Fruit	0[1] 0 1 3 7	0.09 0.23 0.20 0.15 0.07	Placke, F. J. 1997i RA-2117/96
Spain Ruescas 1996 (Virginia)	480 SC	0.22	0.014	3	Fruit	0[1] 0 1 3 7	0.08 0.26 0.26 0.18 0.07	Placke, F. J. 1997i RA-2117/96

1 sampling before last application

Table 45. Thiacloprid residues resulting from foliar application to melons (field use).

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Greece Larisa 1995 (Midi Star)	480 SC	0.12	0.012	3	Peel	0[1] 0 3 7 10	0.03 0.08 0.02 0.02 < 0.02	Placke, F. J. 1997j RA-2061/95
					Pulp	0[1] 0 3 7 10	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	
					Whole fruit[2]	0[1] 0 3 7 10	< 0.02 0.04 < 0.02 < 0.02 < 0.02	
Italy Trinitapoli 1995 (Leglend)	480 SC	0.12	0.012	3	Peel	0[1] 0 3 7 10	0.02 0.23 0.08 0.05 < 0.02	Placke, F. J. 1997j RA-2061/95
					Pulp	0[1] 0 3 7 10	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	
					Whole fruit[2]	0[1]	< 0.02	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
						0 3 7 10	0.09 0.03 0.02 < 0.02	
Greece Larisa 1996 (Gold Star F1)	480 SC	0.14	0.014	3	Whole fruit Peel Pulp Whole fruit[2]	0[1] 0 1 3 7 3 7 3 7	0.04 0.05 0.06 0.36 0.28 < 0.02 < 0.02 0.06 0.04	Placke, F. J. 1997k RA-2118/96
Italy Lequile 1996 (Galia)	480 SC	0.14	0.014	3	Whole fruit Peel Pulp Whole fruit[2]	0[1] 0 1 3 7 3 7 3 7	0.03 0.06 0.06 0.23 0.23 < 0.02 < 0.02 0.06 0.06	Placke, F. J. 1997k RA-2118/96
France Verlhac7Tescou 2000 (Figaro)	480 SC	0.14	0.014	3	Whole fruit Peel Pulp	0 3 3 3	0.07 0.05 0.13 < 0.02	Schoening, R. and Nuesslein, F. 2001b RA-2115/00
France Sarrians 2000 (Sirio)	480 SC	0.14	0.014	3	Whole fruit Peel Pulp	0 3 3 3	0.05 0.02 0.05 < 0.02	Schoening, R. and Nuesslein, F. 2001b RA-2115/00

1 sampling before last application

2 calculated

Table 46. Thiacloprid residues resulting from foliar application to melons (glasshouse use).

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Japan, Ishikawa 1997 (Arlseseinunatu II)	WG, 30	0.38	0.015	3	Thiacloprid: Pulp	0[1] 1 3	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005) < 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005) < 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 1997d, 1997e, 1997f, 1997g

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
							6	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
						Thiacloprid-amide: Pulp	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							1	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							3	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							6	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
Japan, Aichi	1997 (Natsukei No. 15)	WG, 30	0.38	0.015	3	Thiacloprid: Pulp	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 1997d, 1997e, 1997f, 1997g
							1	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							3	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							7	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
						Thiacloprid-amide: Pulp	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							1	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							3	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
						7	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	

1 sampling before last application

2 replicate analysis

Table 47. Thiacloprid residues resulting from foliar application to watermelons (field use).

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Greece Larisa 1995 (Crimson Sweet)	480 SC	0.12	0.012	3	Peel	0[1] 0 3 7 10	< 0.02 0.03 < 0.02 < 0.02 < 0.02	Placke, F. J. 1997j RA-2061/95
					Pulp	0[1] 0 3 7 10	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	
					Whole fruit[2]	0[1] 0 3 7 10	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	
Spain Gavá 1995 (Super Sugarbaby)	480 SC	0.12	0.012	3	Peel	0[1] 0 3 7 10	0.04 0.07 0.03 < 0.02 < 0.02	Placke, F. J. 1997j RA-2061/95
					Pulp	0[1] 0 3 7 10	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	
					Whole fruit[2]	0[1] 0 3 7 10	< 0.02 0.03 < 0.02 < 0.02 < 0.02	
Spain Gavá 1996 (Patanegra)	480 SC	0.14	0.014	3	Whole fruit	0[1] 0 1 3 7	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Placke, F. J. 1997k RA-2118/96
					Peel	3 7	< 0.02 < 0.02	
					Pulp	3 7	< 0.02 < 0.02	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Spain La Almunia 1996 (Meridiam)	480 SC	0.14	0.014	3	Whole fruit	0[1] 0 1 3 7	0.03 0.08 0.07 0.06 0.06	Placke, F. J. 1997k RA-2118/96
					Peel	3 7	0.10 < 0.02	
					Pulp	3 7	< 0.02 < 0.02	

1 sampling before last application

2 calculated

Table 48. Thiacloprid residues resulting from foliar application to watermelons (glasshouse use).

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Japan, Ishikawa 2002 (Ajihimitsu)	30 WG	0.3	0.015	3	Thiacloprid: Pulp	0[1]	< 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01)	Anon. 2002f, 2002g
						1	0.05, 0.04, 0.04[2], 0.04[2] (0.04)	
						3	0.1, 0.1, 0.09[2], 0.09[2] (0.1)	
						7	0.08, 0.07, 0.07[2], 0.07[2] (0.07)	
					Thiacloprid-amide: Pulp	0[1]	< 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01)	
						1	< 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01)	
						3	< 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01)	
						7	< 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01)	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Japan, Miyazaki 2002 (Madarball No. 2)	30 WG	0.36	0.015	3	Thiacloprid: Pulp	0[1]	< 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01)	Anon. 2002f, 2002g
						1	0.08, 0.07, 0.07[2], 0.07[2] (0.07)	
						3	0.02, 0.02, 0.02[2], 0.02[2] (0.02)	
						7	0.06, 0.06, 0.05[2], 0.05[2] (0.06)	
					Thiacloprid-amide: Pulp	0[1]	< 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01)	
						1	< 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01)	
						3	< 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01)	
						7	< 0.01, < 0.01, < 0.01[2], < 0.01[2] (< 0.01)	

1 sampling before last application

2 replicate analysis

Table 49. Thiacloprid residues resulting from foliar application to tomatoes (field use).

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Italy Andria 1995 (EXH 98063)	480 SC	0.18	0.018	2	Fruit	0[1] 0 1 3 7	0.02 0.17 0.09 <u>0.03</u> 0.02	Placke, F. J. 1997l RA-2068/95
France Pernes les Fontaines 1995 (Levica)	480 SC	0.18	0.064	2	Fruit	0[1] 0 1 3 7	0.02 0.07 0.07 <u>0.02</u> < 0.02	Placke, F. J. 1997l RA-2068/95
France Tarascon 1995 (Cannegrow)	480 SC	0.18	0.064	2	Fruit	0[1] 0 1 3 8	0.04 0.14 0.12 <u>0.03</u> 0.03	Placke, F. J. 1997l RA-2068/95

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Italy Borgo Piave 1995 (Sunseed 6078)	480 SC	0.18	0.018	2	Fruit	0[1] 0 1 3 7	0.03 0.08 0.06 <u>0.05</u> 0.05	Placke, F. J. 1997l RA-2068/95
Italy Giorio 1996 (Marmende)	480 SC	0.22	0.014	2	Fruit	0 3 7	0.14 0.12 <u>0.16</u>	Placke, F. J. 1997n RA-2122/96
France Les Valayans 1996 (Lerika)	480 SC	0.22	0.014	2	Fruit	0 3 7	0.06 <u>0.04</u> 0.02	Placke, F. J. 1997n RA-2122/96
France Tarascon 1996 (Cannegrow)	480 SC	0.22	0.014	2	Fruit	0 3 7	0.18 <u>0.09</u> 0.03	Placke, F. J. 1997n RA-2122/96
Italy Andria 1996 (Red Setter)	480 SC	0.14	0.014	2	Fruit	0 3 7	0.16 0.10 0.03	Placke, F. J. 1997n RA-2122/96

1 sampling before last application

Table 50. Thiacloprid residues resulting from foliar application to tomatoes (greenhouse use).

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Germany Langenfeld-Reusrath 1995 (Hildares)	480 SC	0.18	0.012	3	Fruit	0[1] 0 1 3 7	0.11 0.26 0.19 <u>0.15</u> 0.14	Placke, F. J. 1997m RA-2067/95
Germany Langenfeld-Reusrath 1995 (Piranto)	480 SC	0.18	0.012	3	Fruit	0[1] 0 1 3 7	0.02 0.22 0.20 <u>0.25</u> 0.03	Placke, F. J. 1997m RA-2067/95
Spain La Redonda 1995 (Daniela)	480 SC	0.18	0.012	3	Fruit	0[1] 0 1 3 7	0.16 0.15 0.25 <u>0.18</u> 0.12	Placke, F. J. 1997m RA-2067/95
Spain Ruescas 1995 (Billante)	480 SC	0.18	0.012	3	Fruit	0[1] 0 1 3 7	0.08 0.28 0.20 0.14 <u>0.19</u>	Placke, F. J. 1997m RA-2067/95
Germany Langenfeld 1996 (Ferrari)	480 SC	0.22	0.014	3	Fruit	0 3 7	0.23 <u>0.12</u> 0.08	Placke, F. J. 1997o RA-2123/96
Germany Leichlingen 1996 (Panovy)	480 SC	0.22	0.014	3	Fruit	0 3 7	0.10 <u>0.07</u> 0.06	Placke, F. J. 1997o RA-2123/96
Spain Ruescas 1996 (Brillante)	480 SC	0.22	0.014	3	Fruit	0 3 7	0.22 0.24 <u>0.29</u>	Placke, F. J. 1997o RA-2123/96

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
France Noves 1996 (Cecilia)	480 SC	0.22	0.014	3	Fruit	0 3 7	0.18 <u>0.12</u> 0.09	Placke, F. J. 1997o RA-2123/96
Japan, Nagano 1997 (Momotaro)	30 WG	0.38	0.015	3	Thiacloprid: Whole fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 1997x, 1997y, 1997z, 1997aa
						1	0.17, 0.16, 0.12[2], 0.11[2] (0.14)	
						3	0.15, 0.15, 0.11[2], 0.10[2] (0.13)	
						7	0.21, 0.21, 0.13[2], 0.12[2] (0.17)	
					Thiacloprid-amide: Whole Fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
						1	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
						3	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
						7	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Japan, Wakayama	1997 (Ohgatafukuju)	30 WG	0.38	0.015	3	Thiacloprid: Whole fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 1997x, 1997y, 1997z, 1997aa
							1	0.09, 0.08, 0.06[2], 0.06[2] (0.07)	
							3	0.05, 0.05, 0.06[2], 0.05[2] (0.05)	
							7	0.08, 0.08, 0.04[2], 0.04[2] (0.06)	
						Thiacloprid-amide: Whole Fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							1	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							3	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							7	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	

1 sampling before last application

2 replicate analysis

Table 51. Thiacloprid residues resulting from drip application to tomatoes (greenhouse use).

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Belgium Putte	1998 (Tradino)	480 SC	0.25[1]	0.0096	1	Fruit	1	< 0.02	Schoening, R. 1999c RA-2072/98
							3	< 0.02	
							7	< 0.02	
							14	0.02	
							21	0.02	
							28	< 0.02	
							35	< 0.02	
							49	< 0.02	
Belgium Katelinje-Waver	1998 (Fausto)	480 SC	0.24[1]	0.0096	1	Fruit	1	< 0.02	Schoening, R. 1999c RA-2072/98
							3	< 0.02	
							7	< 0.02	
							14	0.02	
							21	0.02	
							28	< 0.02	
							35	< 0.02	
							49	< 0.02	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Belgium Putte 1998 (Tradino)	480 SC	0.24[1]	0.0096	1	Fruit	1 3 7 14 21 28 35 49	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Schoening, R. 1999c RA-2072/98
Belgium O-L-V Waver 1998 (Blitz)	480 SC	0.23[1]	0.0096	1	Fruit	1 3 7 14 21 28 35 49	< 0.02 < 0.02 0.03 0.03 < 0.02 < 0.02 < 0.02 < 0.02	Schoening, R. 1999c RA-2072/98
Belgium O-L-V Waver 1999 (Style)	480 SC	0.24[1]	0.096	1	Fruit	1 3 7 15 21 28 35 49	< 0.02 < 0.02, < 0.02 (< 0.02) 0.02 0.03 0.03 0.02 < 0.02 < 0.02	Schoening, R. 2000b RA-2072/99
Netherlands Steenbergen 1999 (Jamaica)	480 SC	0.24[1]	0.096	1	Fruit	1 3 7 14 21 28 35 49	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Schoening, R. 2000b RA-2072/99
Belgium Katelinje-Waver 1999 (Tradiro)	480 SC	0.22[1]	0.096	1	Fruit	1 3 7 14 21 28 35 49	< 0.02 < 0.02 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Schoening, R. 2000b RA-2072/99
Netherlands Wouwse Plantage 1999 (Ambiance)	480 SC	0.25	0.096	1	Fruit	1 3 7 14 21 28 35 49	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Schoening, R. 2000b RA-2072/99

1 corresponding to 0.0096 kg ai per 1000 plants

Table 52. Thiacloprid residues resulting from foliar application to sweet peppers (field use).

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Spain Viladecans 1995 (Lipari)	480 SC	0.18	0.012	2	Fruit	0[1] 0 1 3 7	< 0.02 0.19 0.24 <u>0.06</u> 0.04	Placke, F. J. 1997p RA-2070/95
Italy Trinitapoli 1995 (Antares)	480 SC	0.18	0.018	2	Fruit	0[1] 0 1 3 7	< 0.02 0.05 0.05 <u>0.05</u> < 0.02	Placke, F. J. 1997p RA-2070/95
Italy C. da S. Giorgio 1995 (Pacific)	480 SC	0.18	0.018	2	Fruit	0[1] 0 1 3 7	0.05 0.15 0.16 <u>0.10</u> 0.07	Placke, F. J. 1997p RA-2070/95
Spain Malgrat de Mar 1995 (Italiano)	480 SC	0.18	0.012	2	Fruit	0[1] 0 1 3 7	< 0.02 0.41 0.24 <u>0.11</u> 0.10	Placke, F. J. 1997p RA-2070/95
Spain Vilanovadel Valles 1996 (Largo italiano)	480 SC	0.22	0.014	2	Fruit	0 3 7	0.49 <u>0.45</u> 0.21	Placke, F. J. 1997r RA-2119/96
Italy Pigno 1996 (Rino)	480 SC	0.22	0.014	2	Fruit	0 3 7	0.19, 0.18 (0.19) 0.17, 0.15 (0.16) 0.21, 0.20 (<u>0.21</u>)	Placke, F. J. 1997r RA-2119/96
Italy Andria 1996 (Antares)	480 SC	0.14	0.014	2	Fruit	0 3 7	0.07, 0.07 (0.07) 0.07, 0.07 (0.07) 0.04, 0.04 (0.04)	Placke, F. J. 1997r RA-2119/96
France Pernes les Fontaines 1996 (Lipari)	480 SC	0.22	0.014	2	Fruit	0 3 7	0.15, 0.14 (0.15) 0.07, 0.07 (0.07) 0.09, 0.06 (<u>0.08</u>)	Placke, F. J. 1997r RA-2119/96

1 sampling before last application

Table 53. Thiacloprid residues resulting from foliar application to sweet peppers (greenhouse use).

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Netherlands GS Breda 1995 (Cuby)	480 SC	0.18	0.012	3	Fruit	0[1] 0 1 3 7	0.07 0.09 0.09 <u>0.07</u> 0.05	Placke, F. J. 1997q RA-2069/95
Netherlands SW Heerle 1995 (Spirit)	480 SC	0.18	0.012	3	Fruit	0[1] 0 1 3 7	0.07 0.13 0.14 0.08 <u>0.10</u>	Placke, F. J. 1997q RA-2069/95

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Spain Ruescas 1995 (Atol)	480 SC	0.18	0.012	3	Fruit	0[1] 0 1 3 7	0.27 0.54 0.47 0.37 0.34	Placke, F. J. 1997q RA-2069/95
Spain Ruescas 1995 (Espartaco)	480 SC	0.18	0.012	3	Fruit	0[1] 0 1 3 7	0.49 0.72 0.65 0.36 0.38	Placke, F. J. 1997q RA-2069/95
Spain El Ejido 1996 (Dallas)	480 SC	0.22	0.014	3	Fruit	0 3 7	0.48 0.37 0.32	Placke, F. J. 1997s RA-2120/96
France St. Remy de Provence 1996 (Cyrano)	480 SC	0.22	0.014	3	Fruit	0 3 7	0.13 0.11 0.09	Placke, F. J. 1997s RA-2120/96
Spain El Ejido 1996 (Mazurka)	480 SC	0.22	0.014	3	Fruit	0 3 7	0.36 0.33 0.21	Placke, F. J. 1997s RA-2120/96
France Les Sablons 1996 (Laser)	480 SC	0.22	0.014	3	Fruit	0 3 7	0.13 0.08 0.04	Placke, F. J. 1997s RA-2120/96
Japan, Kochi 1997 (Tosahime)	30 WG	0.3	0.015	3	Thiacloprid: Whole fruit Thiacloprid-amide: Whole Fruit	 0[1] 1 3 7 0[1] 1 3 7	 < 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005) 1.3, 1.2, 0.97[2], 0.95[2] (1.1) 0.81, 0.80, 0.89[2], 0.83[2] (0.83) 0.64, 0.58, 0.58[2], 0.56[2] (0.59) < 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005) < 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005) < 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005) < 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 1996, 1996a, 1996b, 1996c

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Japan, Miyazaki	1997 (TosahikariD)	30 WG	0.38	0.015	3	Thiacloprid: Whole fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 1996, 1996a, 1996b, 1996c
							1	2.2, 2.0, 1.9[2], 1.8[2] (2.0)	
							3	1.7, 1.6, 1.5[2], 1.5[2] (1.6)	
							7	1.5, 1.4, 1.3[2], 1.3[2] (1.4)	
						Thiacloprid-amide: Whole Fruit	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							1	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							3	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							7	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	

1 sampling before last application

2 replicate analysis

Table 54. Thiacloprid residues resulting from drip application to peppers (greenhouse use).

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Belgium Katelinje-Waver	1998 (Mazurka)	480 SC	0.30[1]	0.0096	1	Fruit	1	< 0.02	Schoening, R. 1999c RA-2072/98
							3	< 0.02	
							7	0.03	
							14	0.05	
							21	0.05	
							28	0.04	
							35	< 0.02	
							49	< 0.02	
Belgium Katelinje-Waver	1998 (Meteor)	480 SC	0.41[1]	0.0096	1	Fruit	1	< 0.02	Schoening, R. 1999c RA-2072/98
							3	< 0.02	
							7	0.03	
							14	0.05	
							21	0.04	
							28	0.02	
							35	< 0.02	
							49	< 0.02	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Belgium Katelinje-Waver 1998 (Meteor)	480 SC	0.36[1]	0.0096	1	Fruit	1 3 7 14 21 28 35 49	< 0.02 < 0.02 0.04 <u>0.05</u> 0.04 0.02 < 0.02 < 0.02	Schoening, R. 1999c RA-2072/98
Belgium Katelinje-Waver 1998 (Mazurka)	480 SC	0.28[1]	0.0096	1	Fruit	1 3 7 14 21 28 35 49	< 0.02 < 0.02 <u>0.05</u> 0.04 0.05 0.04 < 0.02 < 0.02	Schoening, R. 1999c RA-2072/98
Netherlands Steenbergen 1999 (Fiesta)	480 SC	0.29[1]	0.096	1	Fruit	1 3 7 14 21 28 35 49	< 0.02 < 0.02 0.02 <u>0.07</u> 0.07 0.06 0.05 < 0.02	Schoening, R. 2000b RA-2072/99
Belgium Rumst 1999 (Meteor)	480 SC	0.22[1]	0.096	1	Fruit	1 3 7 14 21 28 35 49	< 0.02 < 0.02 0.03 <u>0.04</u> < 0.02 < 0.02 < 0.02 < 0.02	Schoening, R. 2000b RA-2072/99
Belgium Katalijne-Waver 1999 (Mandy)	480 SC	0.32[1]	0.096	1	Fruit	1 3 8 14 21 28 35 49	< 0.02 0.02 <u>0.06</u> 0.05 0.05 0.02 < 0.02 < 0.02	Schoening, R. 2000b RA-2072/99
Netherlands Steenbergen 1999 (Spirit)	480 SC	0.32[1]	0.096	1	Fruit	1 3 7 14 21 28 35 49	< 0.02 < 0.02 < 0.02 0.02 <u>0.04</u> 0.03 0.03 < 0.02	Schoening, R. 2000b RA-2072/99

1 corresponding to 0.0096 kg ai per 1000 plant

Root and tuber vegetables

Table 55. Thiacloprid residues resulting from foliar application to potatoes.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Germany Burscheid 1997 (Hansa)	480 SC	0.096	0.012	3	Thiacloprid only: Tuber Total residue[1] Tuber	 0 21 0 21	 < 0.02 < 0.02 < 0.05 < 0.05	Schoening, R. 2000c RA-2150/97
France Quatremare 1997 (Lisa)	480 SC	0.096	0.034	3	Thiacloprid only: Tuber Total residue[1] Tuber	 0 21 0 21	 < 0.02 < 0.02 < 0.05 < 0.05	Schoening, R. 2000c RA-2150/97
Germany Burscheid 1998 (Hansa)	480 SC	0.096	0.024	3	Thiacloprid only: Tuber Total residue[1] Tuber	 0 21 0 21	 < 0.02 < 0.02 < 0.05 < 0.05	Schoening, R. 2000d RA-2068/98
United Kingdom Thurston 1998 (Desiree)	480 SC	0.096	0.012	3	Thiacloprid only: Tuber Total residue[1] Tuber	 0 21 0 21	 < 0.02 < 0.02 < 0.05 < 0.05	Schoening, R. 2000d RA-2068/98
Germany Monheim 1998 (Hansa)	480 SC	0.096 0.096 0.096	0.012 0.024 0.024	3	Thiacloprid only: Tuber Total residue[1] Tuber	 0 21 0 21	 < 0.02 < 0.02 < 0.05 < 0.05	Schoening, R. 2000d RA-2068/98
United Kingdom Inham Hall 1998 (Fianna)	480 SC	0.096	0.032	3	Thiacloprid only: Tuber Total residue[1] Tuber	 0 21 0 21	 < 0.02 < 0.02 < 0.05 < 0.05	Schoening, R. 2000d RA-2068/98
France Quatremare 1998 (Mona Lisa)	480 SC	0.096	0.012	3	Thiacloprid only: Tuber Total residue[1] Tuber	 0 21 0 21	 < 0.02 < 0.02 < 0.05 < 0.05	Schoening, R. 2000d RA-2068/98
Belgium Soreé 1998 (Bintje)	480 SC	0.096		3	Thiacloprid only: Tuber Total residue[1]	0 21 0 21	< 0.02 < 0.02 < 0.05 < 0.05	Schoening, R. 2000d RA-2068/98
Spain La Garriga 1997 (Red Pontiac)	480 SC	0.09 0.096 0.096	0.012 0.012 0.012	3	Thiacloprid only: Tuber Total residue[1] Tuber	 0 22 0 22	 < 0.02 < 0.02 < 0.05 < 0.05	Schoening, R. 2000e RA-2151/97

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Italy Molino dei Torti 1997 (Mona Lisa)	480 SC	0.096	0.012	3	Thiacloprid only: Tuber Total residue[1] Tuber	 0 21 0 21	 < 0.02 < 0.02 < 0.05 < 0.05	Schoening, R. 2000e RA-2151/97
Spain Cabrera de Mar 1998 (Jerla)	480 SC	0.096 0.09 0.096	0.012 0.012 0.012	3	Thiacloprid only: Tuber Total residue[1]	0 21 0 21	< 0.02 < 0.02 < 0.05 < 0.05	Schoening, R. 2000f RA-2069/98
Italy Borgo Piave 1998 (Symfonia)	480 SC	0.096	0.012	3	Thiacloprid only: Tuber Total residue[1] Tuber	 0 7 14 21 0 7 14 21	 < 0.02 < 0.02 < 0.02 < 0.02 < 0.05 < 0.05 < 0.05 < 0.05	Schoening, R. 2000f RA-2069/98
Italy Cervesina 1998 (Mona Lisa)	480 SC	0.096	0.012	3	Thiacloprid only: Tuber Total residue[1]	0 7 14 21 0 7 14 21	< 0.02 < 0.02 < 0.02 < 0.02 < 0.05 < 0.05 < 0.05 < 0.05	Schoening, R. 2000f RA-2069/98
France Les Valayans 1998 (Mona Lisa)	480 SC	0.096	0.012	3	Thiacloprid only: Tuber Total residue[1] Tuber	 0 21 0 21	 < 0.02 < 0.02 < 0.05 < 0.05	Schoening, R. 2000f RA-2069/98
France Noves 1998 (Mona Lisa)	480 SC	0.096	0.012	3	Thiacloprid only: Tuber Total residue[1] Tuber	 0 21 0 21	 < 0.02 < 0.02 < 0.05 < 0.05	Schoening, R. 2000f RA-2069/98
Spain Vilanova del Valles 1998 (Ped Pontiac)	480 SC	0.096 0.096 0.085	0.012	3	Thiacloprid only: Tuber Total residue[1] Tuber	 0 21 0 21	 < 0.02 < 0.02 < 0.05 < 0.05	Schoening, R. 2000f RA-2069/98
Brazil Facenda Rodeio 1998 (Jaete Bintje)	480 SC	0.14	0.024	4	Thiacloprid only: Tuber	21	< 0.02	Lancas, F. M. 1998d RE-059/98
Brazil Facenda Rodeio 1998 (Jaete Bintje)	480 SC	0.29	0.048	4	Thiacloprid only: Tuber	21	< 0.02	Lancas, F. M. 1998d RE-059/98

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Japan, Ushiku 1997 (Danshaku)	30 WG	0.3	0.015	3	Thiacloprid: Tuber, washed	0^2	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	Anon. 1997p, 1997q, 1997r, 1997s
						7	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	
						14	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	
						21	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	
					Thiacloprid-amide: Tuber, washed	0^2	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	
						7	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	
						14	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	
						21	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	
Japan, Hiroshima 1997 (Nourin No. 1)	30 WG	0.3	0.015	3	Thiacloprid: Tuber, washed	0^2	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	Anon. 1997p, 1997q, 1997r, 1997s
						7	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	
						14	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	
						21	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
					Thiacloprid-amide: Tuber, washed	0[2]	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	
						7	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	
						14	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	
						21	< 0.005, < 0.005, < 0.005[3], < 0.005[3] (< 0.005)	

1 Determined as 6-CAN

2 sampling before last application

3 replicate analysis

Cereals

Table 56. Thiacloprid residues resulting from foliar application to wheat.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Germany Burscheid 2000 (Lavett)	480 SC	0.062	0.021	2	Ear	-1	< 0.02	Schoening, R. and Eberhardt, R. 2001 RA-2120/00
						0	0.69	
						7	0.15	
						14	0.08	
					Rest of Plant	-1	< 0.02	
						0	1.3	
						7	0.15	
						14	0.09	
					Grain	21	< 0.02	
						29	< 0.02	
					Straw	21	0.11	
						29	0.14	
France La Chapelle Vendomoise 2000 (Florence Aurore)	480 SC	0.067 0.069	0.021 0.022	2	Ear	0[1]	0.16	Schoening, R. and Eberhardt, R. 2001 RA-2120/00
						0	0.83	
						7	0.36	
						14	0.51	
					Rest of Plant	0[1]	0.53	
						0	1.9	
						7	0.54	
						14	0.70	
					Grain	21	< 0.02	
					Straw	21	0.53	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Germany Monheim 2000 (Lavett)	480 SC	0.062	0.021	2	Ear Rest of Plant Grain Straw	0 21 0 21 30 30	0.58 0.05 <u>1.7</u> 0.05 < 0.<u>02</u> <u>0.07</u>	Schoening, R. and Eberhardt, R. 2001 RA-2120/00
France Villexanton 2000 (Lloyd)	480 SC	0.066 0.062	0.022 0.021	2	Ear Rest of Plant Grain Straw	0 0 21 21	0.84 <u>1.3</u> <u>0.04</u> <u>0.89</u>	Schoening, R. and Eberhardt, R. 2001 RA-2120/00
France Montamat 2000 (Apache)	480 SC	0.062	0.021	2	Ear Rest of Plant Grain Straw 	0[1] 0 7 0[1] 0 7 14 22 14 22	0.20, 0.18 (0.19) 0.87, 0.88 (0.88) 1.4, 1.3 (1.4) 0.25, 0.23 (0.24) 1.9, 1.8 (1.9) 2.4, 1.9 (<u>2.2</u>) 0.02, < 0.02 (0.02) 0.03, 0.03 (<u>0.03</u>) 1.6, 1.6 (<u>1.6</u>) 1.4, 1.4 (1.4)	Schoening, R. and Sur, R. 2001 RA-2121/00
France Martres 2000 (Sidéral)	480 SC	0.062	0.021	2	Ear Rest of Plant Grain Straw	0[1] 0 20 20	1.1 <u>1.8</u> <u>0.03</u> <u>0.97</u>	Schoening, R. and Sur, R. 2001 RA-2121/00
Germany Burscheid 2002 (Picolo)	110 OD	0.05	0.017	2	Ear Rest of Plant Grain Straw	0[1] 0 21 0[1] 0 21 35 35	0.04 0.43 0.03 0.03 <u>1.2</u> 0.04 < 0.<u>02</u> <u>0.07</u>	Schoening, R. 2004 RA/2177/02

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
France Pithienvile 2002 (Apache)	110 OD	0.05	0.017	2	Ear Rest of Plant Grain Straw	0[1] 0 0[1] 0 21 35 21 35	0.2 0.71 0.6 1.8 0.04 < 0.02 1.7 0.96	Schoening, R. 2004 RA/2177/02
Germany Burscheid 2002 (Picolo)	110 OD	0.05	0.017	1	Ear Rest of Plant Grain Straw	0 21 0 21 35 35	0.36 0.04 1.3 0.04 < 0.02 0.06	Schoening, R. 2004a RA-2177/02
France Pithienvile 2002 (Apache)	110 OD	0.05	0.017	1	Ear Rest of Plant Grain Straw	0 0 21 35 21 35	0.55 1.2 0.03 0.02 1.2 0.56	Schoening, R. 2004a RA-2178/02

1 sampling before last application

Table 57. Thiacloprid residues resulting from foliar application to barley.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Germany Burscheid 2000 (Baronesse)	480 SC	0.062	0.021	2	Ear Rest of Plant Grain Straw 	0[1] 0 6 14 0[1] 0 6 14 22 29 22 29	< 0.02 0.90 0.11 0.08 0.04 1.5 0.18 0.06 0.03 0.06 0.05 0.07	Schoening, R. and Eberhardt, R. 2002 RA-2122/00
France Monnaie 2000 (Prisma)	480 SC	0.062 0.062	0.021 0.019	2	Ear Rest of Plant Grain	0[1] 0 8 14 0[1] 0 8 14 21	0.53 2.6 0.61 0.36 1.8 3.4 1.6 0.44 0.12	Schoening, R. and Eberhardt, R. 2002 RA-2122/00

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
					Straw	21	0.41	
Germany Monheim 2000 (Alexis)	480 SC	0.062	0.021	2	Ear Rest of Plant Grain Straw	0 0 20 30 20 30	0.97 1.6 < 0.02 < 0.02 0.08 0.04	Schoening, R. and Eberhardt, R. 2002 RA-2122/00
France Courcy 2000 (Cork)	480 SC	0.068 0.062	0.021 0.021	2	Ear Rest of Plant Grain Straw	0 0 22 22	0.82 1.1 0.05 0.29	Schoening, R. and Eberhardt, R. 2002 RA-2122/00
France St. Pey De Castet 2000 (Gaelic)	480 SC	0.062 0.075	0.021 0.021	2	Ear Rest of Plant Grain Straw	0[1] 0 7 15 0[1] 0 7 15 21 21	0.52 2.0 1.5 1.4 0.25 1.2 0.76 0.99 0.45 0.88	Schoening, R. 2001e RA-2123/00
France Soulan 2000 (Nikel)	480 SC	0.062	0.021	2	Ear Rest of Plant Grain Straw	0 0 21 21	0.88 0.90 0.11 0.15	Schoening, R. 2001e RA-2123/00

1 sampling before last application

Table 58. Thiacloprid residues resulting from foliar application to rice

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
India Chakdaha 2000 (Swama masuri)	240 SC	0.18		1	Whole plant w/o roots Grain Husk Straw	0 1 3 7 15 30 39 39 39	12.0 10.2 8.5 6.98 3.0 < 0.001[1] < 0.001[1] < 0.001[1] < 0.001[1]	Anon. 2002 India-Rice-2002 Season-I, Replicate 1
	240 SC	0.18		1	Whole plant w/o roots	0 1 3 7 15 30	8.1 7.1 6.0 4.4 4.1 < 0.001[1]	Season-I, Replicate 2

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
					Grain	39	< 0.001[1]	
					Husk	39	< 0.001[1]	
					Straw	39	< 0.001[1]	
	240 SC	0.18		1	Whole plant w/o roots	0	9.1	Season-I, Replicate 3
						1	8.96	
						3	7.99	
						7	4.9	
						15	2.9	
						30	< 0.001[1]	
					Grain	39	< 0.001[1]	
					Husk	39	< 0.001[1]	
					Straw	39	< 0.001[1]	
India Chakdaha 2000 (Swama masuri)	240 SC	0.36		1	Whole plant w/o roots	0	18.9	Anon. 2002 India-Rice-2002
						1	15.98	
						3	15.1	
						7	10.9	
						15	3.7	Season-I, Replicate 1
						30	< 0.001[1]	
					Grain	39	< 0.001[1]	
					Husk	39	< 0.001[1]	
					Straw	39	< 0.001[1]	
	240 SC	0.36		1	Whole plant w/o roots	0	17.4	Season-I, Replicate 2
						1	16.8	
						3	11.6	
						7	8.8	
						15	4.0	
						30	< 0.001[1]	
					Grain	39	< 0.001[1]	
					Husk	39	< 0.001[1]	
					Straw	39	< 0.001[1]	
	240 SC	0.36		1	Whole plant w/o roots	0	16.1	Season-I, Replicate 3
						1	14.3	
						3	14.0	
						7	10.1	
						15	6.9	
						30	< 0.001[1]	
					Grain	39	< 0.001[1]	
					Husk	39	< 0.001[1]	
					Straw	39	< 0.001[1]	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
India Chakdaha 2000 (Khitish)	240 SC	0.18		1	Whole plant w/o roots	0 1 3 7 15 30	11.1 8.7 8.2 4.5 2.7 < 0.001[1]	Anon. 2002 India-Rice-2002 Season-II, Replicate 1
					Grain	29	< 0.001[1]	
					Husk	29	< 0.001[1]	
					Straw	29	< 0.001[1]	
	240 SC	0.18		1	Whole plant w/o roots	0 1 3 7 15 30	8.9 9.1 7.4 5.9 5.2 < 0.001[1]	Season-II, Replicate 2
					Grain	29	< 0.001[1]	
					Husk	29	< 0.001[1]	
					Straw	29	< 0.001[1]	
	240 SC	0.18		1	Whole plant w/o roots	0 1 3 7 15 30	9.99 10.1 8.5 6.9 3.2 < 0.001[1]	Season-II, Replicate 3
					Grain	29	< 0.001[1]	
					Husk	29	< 0.001[1]	
					Straw	29	< 0.001[1]	
India Chakdaha 2000 (Khitish)	240 SC	0.36		1	Whole plant w/o roots	0 1 3 7 15 30	19.5 17.9 12.6 11.9 4.9 < 0.001[1]	Anon. 2002 India-Rice-2002 Season-II, Replicate 1
					Grain	29	< 0.001[1]	
					Husk	29	< 0.001[1]	
					Straw	29	< 0.001[1]	
	240 SC	0.36		1	Whole plant w/o roots	0 1 3 7 15 30	16.6 14.9 15.3 9.1 6.2 < 0.001[1]	Season-II, Replicate 2
					Grain	29	< 0.001[1]	
					Husk	29	< 0.001[1]	
					Straw	29	< 0.001[1]	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
	240 SC	0.36		1	Whole plant w/o roots	0	17.0	Season-II, Replicate 3
						1	17.5	
						3	16.3	
						7	12.3	
						15	5.9	
						30	< 0.001[1]	
					Grain	29	< 0.001[1]	
					Husk	29	< 0.001[1]	
					Straw	29	< 0.001[1]	
India Chakdaha 2001 (Swama masuri)	240 SC	0.18		1	Whole plant w/o roots	0	8.98	Anon. 2002 India-Rice-2002 Season-III, Replicate 1
						1	8.4	
						3	8.5	
						7	5.3	
						15	2.5	
						30	< 0.001[1]	
					Grain	44	< 0.001[1]	
					Husk	44	< 0.001[1]	
					Straw	44	< 0.001[1]	
	240 SC	0.18		1	Whole plant w/o roots	0	10.96	Season-III, Replicate 2
						1	7.1	
						3	7.9	
						7	6.7	
						15	3.9	
						30	< 0.001[1]	
					Grain	44	< 0.001[1]	
					Husk	44	< 0.001[1]	
					Straw	44	< 0.001[1]	
	240 SC	0.18		1	Whole plant w/o roots	0	9.8	Season-III, Replicate 3
						1	10.7	
						3	6.6	
						7	4.98	
						15	3.6	
						30	< 0.001[1]	
					Grain	44	< 0.001[1]	
					Husk	44	< 0.001[1]	
					Straw	44	< 0.001[1]	
India Chakdaha 2001 (Swama masuri)	240 SC	0.36		1	Whole plant w/o roots	0	16.7	Anon. 2002 India-Rice-2002 Season-III, Replicate 1
						1	14.2	
						3	13.96	
						7	10.2	
						15	3.4	
						30	< 0.001[1]	
					Grain	44	< 0.001[1]	
					Husk	44	< 0.001[1]	
					Straw	44	< 0.001[1]	

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
		240 SC	0.36		1	Whole plant w/o roots	0	15.9	Season-III, Replicate 2
							1	16.6	
							3	12.2	
							7	8.4	
							15	5.0	
							30	< 0.001[1]	
						Grain	44	< 0.001[1]	
						Husk	44	< 0.001[1]	
						Straw	44	< 0.001[1]	
		240 SC	0.36		1	Whole plant w/o roots	0	18.1	Season-III, Replicate 3
							1	14.7	
							3	14.8	
							7	12.0	
							15	5.5	
							30	< 0.001[1]	
						Grain	44	< 0.001[1]	
						Husk	44	< 0.001[1]	
						Straw	44	< 0.001[1]	

1 limit of detection

Table 59: Thiacloprid residues resulting from granular application to rice.

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Japan Miyagi	1997 (Sasanishiki)	GR, 15	1.5	-	1	Thiacloprid: Grain	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 1997t, 1997u, 1997v, 1997w
							152	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
						Thiacloprid-amide: Grain	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
							152	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	
Japan, Kagoshima	1997 (Koshihikari)	GR, 15	1.5	-	1	Thiacloprid: Grain	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2] (< 0.005)	Anon. 1997t, 1997u, 1997v, 1997w
							117	< 0.005, < 0.005, < 0.005[2], < 0.005[2]	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
							(< 0.005)	
					Thiacloprid-amide:			
					Grain	0[1]	< 0.005, < 0.005, < 0.005[2], < 0.005[2]	
							(< 0.005)	
						117	< 0.005, < 0.005, < 0.005[2], < 0.005[2]	
							(< 0.005)	

1 sampling before last application

2 replicate analysis

Table 60. Thiacloprid residues resulting from foliar application to maize.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
France	110	0.075	0.025	2	Green material	0[1]	0.31	Diot, R.
Saint Symphorien	OD					0	1.7	2005
d'Ancelles						2	0.89	RA-2512/04
						7	0.76	
2004						21	0.56	
(Texxud)								
					Ear, corn	0[1]	< 0.01	
						0	< 0.01	
						2	< 0.01	
						7	< 0.01	
						21	< 0.01	
					Kernel	30	< 0.01	
Germany	110	0.075	0.025	2	Green material	0[1]	< 0.05	Diot, R.
Leverkusen	OD					0	0.90	2005
						2	0.18	RA-2512/04
2004						7	0.10	
(Justine)						21	< 0.05	
					Ear, corn	0[1]	< 0.01	
						0	0.03	
						2	0.03	
						7	0.02	
						21	0.01	
					Kernel	30	< 0.01	
Germany	110	0.075	0.025	2	Green material	0[1]	0.21	Diot, R.
Gersthofen	OD					0	0.85	2005
						2	0.67	RA-2512/04
2004						7	0.42	
(Banguy)						22	0.28	
					Ear, corn	0[1]	0.01	
						0	0.02	
						2	0.04	
						7	0.03	
						22	0.04	
					Kernel	31	< 0.01	

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
France Bacquepuis 2004 (Anjou 285)	110 OD	0.075	0.025	2	Green material Ear, corn Kernel	0[1] 0 2 7 21 0[1] 0 2 7 21 31	0.17 0.87 0.81 0.55 0.66 < 0.01 0.02 0.03 0.02 0.02 < 0.01	Diot, R. 2005 RA-2512/04
France Ambérieux 2004 (34B23)	110 OD	0.075	0.025	2	Green material Ear, corn Kernel	0[1] 0 3 7 22 0[1] 0 3 7 22 31	0.33 1.7 1.7 1.1 0.58 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	Diot, R. 2005a RA-2511/04
Spain Vila-sacra 2004 (PR32R42)	110 OD	0.075	0.025	2	Green material Ear, corn Kernel	0[1] 0 3 7 21 0[1] 0 3 7 21 30	0.12 0.46 0.36 0.32 0.10 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	Diot, R. 2005a RA-2511/04
Italy Albaro Di Ronco All'Adige 2004 (PR-33-A46)	110 OD	0.075	0.025	2	Green material Ear, corn Kernel	0[1] 0 3 7 21 0[1] 0 3 7 21 28	0.14 0.74 0.71 0.63 0.13 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	Diot, R. 2005a RA-2511/04

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Greece Xehasmeni-Imathias 2004 (Brasco)		110 OD	0.075	0.025	2	Green material	0[1] 0 2 7 21	0.62 1.9 1.2 1.3 1.8	Diot, R. 2005a RA-2511/04
						Ear, corn	0[1] 0 2 7 21	0.04, 0.04, 0.03 (0.04) 0.16, 0.15, 0.15 (0.15) 0.08, 0.07, 0.08 (0.08) 0.11, 0.11, 0.11 (0.11) 0.14, 0.12, 0.12 (<u>0.13</u>)	
						Kernel	31	< 0.01	

1 sampling before last application

Nuts and seeds

Table 61. Thiacloprid residues resulting from foliar application to walnuts.

Location (variety)	Year	Form	Application			Analysis			Reference, Report No.
			kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Italy Villadose 2003 (Lara)		480 SC	0.17 0.19	0.03 0.03	2	Nutmeat	0 3 7 14	< 0.005 < 0.005 < 0.005 < 0.005	Baravelli, P. L. 2003 AGRI012/03DEC
Italy Villadose 2003 (Lara)		480 SC	0.17 0.19	0.03 0.03	2	Nutmeat	0 3 7 14	< 0.005 < 0.005 < 0.005 < 0.005	Baravelli, P. L. 2003 AGRI012/03DEC
Italy San Dona di Piave 2003 (Lara)		480 SC	0.19 0.19	0.03 0.03	2	Nutmeat	0 3 7 14	< 0.005 < 0.005 < 0.005 < 0.005	Baravelli, P. L. 2003 AGRI012/03DEC
Italy San Dona di Piave 2003 (Lara)		480 SC	0.19 0.19	0.03 0.03	2	Nutmeat	0 3 7 14	< 0.005 < 0.005 < 0.005 < 0.005	Baravelli, P. L. 2003 AGRI012/03DEC

Table 62. Thiacloprid residues resulting from foliar application to almonds

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
USA California 2000 (Monterey)	480 SC	0.14 0.15 0.15	0.029	3	Thiacloprid only: Nutmeat Hulls Total residue[1]: Nutmeat Hulls	 14 14 14 14	 < 0.01 1.4 < 0.01 1.4	Harbin, A. M. 2004 110307
USA California, Kerman 2000 (Monterey)	480 SC	0.14 0.14 0.14	0.005	3	Thiacloprid only: Nutmeat Hulls Total residue[1]: Nutmeat Hulls	 14 14 14 14	 < 0.01 1.5 < 0.01 1.5	Harbin, A. M. 2004 110307
USA California, Hughson 2000 (Merced)	480 SC	0.14 0.14 0.15	0.028 0.028 0.029	3	Thiacloprid only: Nutmeat Hulls Total residue[1]: Nutmeat Hulls	 14 14 14 14	 < 0.01 0.99 < 0.01 1.0	Harbin, A. M. 2004 110307
USA California, Hughson 2000 (Merced)	480 SC	0.14 0.14 0.14	0.006 0.006 0.007	3	Thiacloprid only: Nutmeat Hulls Total residue[1]: Nutmeat Hulls	 14 14 14 14	 < 0.01 1.3 < 0.01 1.3	Harbin, A. M. 2004 110307
USA California, Porterville 2000 (Mission)	480 SC	0.14 0.14 0.14	0.025 0.026 0.025	3	Thiacloprid only: Nutmeat Hulls Total residue[1]: Nutmeat Hulls	 10 10 10 10	 < 0.01 2.1 < 0.01 2.1	Harbin, A. M. 2004 110307
USA California, Porterville 2000 (Mission)	480 SC	0.14 0.14 0.14	0.006 0.006 0.007	3	Thiacloprid only: Nutmeat Hulls Total residue[1]: Nutmeat Hulls	 10 10 10 10	 < 0.01 1.8 < 0.01 1.8	Harbin, A. M. 2004 110307

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
USA California, Glenn 2000 (Non-Pareil)	480 SC	0.14 0.14 0.14	0.038 0.038 0.033	3	Thiacloprid only: Nutmeat Hulls Total residue[1]: Nutmeat Hulls	 14 14 14 14	 < 0.01 1.8 < 0.01 1.8	Harbin, A. M. 2004 110307
USA California, Glenn 2000 (Non-Pareil)	480 SC	0.14	0.007	3	Thiacloprid only: Nutmeat Hulls Total residue[1]: Nutmeat Hulls	 14 14 14 14	 < 0.01 3.4 < 0.01 3.4	Harbin, A. M. 2004 110307
USA California, Glenn 2000 (Non-Pareil)	70 WG	0.14 0.14 0.14	0.038 0.038 0.033	3	Thiacloprid only: Nutmeat Hulls Total residue[1]: Nutmeat Hulls	 14 14 14 14	 0.01 2.0 0.01 2.0	Harbin, A. M. 2004 110307
USA California, Glenn 2000 (Non-Pareil)	70 WG	0.14	0.007	3	Thiacloprid only: Nutmeat Hulls Total residue[1]: Nutmeat Hulls	 14 14 14 14	 < 0.01 4.9 < 0.01 4.9	Harbin, A. M. 2004 110307
USA California, Terra Bella 2000 (Monterey)	480 SC	0.14	0.022	3	Thiacloprid only: Nutmeat Hulls Total residue[1]: Nutmeat Hulls	 8 14 20 23 8 14 20 23 8 14 20 23 8 14 20 23	 < 0.01 < 0.01 < 0.01 < 0.01 2.3 3.3 2.8 1.8 < 0.01 < 0.01 < 0.01 < 0.01 2.3 3.3 2.8 1.8	Harbin, A. M. 2004 110307

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
USA California, Terra Bella 2000 (Monterey)	480 SC	0.14	0.007	3	Thiacloprid only: Nutmeat Hulls Total residue[1]: Nutmeat Hulls 	 8 14 20 23 8 14 20 23 8 14 20 23 8 14 20 23	 < 0.01 < 0.01 < 0.01 < 0.01 3.8 3.7 4.5 2.4 < 0.01 < 0.01 < 0.01 < 0.01 3.8 3.7 4.6 2.5	Harbin, A. M. 2004 110307
USA California, Terra Bella 2000 (Monterey)	70 WG	0.14	0.022	3	Thiacloprid only: Nutmeat Hulls Total residue[1]: Nutmeat Hulls 	 8 14 20 23 8 14 20 23 8 14 20 23 8 14 20 23	 < 0.01 < 0.01 < 0.01 < 0.01 2.0 3.2 2.6 1.4 < 0.01 < 0.01 < 0.01 < 0.01 2.1 3.2 2.6 1.4	Harbin, A. M. 2004 110307
USA California, Terra Bella 2000 (Monterey)	70 WG	0.14	0.007	3	Thiacloprid only: Nutmeat Hulls Total residue[1]: Nutmeat Hulls 	 8 14 20 23 8 14 20 23 8 14 20 23 8 14 20 23	 < 0.01 < 0.01 < 0.01 < 0.01 2.4 3.3 2.4 2.3 < 0.01 < 0.01 < 0.01 < 0.01 2.4 3.4 2.4 2.4	Harbin, A. M. 2004 110307

1 Determined as sum of thiacloprid, thiacloprid -amide and 4-hydroxy-thiacloprid-amide

Table 63. Thiacloprid residues resulting from foliar application to pecan.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
USA Georgia, Chula 2000 (Summer)	480 SC	0.14 0.15 0.14	0.023 0.027 0.025	3	Thiacloprid only: Nutmeat Total residue[1]: Nutmeat	 13 13	 < 0.01 < 0.01	Harbin, A. M. 2004 110307
USA Georgia, Chula 2000 (Summer)	480 SC	0.14 0.14 0.14	0.004 0.005 0.005	3	Thiacloprid only: Nutmeat Total residue[1]: Nutmeat	 13 13	 < 0.01 < 0.01	Harbin, A. M. 2004 110307
USA Louisiana, Alexandria 2000 (Cape Fear)	480 SC	0.14	0.029	3	Thiacloprid only: Nutmeat Total residue[1]: Nutmeat	 14 14	 < 0.01 < 0.01	Harbin, A. M. 2004 110307
USA Louisiana, Alexandria 2000 (Cape Fear)	480 SC	0.14	0.006	3	Thiacloprid only: Nutmeat Total residue[1]: Nutmeat	 14 14	 < 0.01 < 0.01	Harbin, A. M. 2004 110307
USA Louisiana, Alexandria 2000 (Cape Fear)	70 WG	0.14	0.029	3	Thiacloprid only: Nutmeat Total residue[1]: Nutmeat	 14 14	 < 0.01 < 0.01	Harbin, A. M. 2004 110307
USA Louisiana, Alexandria 2000 (Cape Fear)	70 WG	0.14	0.006	3	Thiacloprid only: Nutmeat Total residue[1]: Nutmeat	 14 14	 < 0.01 < 0.01	Harbin, A. M. 2004 110307
USA Texas, Boling 2000 (Choctaw)	480 SC	0.14	0.029	3	Thiacloprid only: Nutmeat Total residue[1]: Nutmeat	 14 14	 < 0.01 < 0.01	Harbin, A. M. 2004 110307
USA Texas, Boling 2000 (Choctaw)	480 SC	0.14	0.006	3	Thiacloprid only: Nutmeat Total residue[1]: Nutmeat	 14 14	 < 0.01 < 0.01	Harbin, A. M. 2004 110307
USA Texas, Uvalde 2000 (Stuart)	480 SC	0.14 0.14 0.15	0.033 0.035 0.033	3	Thiacloprid only: Nutmeat Total residue[1]: Nutmeat	 12 12	 < 0.01 < 0.01	Harbin, A. M. 2004 110307
USA Texas, Uvalde 2000 (Stuart)	480 SC	0.14	0.007	3	Thiacloprid only: Nutmeat Total residue[1]: Nutmeat	 12 12	 < 0.01 < 0.01	Harbin, A. M. 2004 110307

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
USA Georgia, Albany 2000 (Stuart)	480 SC	0.14 0.14 0.14	0.027 0.029 0.027	3	Thiacloprid only: Nutmeat Total residue[1]: Nutmeat	 9 14 19 23 9 14 19 23	 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	Harbin, A. M. 2004 110307
USA Georgia, Albany 2000 (Stuart)	480 SC	0.14	0.005	3	Thiacloprid only: Nutmeat Total residue[1]: Nutmeat	 9 14 19 23 9 14 19 23	 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	Harbin, A. M. 2004 110307
USA Georgia, Albany 2000 (Stuart)	70 WG	0.14 0.14 0.14	0.027 0.029 0.027	3	Thiacloprid only: Nutmeat Total residue[1]: Nutmeat	 9 14 19 23 9 14 19 23	 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	Harbin, A. M. 2004 110307
USA Georgia, Albany 2000 (Stuart)	70 WG	0.14	0.005	3	Thiacloprid only: Nutmeat Total residue[1]: Nutmeat	 9 14 19 23 9 14 19 23	 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	Harbin, A. M. 2004 110307

1 Determined as sum of thiacloprid, thiacloprid -amide and 4-hydroxy-thiacloprid-amide

Oilseeds

Table 64. Thiacloprid residues resulting from foliar application to oilseed rape.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Hungary Kaposvár-Dénes-major 2000 (Valeska)	480 SC	0.096	0.031	1	Seed	31	0.02, < 0.02, < 0.02 (0.02)	Orosz, F. 2000b 00-BAY-AA-14-04
France Villettes 2001 (Pollen)	240 OD	0.096	0.032	1	Green material Straw Seed	0 30 30	1.0 0.25 0.07	Schoening, R. 2002e RA-2171/01
Germany Burscheid 2001 (Express)	240 OD	0.096	0.032	1	Green material Pod Rest of Plant Seed Straw	0 7 14 7 14 22 29 33 29	1.6 1.4 0.50 0.71 0.08 0.06 0.05 0.04 0.28	Schoening, R. 2002e RA-2171/01
Germany Burscheid 2002 (Licondor)	240 OD	0.12	0.04	1	Pod Rest of Plant Seed Straw	0 0 31 31	2.6 2.2 < 0.02 0.18	Billian, P. and Schoening, R. 2003d RA-2025/02
France Etrepagny 2002 (Zenith)	240 OD	0.12	0.04	1	Pod Rest of Plant Seed Straw	0 7 14 22 0 7 14 22 30 35 30	1.9 0.31 0.22 0.18 1.1 0.17 0.08 0.04 < 0.02 < 0.02 0.31	Billian, P. and Schoening, R. 2003d RA-2025/02
Sweden Dalby 2002 (Stratos)	240 OD	0.12	0.04	1	Pod Rest of Plant Seed Straw	0 0 29 29	3.1 1.9 0.05 0.07	Billian, P. and Schoening, R. 2003d RA-2025/02

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Germany Boerrstadt 2002 (Capitol)	240 OD	0.12	0.04	1	Pod Rest of Plant Seed Straw	0 0 30 30	2.2 <u>1.4</u> <u>0.07</u> 0.40	Billian, P. and Schoening, R. 2003d RA-2025/02
France Chambourg 2002 (Pollen)	240 OD	0.12	0.04	1	Pod Rest of Plant Seed Straw	0 7 14 21 0 7 14 21 30 36 30	0.58 0.85 0.60 0.66 <u>1.5</u> 0.12 0.05 0.04 0.08 <u>0.10</u> 0.45	Billian, P. and Schoening, R. 2003d RA-2025/02
Germany Worms-Heppenheim 2002 (Zenith)	240 OD	0.12	0.04	1	Pod Rest of Plant Seed Straw	0 7 14 0 7 14 21 29 33 29	2.1 1.1 0.69 <u>1.1</u> 0.30 0.13 <u>0.22</u> 0.16 0.18 0.18	Billian, P. and Schoening, R. 2003d RA-2025/02
Spain Vilademuls 2001 (Fabiola)	240 OD	0.12	0.032	1	Green material Seed Straw	0 30 40 40	<u>1.7</u> 0.14 <u>0.09</u> 0.17	Schoening, R. 2002f RA-2172/01
France Varennes 2001 (Constant)	240 OD	0.088	0.032	1	Green material Pod Rest of Plant Seed Straw	0 7 14 7 14 21 29 34 29	<u>1.2</u> 0.54 0.76 0.18 0.15 0.06 0.05 <u>0.07</u> 0.95	Schoening, R. 2002f RA-2172/01

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Spain Vilobi D'Onyar 2001 (Fabiola)	240 OD	0.12	0.04	1	Pod Rest of Plant Seed Straw	0 7 13 20 0 7 13 20 30 34 30	2.5 1.4 1.2 1.4 <u>1.1</u> 0.38 0.22 0.17 <u>0.33</u> 0.30 0.87	Billian, P. and Schoening, R. 2003e RA-2026/02
France Bouloc 2002 (Olara)	240 OD	0.11	0.04	1	Pod Rest of Plant Seed Straw	0 0 30 30	2.3 <u>1.1</u> <u>0.03</u> 0.10	Billian, P. and Schoening, R. 2003e RA-2026/02

Single underlined values were used for the evaluation of rape forage

Double underlined values were used for the evaluation of rape seed and white mustard seed

Table 65. Thiacloprid residues resulting from foliar application to cotton.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Spain Coria del Rio 1997 (Bravo)	480 SC	0.096	0.019	3	Thiacloprid only: Seed Total residue[1]: Seed	 21 21	 < <u>0.02</u> 0.94	Schoening, R. and Sur, R. 2000a RA-2115/97 Replicate 1
Spain Coria del Rio 1997 (Bravo)	480 SC	0.096	0.019	3	Thiacloprid only: Seed Total residue[1]: Seed	 21 21	 < <u>0.02</u> 1.1	Schoening, R. and Sur, R. 2000a RA-2115/97 Replicate 2
Greece Alexandria 1997 (Tempra)	480 SC	0.096	0.0096	3	Thiacloprid only: Seed Total residue[1]: Seed	 20 20	 < <u>0.02</u> 1.4	Schoening, R. and Sur, R. 2000a RA-2115/97
Greece Alexandria 1997 (Bravo)	480 SC	0.096	0.096	3	Thiacloprid only: Seed Total residue[1]: Seed	 21 21	 < <u>0.02</u> 2.0	Schoening, R. and Sur, R. 2000a RA-2115/97
Spain Palomares 1998 (Bravo)	480 SC	0.096	0.019	3	Thiacloprid only: Seed Total residue[1]: Seed	 21 21	 < <u>0.02</u> 0.76	Schoening, R. and Sur, R. 2000b RA-2073/98 Replicate 1

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Spain Palomares 1998 (Bravo)	480 SC	0.096	0.019	3	Thiacloprid only: Seed Total residue[1]: Seed	 21 21	 < 0.02 1.0	Schoening, R. and Sur, R. 2000b RA-2073/98 Replicate 2
Greece Trikala 1998 (Bravo)	480 SC	0.096	0.0096	3	Thiacloprid only: Seed Total residue[1]: Seed	 21 21	 < 0.02 1.7	Schoening, R. and Sur, R. 2000b RA-2073/98
Greece Alexandria 1998 (Bravo)	480 SC	0.096	0.0096	3	Thiacloprid only: Seed Total residue[1]: Seed	 21 21	 < 0.02 0.83	Schoening, R. and Sur, R. 2000b RA-2073/98
USA Louisiana, Cheneyville 1997 (Delta and Pine Land)	480 SC	0.11	0.12	3	Total residue[1]: Seed Gintrash	 13 13	 0.24, 0.33 (0.29) 9.5, 10.7 (10.1)	Koch, A. 1999 109011
USA Louisiana, Cheneyville 1997 (Delta and Pine Land)	70 WG	0.11	0.12	3	Total residue[1]: Seed Gintrash	 13 13	 0.46, 0.42 (0.44) 9.4, 8.6 (9.0)	Koch, A. 1999 109011
USA Arkansas, Shoffner 1997 (Suregrow 125)	480 SC	0.11	0.15	3	Total residue[1]: Seed gintrash	 15 15	 0.49, 0.38 (0.44) 10.9, 8.2 (9.6)	Koch, A. 1999 109011
USA Arkansas, Shoffner 1997 (Suregrow 125)	70 WG	0.11	0.15	3	Total residue[1]: Seed Gintrash	 15 15	 0.23, 0.21 (0.22) 8.6, 9.2 (8.9)	Koch, A. 1999 109011
USA Texas, Bernard 1997 (Deltapine 33B)	480 SC	0.11	0.12	3	Total residue[1]: Seed	 14	 0.84, 0.62 (0.73)	Koch, A. 1999 109011
USA Texas, Bernard 1997 (Deltapine 33B)	70 WG	0.11	0.12	3	Total residue[1]: Seed	 14	 0.31, 0.75 (0.53)	Koch, A. 1999 109011
USA Texas, Floydada 1997 (HS-26)	480 SC	0.11	0.12	3	Total residue[1]: Seed	 14	 0.09, 0.11 (0.10)	Koch, A. 1999 109011
USA Texas, Floydada 1997 (HS-26)	70 WG	0.11	0.13	3	Total residue[1]: Seed	 14	 0.08, 0.08 (0.08)	Koch, A. 1999 109011

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
USA Texas, Olton 1997 (Paymaster 145)	480 SC	0.11	0.13	3	Total residue[1]: Seed Gintrash	 14 14	 0.70, 0.64 (0.67) 8.4, 8.0 (8.2)	Koch, A. 1999 109011
USA Texas, Olton 1997 (Paymaster 145)	70 WG	0.11	0.13	3	Total residue[1]: Seed Gintrash	 14 14	 0.54, 0.50 (0.52) 9.7, 8.1 (8.9)	Koch, A. 1999 109011
USA Oklahoma, Eakly 1997 (Paymaster 183)	480 SC	0.11	0.12	2	Total residue[1]: Seed Gintrash	 17 17	 0.14, 0.23 (0.19) 1.1, 1.3 (1.2)	Koch, A. 1999 109011
USA Oklahoma, Eakly 1997 (Paymaster 183)	70 WG	0.11	0.12	3	Total residue[1]: Seed Gintrash	 17 17	 0.20, 0.18 (0.19) 1.4, 0.95 (1.2)	Koch, A. 1999 109011
USA Oklahoma, Dill City 1997 (Paymaster HS-200)	480 SC	0.11	0.13	3	Total residue[1]: Seed Gintrash	 20 20	 0.56, 0.55 (0.56) 2.5, 1.7 (2.1)	Koch, A. 1999 109011
USA Oklahoma, Dill City 1997 (Paymaster HS-200)	70 WG	0.11	0.13	3	Total residue[1]: Seed Gintrash	 20 20	 0.33, 0.41, (0.37) 2.3, 3.2 (2.8)	Koch, A. 1999 109011
USA California, Madera 1997 (Maxxa)	480 SC	0.11	0.15	3	Total residue[1]: Seed	 15	 < 0.05, < 0.05 < 0.05)	Koch, A. 1999 109011
USA California, Madera 1997 (Maxxa)	70 WG	0.11	0.15	3	Total residue[1]: Seed	 15	 < 0.05, < 0.05 (< 0.05)	Koch, A. 1999 109011
USA California, Kerman 1997 (Maxxa)	480 SC	0.11	0.15	3	Total residue[1]: Seed	 14	 0.15, 0.11 (0.13)	Koch, A. 1999 109011
USA California, Kerman 1997 (Maxxa)	70 WG	0.11	0.15	3	Total residue[1]: Seed	 14	 0.21, 0.26 (0.24)	Koch, A. 1999 109011

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
USA Mississippi, Benoit 1997 (Deltapine 50)	480 SC	0.11	0.20	3	Total residue[1]: Seed gintrash	 8 12 17 21 8 12 17 21	 0.11, 0.13 (0.12) 0.09, 0.23 (0.16) 0.26, 0.25 (0.26) 0.46, 0.22 (0.34) 3.5, 5.0 (4.3) 3.3, 3.9 (3.6) 2.4, 2.4 (2.4) 2.9, 2.2 (2.6)	Koch, A. 1999 109011
USA Mississippi, Benoit 1997 (Deltapine 50)	70 WG	0.11	0.21	3	Total residue[1]: Seed Gintrash	 12 12	 < 0.05, 0.11 (0.08) 3.1, 3.8 (3.5)	Koch, A. 1999 109011
USA California, Fresno 1997 (Maxxa)	480 SC	0.11	0.12	3	Total residue[1]: Seed	 14	 0.34, 0.15 (0.25)	Koch, A. 1999 109011
USA Georgia, Tifton 1997 (DPL5415)	480 SC	0.11	0.13	3	Total residue[1]: Seed	 16	 0.48, 0.54 (0.51)	Koch, A. 1999 109011
USA Georgia, Tifton 1997 (DPL5415)	70 WG	0.11	0.13	3	Total residue[1]: Seed	 16	 0.49, 0.63 (0.56)	Koch, A. 1999 109011

1 determined as 6-CNA

Table 66. Thiacloprid residues resulting from foliar application to sunflowers.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Hungary Fonó Somogy 2000 (Arena)	480 SC	0.097	0.031	1	Seed	30	0.02, 0.03, 0.03 (0.03)	Orosz, F. 2000c 00-BAY-AA-14-05

Tea

Table 67. Thiacloprid residues resulting from foliar application to green tea.

Location Year (variety)	Form	Application			Analysis			Reference, Report No.
		kg ai/ha	kg ai/hL	No	Sample	PHI	Residues	
Japan, Mie 1997 (Yabukita)	30 WG	0.3	0.015	1	Thiacloprid: Leaves	0[1] 3 7 14	< 0.01, < 0.01, < 0.04[2], < 0.04[2] (< 0.04) 9.9, 9.7, 7.9[2], 7.1[2] (8.2) 17, 16, 11[2], 10[2] (14) 5.1, 5.0, 4.3[2], 4.0[2] (4.6)	Fukuda, T. 1998 NR98020
					Thiacloprid-amide: Leaves	0[1] 3 7 14	< 0.01, < 0.01, < 0.04[2], < 0.04[2] (< 0.04) 0.02, 0.01, < 0.04[2], < 0.04[2] (0.04) 0.05, 0.05, 0.06, 0.06 (0.06) 0.02, 0.02, < 0.04[2], < 0.04[2] (0.04)	
Japan, Miyazaki 1997 (Yamanami)	30 WG	0.3	0.015	1	Thiacloprid: Leaves	0[1] 3 7 14	< 0.01, < 0.01, < 0.04[2], < 0.04[2] (< 0.04) 44, 42, 39[2], 38[2] (41) 19, 19, 15[2], 14[2] (17) 4.2, 3.9, 2.9[2], 2.9[2] (3.5)	Fukuda, T. 1998 NR98020
					Thiacloprid-amide: Leaves	0[1] 3 7 14	< 0.01, < 0.01, < 0.04[2], < 0.04[2] (< 0.04) 0.07, 0.06, 0.08[2], 0.08[2] (0.07) 0.07, 0.06, 0.08, 0.08 (0.07) 0.02, 0.02, < 0.04[2], < 0.04[2] (0.04)	

1 sampling before last application

2 replicate analysis

FATE OF RESIDUES IN STORAGE AND PROCESSING

Processing

In hydrolysis experiments designed to simulate typical procesing operations (Riegner, K., 1998) [methylene-^{14}C]-thiacloprid was incubated in aqueous buffer solutions at a concentration of 0.41 mg/L at 90°C (pH 4 for 20 min), 100°C (pH 5 for 60 min) and 120°C (pH 6 for 20 min) (Table 68).

At zero-time and test termination the samples were analysed by HPLC and by thin-layer chromatography. The content of radioactivity was determined by liquid scintillation counting. Material balances were established at each sampling time.

Table 68. Representative hydrolysis conditions.

Hydrolysis Conditions	Sampling time (min)	Content of thiacloprid (% of applied radioactivity *)
pH 4; 90 °C; 20 min.	0	97.6
	20	98.1
pH 5; 100 °C; 60 min.	0	97.0
	60	96.5
pH 6; 120 °C; 20 min.	0	96.9
	20	97.0

After incubation, the radioactivity in the neutralised buffer solutions represented unchanged thiacloprid (96.5–98.1% of the applied radioactivity), demonstrating that no significant hydrolytic degradation had taken place under the simulated processing conditions.

Processing studies on melons and watermelons, apples, peaches, cherries and tomatoes were reported.

Melons

Residues in melons and watermelons incurred as a result of foliar treatment . The specific trial data is presented in Table 45 and Table 47. As relevant processing step the seperation of pulp and peel was investigated. In Table 69 the residues and processing factors for melons and watermelons are summarised.

Table 69. Processing studies on melons.

Location Year (variety)	Sample	PHI	Residues	Processing factor	Reference, Report No.
MELONS					
Greece	Peel	0[1]	0.03		Placke, F. J.
Larisa		0	0.08	2	1997j
		3	0.02	1	RA-2061/95
1995		7	0.02	1	
(Midi Star)		10	< 0.02	nc	
	Pulp	0[1]	< 0.02		
		0	< 0.02	0.5	
		3	< 0.02	nc	
		7	< 0.02	nc	
		10	< 0.02	nc	
	Whole fruit[2]	0[1]	< 0.02		
		0	0.04		
		3	< 0.02		
		7	< 0.02		
		10	< 0.02		

Location Year (variety)	Sample	PHI	Residues	Processing factor	Reference, Report No.
Italy Trinitapoli 1995 (Leglend)	Peel	0[1] 0 3 7 10	0.02 0.23 0.08 0.05 < 0.02	 2.6 2.7 2.5 nc	Placke, F. J. 1997j RA-2061/95
	Pulp	0[1] 0 3 7 10	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	 0.22 0.66 1 nc	
	Whole fruit[2]	0[1] 0 3 7 10	< 0.02 0.09 0.03 0.02 < 0.02		
Greece Larisa 1996 (Gold Star F1)	Whole fruit	0[1] 0 1	0.04 0.05 0.06		Placke, F. J. 1997k RA-2118/96
	Peel	3 7	0.36 0.28	6 7	
	Pulp	3 7	< 0.02 < 0.02	0.33 0.5	
	Whole fruit[2]	3 7	0.06 0.04		
Italy Lequile 1996 (Galia)	Whole fruit	0[1] 0 1	0.03 0.06 0.06		Placke, F. J. 1997k RA-2118/96
	Peel	3 7	0.23 0.23	3.8 3.8	
	Pulp	3 7	< 0.02 < 0.02	0.33 0.33	
	Whole fruit[2]	3 7	0.06 0.06		
France Verlhac7Tescou 2000 (Figaro)	Whole fruit	0 3	0.07 0.05		Schoening, R. and Nuesslein, F. 2001b RA-2115/00
	Peel	3	0.13	2.6	
	Pulp	3	< 0.02	0.4	
France Sarrians 2000 (Sirio)	Whole fruit	0 3	0.05 0.02		Schoening, R. and Nuesslein, F. 2001b RA-2115/00
	Peel	3	0.05	2.5	
	Pulp	3	< 0.02	1	

Location Year (variety)	Sample	PHI	Residues	Processing factor	Reference, Report No.
WATERMELONS					
Greece	Peel	0[1]	< 0.02		Placke, F. J.
Larisa		0	0.03	1.5	1997j
		3	< 0.02	nc	RA-2061/95
1995		7	< 0.02	nc	
(Crimson Sweet)		10	< 0.02	nc	
	Pulp	0[1]	< 0.02		
		0	< 0.02	nc	
		3	< 0.02	nc	
		7	< 0.02	nc	
		10	< 0.02	nc	
	Whole fruit[2]	0[1]	< 0.02		
		0	< 0.02		
		3	< 0.02		
		7	< 0.02		
		10	< 0.02		
Spain	Peel	0[1]	0.04		Placke, F. J.
Gavá		0	0.07	2.3	1997j
		3	0.03	1.5	RA-2061/95
1995		7	< 0.02	nc	
(Super Sugarbaby)		10	< 0.02	nc	
	Pulp	0[1]	< 0.02		
		0	< 0.02	0.66	
		3	< 0.02	nc	
		7	< 0.02	nc	
		10	< 0.02	nc	
	Whole fruit[2]	0[1]	< 0.02		
		0	0.03		
		3	< 0.02		
		7	< 0.02		
		10	< 0.02		
Spain	Whole fruit	0[1]	< 0.02		Placke, F. J.
Gavá		0	< 0.02		1997k
		1	< 0.02		RA-2118/96
1996		3	< 0.02		
(Patanegra)		7	< 0.02		
	Peel	3	< 0.02	nc	
		7	< 0.02	nc	
	Pulp	3	< 0.02	nc	
		7	< 0.02	nc	
Spain	Whole fruit	0[1]	0.03		Placke, F. J.
La Almunia		0	0.08		1997k
		1	0.07		RA-2118/96
1996		3	0.06		
(Meridiam)		7	0.06		
	Peel	3	0.10	1.7	
		7	< 0.02	0.33	
	Pulp	3	< 0.02	0.33	
		7	< 0.02	0.33	

nc: pf cannot be calculated

1 treatment before last application

2 calculated value

RAC: raw agricultural commodity

Residues were below the limit of quantification (< 0.02 mg/kg) in melons before processing, but were quantified in peel or pulp. In cases were residues were measurable in processed fractions, transfer values were calculated by taking residues in whole fruits before processing as equal to the LOQ (0.02 mg/kg).

Apples

Two trials on apples were conducted in Italy and Germany 1995. The application rate was 0.144 kg ai/ha. The water rate was 500 and 1500 L/ha corresponding to a spray concentration of 0.02% or 0.06%, respectively. The two treatments were conducted during fruit development and fruit colouring at an interval of 14 days. The last application was carried out 14 days prior to the expected harvest (recommended waiting period). For processing, apple fruits were taken from the treated and the untreated plot on day 14 (harvest).

The washing of apples was done using domestic practice (see Figure 5). The preparation of dried apples, apple juice, dried pomace and apple sauce simulated the industrial practice at a laboratory scale (see Figures 6, 7 and 8).

The residues of thiacloprid were determined according to method 00419. The recoveries ranged from 77 to 108% at fortification levels of 0.02 and 0.20 mg/kg for fruit, from 92 to 104% at fortification level of 0.02 mg/kg for juice, from 75 to 80% at fortification level of 0.02 mg/kg for pomace, from 88 to 89% at fortification level of 0.02 mg/kg for dried fruit and from 89 to 98% at fortification levels of 0.02 and 0.20 mg/kg for sauce. The limit of quantitation (LOQ) was 0.02 mg/kg.

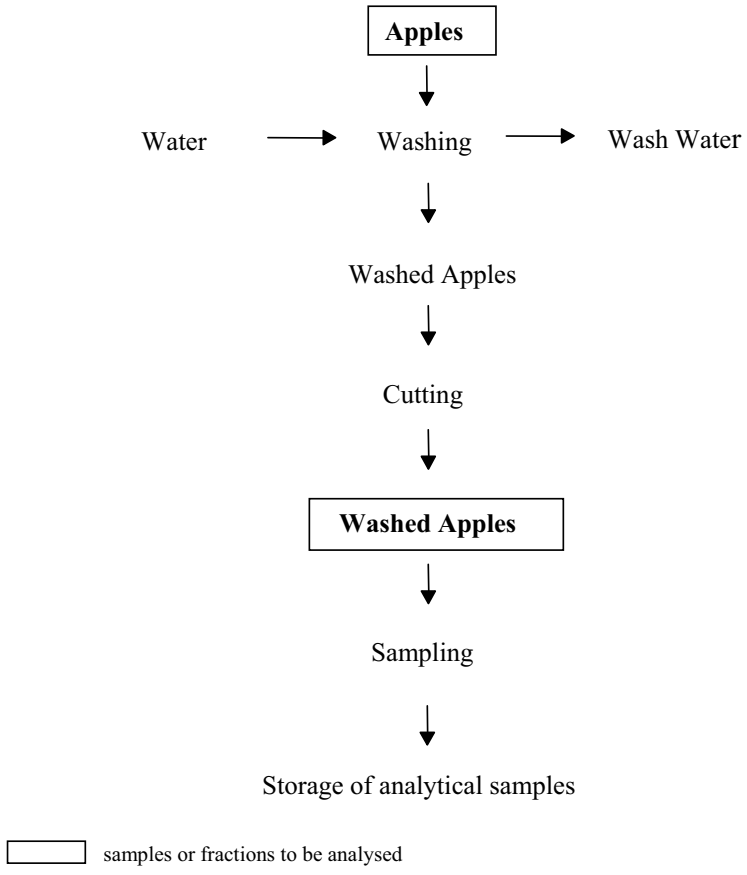


Figure 5. Flow Diagram for the preparation of washed apples.

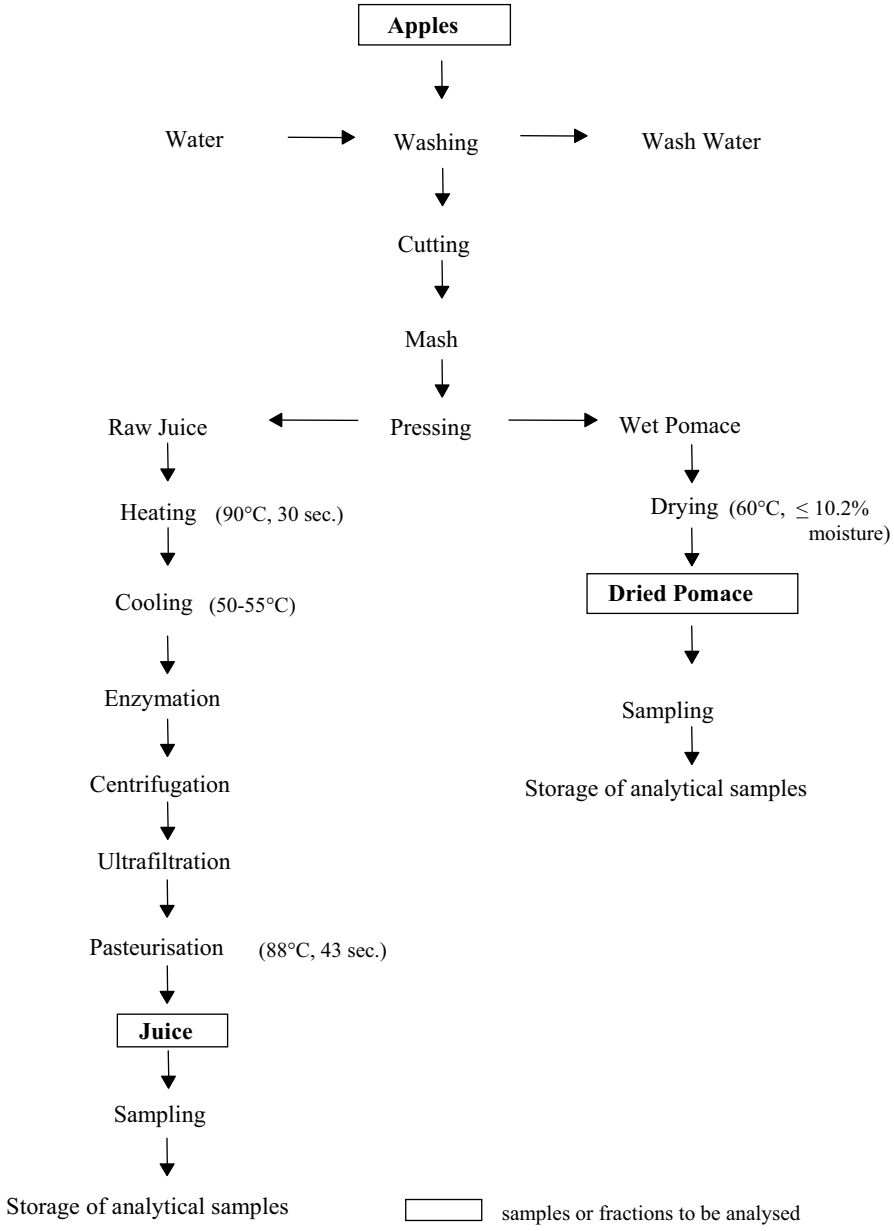


Figure 6. Flow Diagram for the preparation of apple juice and dried pomace.

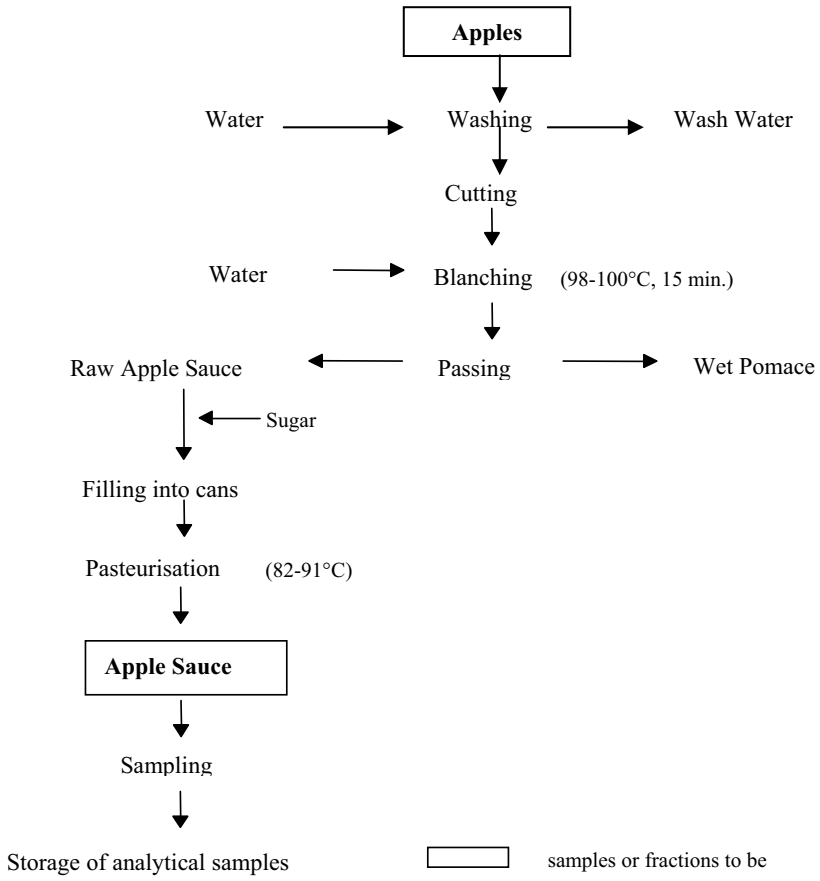


Figure 7. Flow Diagram for the preparation of apple sauce.

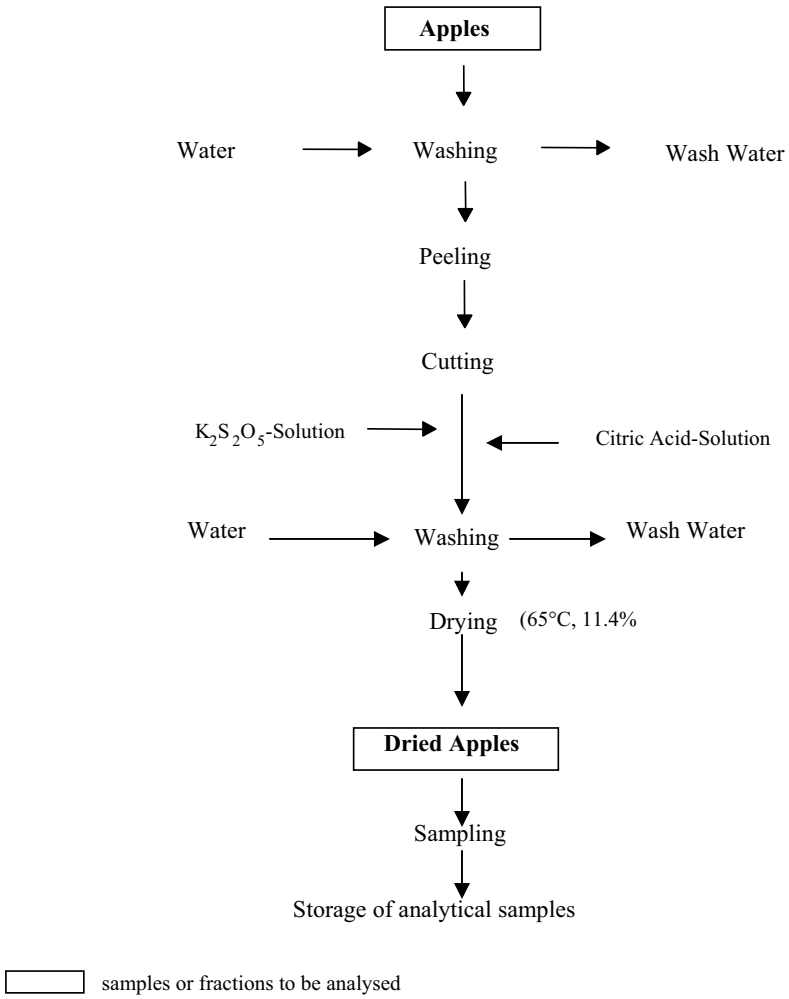


Figure 8. Flow Diagram for the preparation of dried apples.

Table 70. Results from processing studies on apple.

Country Year (Variety)	Application From	No.	kg ai/ha	kg ai/hL	PHI days	Commodity	Thiacloprid mg/kg	Processing factor	Author Date Report No.
Italy Laives 1995 (Granny Smith)	480 SC	2	0.14	0.096	14	Fruit (RAC) Fruit, washed Fruit, dried Juice Sauce Pomace, dried	0.1 0.08, 0.07 (0.08) 0.03, 0.02 (0.03) 0.02, 0.02 (0.02) 0.06, 0.05 (0.06) 0.42, 0.43 (0.43)	 0.8 0.3 0.2 0.6 4.3	Placke, F. J. 1997t RA-3063/95
Germany Monheim 1995 (Golden Delicious)	480 SC	2	0.14	0.023	14	Fruit (RAC) Fruit, washed Fruit, dried Juice Sauce Pomace, dried	0.07 0.06, 0.07 (0.07) 0.06, 0.04 (0.05) < 0.02, < 0.02 (< 0.02) 0.06, 0.06 (0.06) 0.63, 0.59 (0.61)	 1 0.7 0.29 0.86 8.7	Placke, F. J. 1997u RA-3062/95

RAC: raw agricultural commodity

Peaches

Three trials on peaches were performed in Italy and Spain in 1995 and 1996. The application rate was 0.144 kg ai/ha. The water rate was 1500 L/ha corresponding to a spray concentration of 0.02%. The two treatments were conducted during fruit development and fruit colouring at an interval of 14 days. The last application was carried out 14 days prior to the expected harvest (recommended waiting period). All applications were at the required rate. For processing peaches were taken on day 14 from the treated and the untreated plot.

The washing of peaches was done using domestic practice, i.e., washing in standing water and stoning (Figure). The preparation of peach preserves simulated industrial practice at a laboratory scale (Figure). For the preparation of preserves, the peaches were washed in standing water, peeled and stoned. The peeled and stoned peaches were filled into 1L preserving cans and a solution of sugar was added. Then the peach preserves were pasteurised at about 90°C. After pasteurisation, the peach preserve was minced in a mixer.

The residues of thiacloprid were determined according to method 00419. The recoveries ranged from 92 to 104% at fortification levels of 0.02 and 0.20 mg/kg for fruit and from 92 to 99% at a fortification level of 0.02 mg/kg for the peach preserve. The limit of quantitation (LOQ) was 0.02 mg/kg.

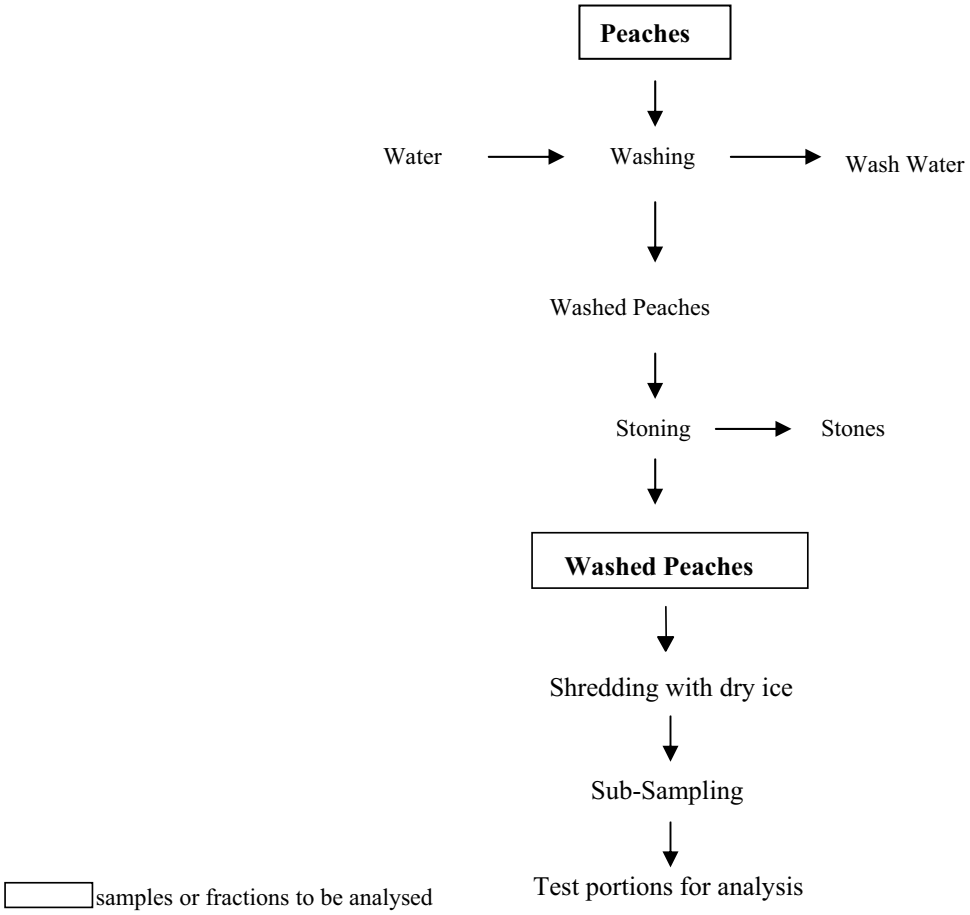


Figure 9. Flow Diagram for the preparation of washed peaches

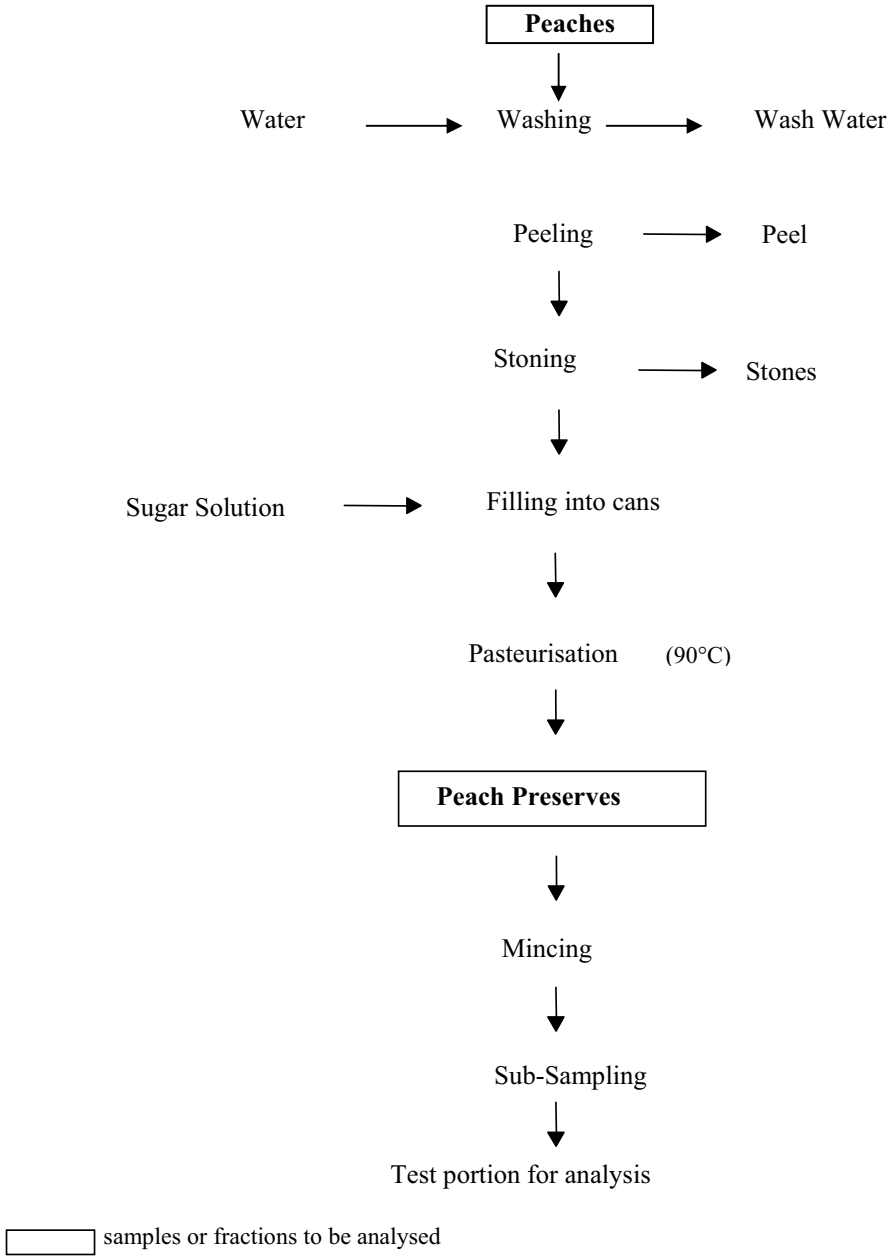


Figure 10. Flow Diagram for the preparation of peach preserve

Table 71. Results from processing studies on peach.

Country Year (Variety)	Application					Commodity	Thiacloprid mg/kg	Processing factor	Author Date Report No.
	From	No.	kg ai/ha	kg ai/hL	PHI days				
Italy Ravenna 1995 (Red Haven)	480 SC	2	0.14	0.096	14	Fruit w/o stone (RAC) Fruit, washed Preserve	0.03 0.02, 0.02 (0.02) < 0.02, < 0.02 (< 0.02)	 0.66 0.66	Placke, F. J. 1997v RA-3064/95

Country Year (Variety)	Application From	No.	kg ai/ha	kg ai/hL	PHI days	Commodity	Thiacloprid mg/kg	Processing factor	Author Date Report No.
Italy Ravenna 1996 (Red Haven)	480 SC	2	0.14	0.096	14	Fruit with stone (RAC) Fruit, washed Preserve	0.03 < 0.02, < 0.02 (< 0.02) < 0.02, < 0.02 (< 0.02)	 0.66 0.66	Placke, F. J. 1997w RA-3121/96
Spain La Fortesa 1996 (Baby Gold 9)	480 SC	2	0.14	0.096	14	Fruit with stone (RAC) Fruit, washed Preserve	0.09 0.06, 0.06 (0.06) < 0.02, < 0.02 (< 0.02)	 0.66 0.22	Placke, F. J. 1997w RA-3121/96

RAC: raw agricultural commodity

Cherries

For cherries one trial was conducted in Germany in 1999 (Schoening, R. and Sur, R., 2000c). Thiacloprid 480 SC was sprayed twice to sour cherry trees at a rate of 0.25 L/ha, corresponding to 0.12 kg ai/ha. The spray volume was 500 L per metre of plant height per hectare. The volume was adapted to the height of the leafy surface (500 L/(ha × m height) not exceeding 1500 L water/ha). The last treatment was performed 14 days prior to harvest. Samples were taken 14 days after the last application. The washing of cherries was done according to household practice. The preparation of cherry preserves simulated the industrial practice at laboratory scale. The processing procedures are described in Figure 11 and Figure 12. Analysis of thiacloprid was done according to method 00548.

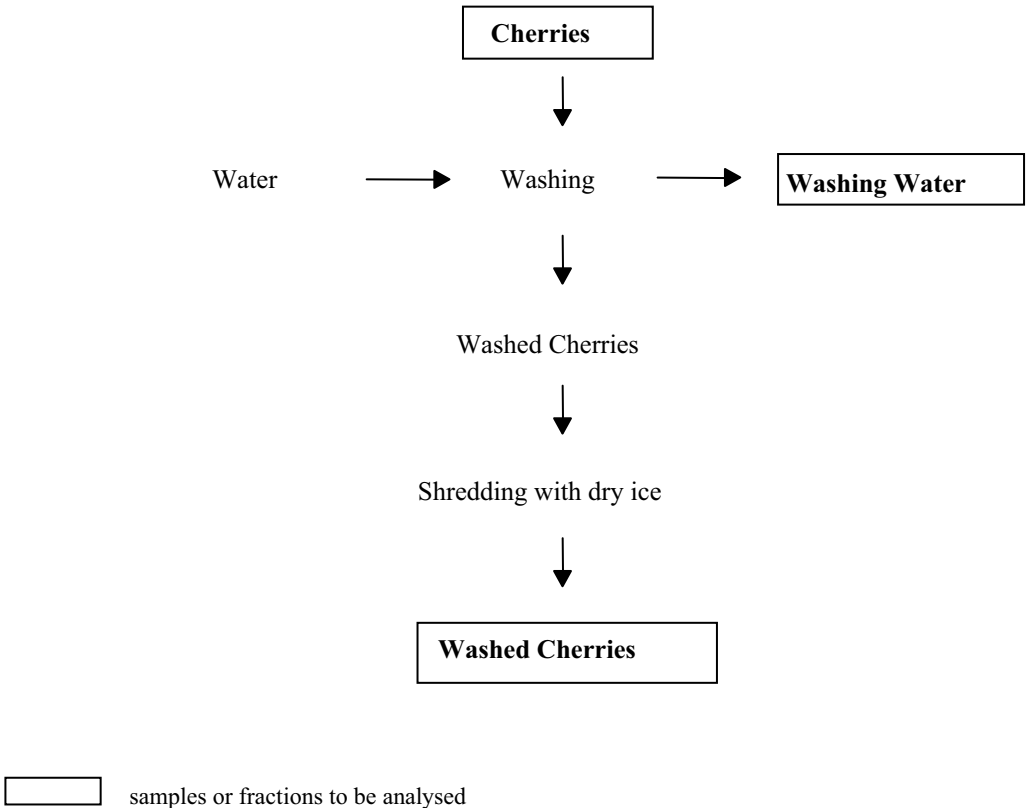


Figure 6. Flow diagram for the preparation of washed cherries.

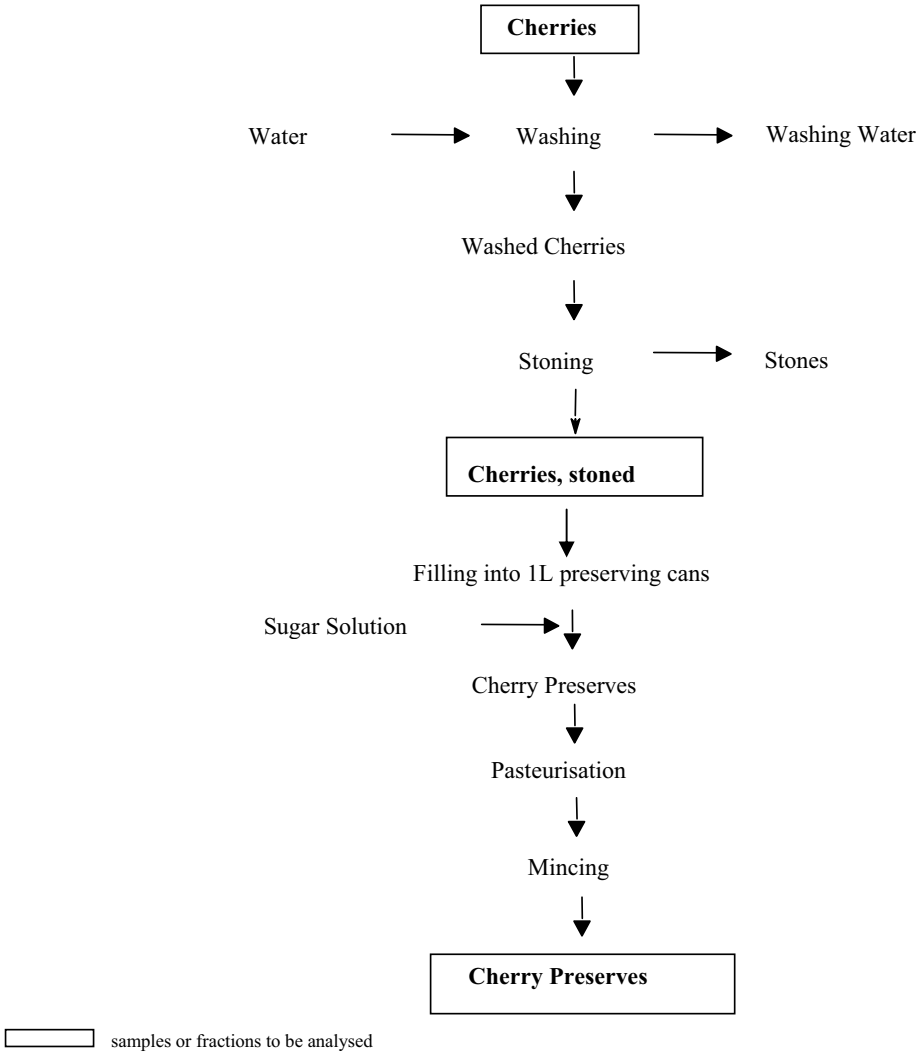


Figure 7. Flow diagram for the preparation of cherry preserve.

Residues of thiacloprid in the raw agricultural commodity (cherry fruit) and processed products harvested at a PHI of 14 days were below the limit of quantitation of 0.02 mg/kg. Therefore transfer factors could not be calculated.

Tomatoes

Two greenhouse trials on tomatoes were performed in Spain and Germany in 1996. The application rate was 0.216 kg ai/ha. The water rate was 1500 L/ha corresponding to a spray concentration of 0.025% and 0.030%, respectively. The treatments were conducted during fruit development and fruit colouring at an interval of 14 days between the first and the second application and an interval of 7 days between the second and third application. The last application was carried out 3 days prior to the expected harvest (recommended harvest interval). For processing, tomato fruits were taken on day 3 from the treated and the untreated plots.

Treated tomatoes were washed and processed into juice, paste and preserves. The washing and peeling was done using domestic practice (see Figure 13); whereas the production of juice, paste and preserve simulated commercial processing at a laboratory scale (see Figures 14 and 15).

For the preparation of juice the tomatoes were washed in standing water and then cut into small pieces. The tomato pieces were heated, with the addition of 100 mL water per 1 kg of tomatoes, to 100°C for about 8–10 minutes to prevent enzymatic reactions. After this blanching process the tomato pulp was passed through a strainer to separate juice and pomace. Sodium chloride (0.5–0.7% relative to the amount of juice) was added to the raw juice. The tomato juice was then filled into preserving cans and pasteurised for 2 minutes at about 90°C.

For the preparation of tomato paste the tomatoes were washed in standing water and then cut into small pieces. The tomato pieces were heated, with the addition of 100 mL water per 1 kg tomatoes, to approximately 100°C for about 8–10 minutes in order to prevent enzymatic reactions. After this blanching process the tomato pulp was passed through a strainer to separate juice and pomace. Subsequently the tomato juice was concentrated to 38–45% dry weight. After concentration the tomato paste was filled into cans. The tomato paste was then pasteurised for 5 minutes at about 90°C.

For the preparation of preserves the tomatoes were transferred into lukewarm water. After a few minutes the peel was removed. The peeled tomatoes were filled into 1L preserving cans with tomato juice added. The preserves were then pasteurised for 2–3 minutes at about 90 °C.

The residues of thiacloprid were determined according to method 00419. The recoveries ranged from 86 to 107% for fruit, from 89 to 108% for juice, from 93 to 97% for paste and from 80 to 98% for preserve at fortification levels of 0.02 and 0.20 mg/kg. The limit of quantification (LOQ) was 0.02 mg/kg.

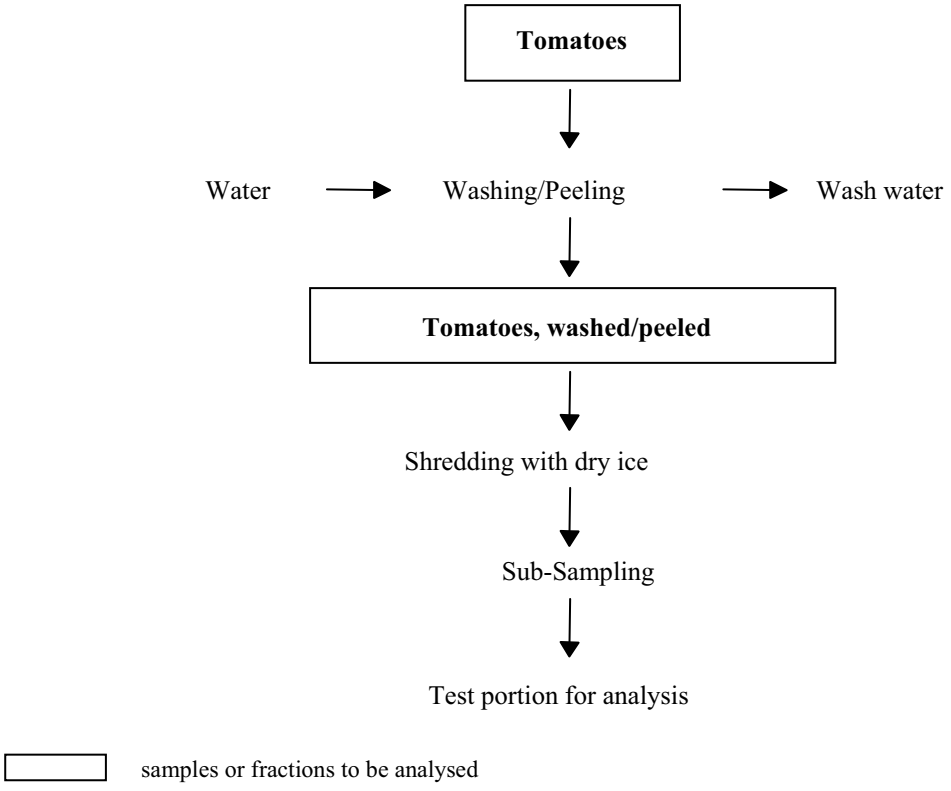


Figure 13. Flow diagram for the preparation of peeled tomatoes.

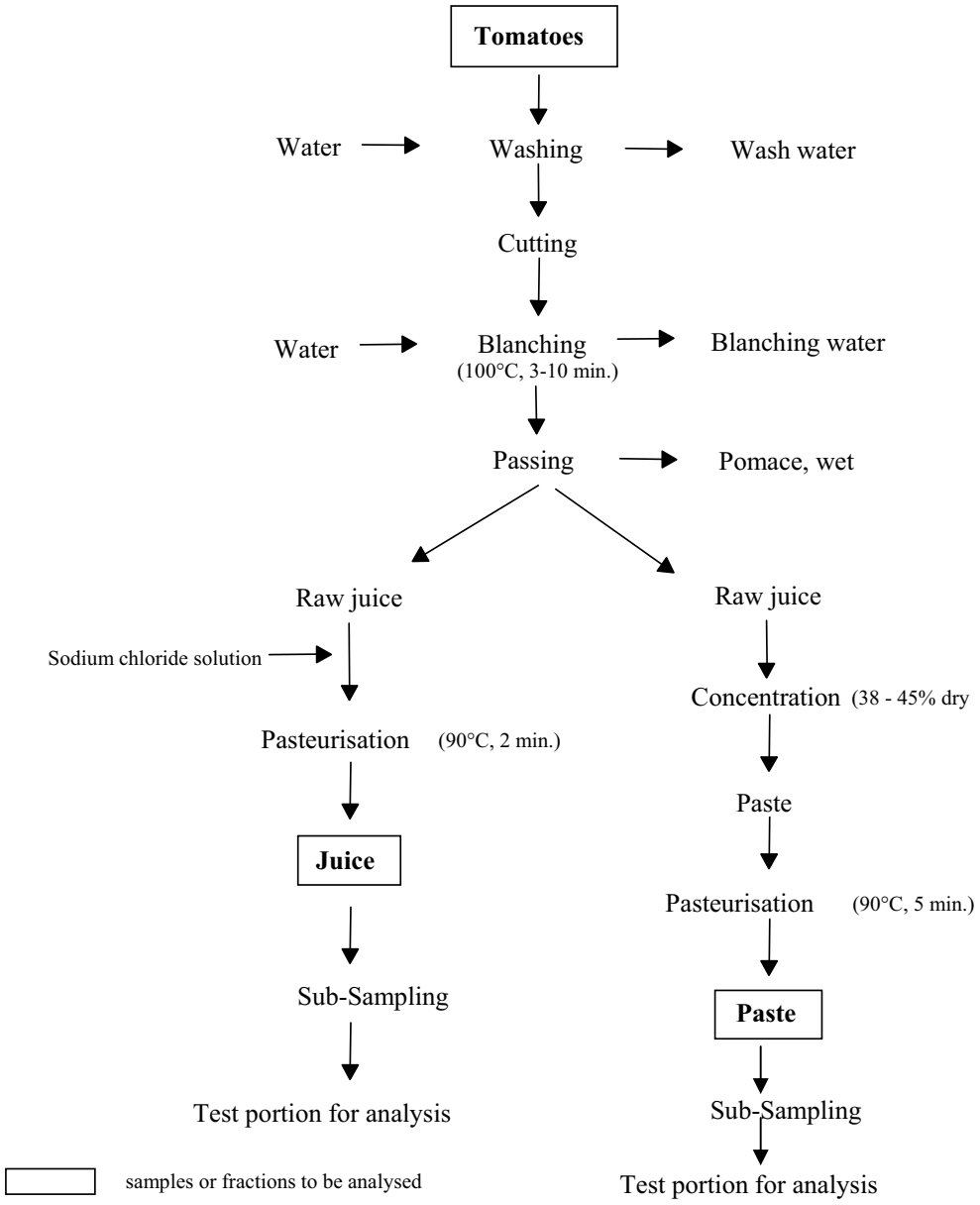


Figure 14. Flow diagram for the preparation of tomato juice and paste

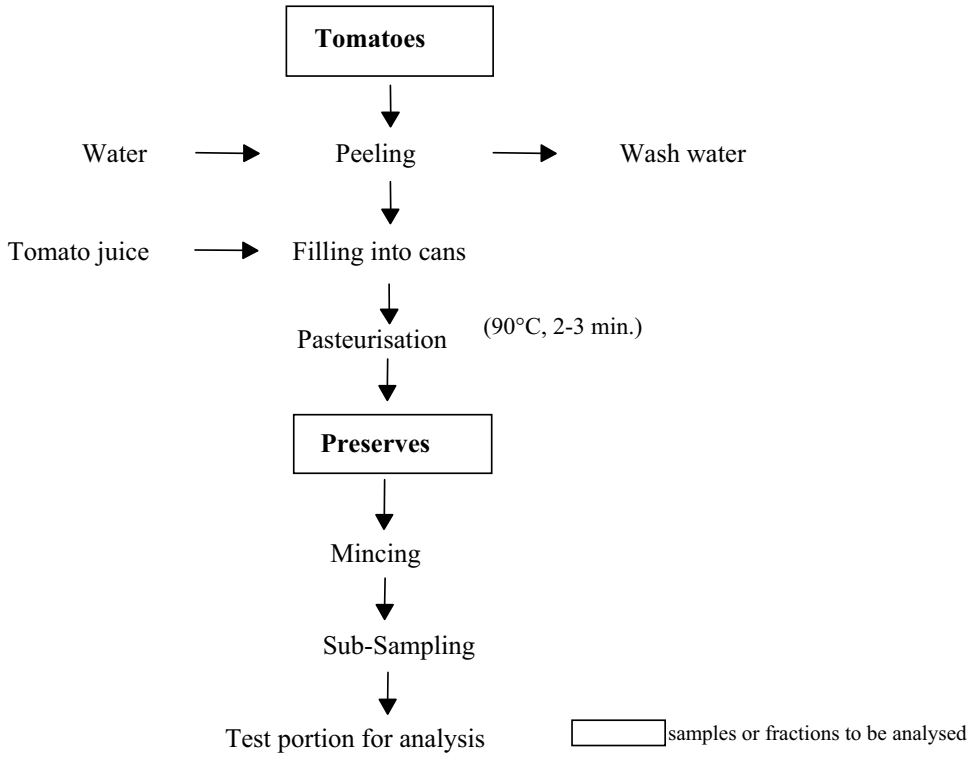


Figure 15. Flow diagram for the preparation of tomato preserves.

Table 72. Results from processing studies on tomato.

Country Year (Variety)	Application					Commodity	Thiacloprid mg/kg	Processing factor	Author Date Report No.
	From	No.	kg ai/ha	kg ai/hL	PHI days				
Spain Ruescas 1996 (Brillante)	480 SC	3	0.22	0.014	3	Fruit (RAC) Fruit, washed Fruit, peeled Paste Juice Preserve	0.24 0.13, 0.12 (0.125) 0.06, 0.05 (0.06) 0.48 0.09, 0.10 (0.10) 0.08, 0.07 (0.08)	 0.54 0.25 2 0.42 0.33	Placke, F. J. 1997x RA-3123/96
Germany Leichlingen 1996 (Ferrari)	480 SC	3	0.18	0.012	3	Fruit (RAC) Fruit, washed Fruit, peeled Paste Juice Preserve	0.07 0.06, 0.06 (0.06) 0.03, 0.03 (0.03) 0.22, 0.22 (0.22) 0.05, 0.05 (0.05) 0.05, 0.05 (0.05)	 0.86 0.43 3.1 0.71 0.71	Placke, F. J. 1997x RA-3123/96

RAC: raw agricultural commodity

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

A ruminant feeding study was reported. No study was available on poultry feeding.

A feeding study on cow was carried out at three dosing levels equivalent to 2.1 (0.07 mg/kg bw) (1×), 6.2 (0.213 mg/kg bw) (3×) and 20.6 ppm (0.655 mg/kg bw) (10×) thiacloprid in the diet

together with an untreated control group (Placke, F. J., 1998b). There were three cows in each of the treatment groups. After acclimatisation, thiacloprid was administered daily to the cows in gelatine capsules for 28 consecutive days. Milk samples were collected and composited for each cow. At the end of the 28-day dosing period, the cows were sacrificed, and kidney, liver, composite fat (omental and perirenal), and composite muscle (flank, leg, and loin) were removed from each cow. Blood was washed from the tissues. The tissues were immediately cut into small pieces, frozen with dry ice, and stored in a freezer below -18°C until processing. Tissues from each cow were kept separate for individual analysis. All milk samples were kept in a freezer below -18°C until analysis and were also individually processed and analysed.

Samples of tissues and milk were analysed for parent and total residues of thiacloprid. The total residues comprising the active substance and all metabolites containing the 6-chloropyridine moiety were determined according to method 00491 (Schoening, R., 1998b), while the active substance residues were determined according to method 00490 (Schoening, R., 1998a).

The results are shown in Table 73 to Table 76. On average of the three cows treated per dose group, liver contained the highest thiacloprid residue levels (0.10 mg/kg) followed by kidney (0.03 mg/kg), milk and muscle (0.02 mg/kg) and fat (0.01 mg/kg) at the 1× dose level. Maximum levels for tissues were 0.02 mg/kg for fat, 0.02 mg/kg for muscle, 0.04 mg/kg for kidney and 0.11 mg/kg for liver.

In the second dose group thiacloprid residue increased to average values of 0.04 mg/kg in milk and fat (highest value 0.04 mg/kg) 0.05 mg/kg in muscle (highest value 0.06 mg/kg), 0.1 mg/kg in kidney (highest value 0.11 mg/kg) and 0.29 mg/kg in liver (highest value 0.32 mg/kg). In the high dose group the findings were 0.17 mg/kg in milk, 0.12 mg/kg in fat (highest value 0.16 mg/kg), 0.16 mg/kg in muscle (highest value 0.18 mg/kg), 0.27 mg/kg in kidney (highest value 0.32 mg/kg) and 0.94 mg/kg in liver (highest value 1.1 mg/kg).

A linear relation between the dose levels and the residue concentrations was observed. In the milk, residues reached a plateau level within five days and no accumulation was observed.

Table 73. Residues in milk 1× dose group (2.1 ppm, 0.07 mg/kg bw).

Days	Cow 4		Cow 5		Cow 6		Average	
	Thiacloprid mg/kg	Total residue[1] mg/kg	Thiacloprid mg/kg	Total residue[1] mg/kg	Thiacloprid mg/kg	Total residue[1] mg/kg	Thiacloprid mg/kg	Total residue[1] mg/kg
1	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
2	0.01	0.01	0.011	0.014	0.016	< 0.01	0.013	0.011
5	0.014	0.02	0.014	0.02	0.021	0.026	0.016	0.022
8	0.014	0.017	0.014	0.017	0.02	0.025	0.016	0.020
11	0.018	0.02	0.020	0.014	0.03	0.025	0.023	0.020
14	0.013	0.017	0.017	0.022	0.022	0.026	0.017	0.022
17	0.017	0.02	0.015	0.02	0.025	0.027	0.019	0.022
20	0.021	0.025	0.013	0.019	0.021	0.022	0.018	0.022
25	0.011	0.016	0.012	0.017	0.016	0.018	0.013	0.017
28	0.012	< 0.01	< 0.01	0.014	0.016	0.015	0.013	0.013

1 determined as 6-CAN

Table 74. Residues in milk 3× dose group (6.2 ppm, 0.213 mg/kg bw).

Days	Cow 6		Cow 8		Cow 9		Average	
	Thiacloprid mg/kg	Total residue[1] mg/kg	Thiacloprid mg/kg	Total residue[1] mg/kg	Thiacloprid mg/kg	Total residue[1] mg/kg	Thiacloprid mg/kg	Total residue[1] mg/kg
1	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
2	0.031	0.05	0.035	0.052	0.04	0.055	0.035	0.052
5	0.035	0.061	0.034	0.064	0.057	0.084	0.042	0.070
8	0.039	0.066	0.046	0.064	0.054	0.08	0.046	0.070
11	0.039	0.055	0.038	0.043	0.050	0.066	0.042	0.055
14	0.032	0.043	0.04	0.047	0.053	0.059	0.042	0.050

Days	Cow 6		Cow 8		Cow 9		Average	
	Thiacloprid mg/kg	Total residue[1] mg/kg	Thiacloprid mg/kg	Total residue[1] mg/kg	Thiacloprid mg/kg	Total residue[1] mg/kg	Thiacloprid mg/kg	Total residue[1] mg/kg
20	0.043	0.059	0.059	0.068	0.059	0.061	0.054	0.063
28	0.033	0.046	0.042	0.051	0.043	0.057	0.039	0.051

1 determined as 6-CNA

Table 75. Residues in milk 10× dose group (20.6 ppm, 0.655 mg/kg bw).

Days	Cow 10		Cow 11		Cow 12		Average	
	Thiacloprid mg/kg	Total residue[1] mg/kg	Thiacloprid mg/kg	Total residue[1] mg/kg	Thiacloprid mg/kg	Total residue[1] mg/kg	Thiacloprid mg/kg	Total residue[1] mg/kg
1	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
2	0.119	0.147	0.097	0.119	0.080	0.104	0.099	0.123
5	0.147	0.185	0.142	0.192	0.123	0.155	0.137	0.177
8	0.143	0.192	0.142	0.182	0.126	0.162	0.137	0.179
11	0.122	0.148	0.139	0.189	0.124	0.152	0.128	0.163
14	0.121	0.153	0.152	0.208	0.146	0.184	0.140	0.182
17	0.122	0.201	0.154	0.212	0.150	0.234	0.142	0.216
20	0.127	0.180	0.171	0.230	0.107	0.122	0.135	0.177
25	0.1	0.144	0.094	0.151	0.120	0.170	0.105	0.155
28	0.096	0.136	0.098	0.170	0.063	0.094	0.086	0.133

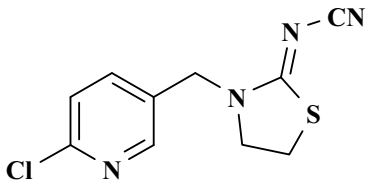
1 determined as 6-CNA

Table 76. Residue concentrations in the edible tissues.

Animal	Liver		Kidney		Muscle		Fat	
	Thiacloprid mg/kg	Total residue[1] mg/kg	Thiacloprid mg/kg	Total residue[1] mg/kg	Thiacloprid mg/kg	Total residue[1] mg/kg	Thiacloprid mg/kg	Total residue[1] mg/kg
Control, Cow 1	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Control, Cow 3	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
1×, Cow 4	0.09, 0.09 (0.09)	0.09, 0.09 (0.09)	0.03, 0.03 (0.03)	0.05, 0.04 (0.05)	0.02, 0.02 (0.02)	0.02, 0.02 (0.02)	0.01, 0.01 (0.01)	0.02, 0.02 (0.02)
1×, Cow 5	0.09, 0.09 (0.09)	0.08, 0.08 (0.08)	0.03, 0.03 (0.03)	0.05, 0.04 (0.05)	0.02, 0.01 (0.02)	0.02, 0.02 (0.02)	0.01, 0.01 (0.01)	0.01, 0.01 (0.01)
1×, Cow 6	0.11, 0.11 (0.11)	0.12, 0.11 (0.12)	0.04, 0.04 (0.04)	0.06, 0.06 (0.06)	0.02, 0.02 (0.02)	0.02, 0.02 (0.02)	0.02, 0.02 (0.02)	0.02, 0.02 (0.02)
1×, mean	0.10	0.10	0.03	0.05	0.02	0.02	0.01	0.02
3×, Cow 7	0.28, 0.26 (0.27)	0.28, 0.27 (0.28)	0.09, 0.09 (0.09)	0.15, 0.14 (0.15)	0.05, 0.05 (0.05)	0.05, 0.05 (0.05)	0.03, 0.03 (0.03)	0.03, 0.03 (0.03)
3×, Cow 8	0.31, 0.32 (0.32)	0.27, 0.29 (0.28)	0.09, 0.09 (0.09)	0.17, 0.20 (0.19)	0.05, 0.05 (0.05)	0.05, 0.05 (0.05)	0.04, 0.04 (0.04)	0.04, 0.04 (0.04)
3×, Cow 9	0.29, 0.27 (0.28)	0.28, 0.30 (0.29)	0.11, 0.11 (0.11)	0.16, 0.18 (0.17)	0.06, 0.06 (0.06)	0.06, 0.06 (0.06)	0.04, 0.04 (0.04)	0.04, 0.04 (0.04)
3×, mean	0.29	0.28	0.10	0.17	0.05	0.05	0.04	0.04
10×, Cow 10	0.91, 0.91 (0.91)	0.87, 0.85 (0.86)	0.23, 0.24 (0.24)	0.51, 0.45 (0.48)	0.15, 0.14 (0.15)	0.15, 0.15 (0.15)	0.12, 0.12 (0.12)	0.14, 0.15 (0.15)
10×, Cow 11	1.1, 1.1 (1.1)	1.1, 1.2 (1.2)	0.32, 0.31 (0.32)	0.61, 0.60 (0.61)	0.18, 0.18 (0.18)	0.19, 0.18 (0.19)	0.12, 0.13 (0.13)	0.16, 0.15 (0.16)
10×, Cow 12	0.81, 0.79 (0.80)	0.81, 0.84 (0.83)	0.26, 0.24 (0.25)	0.48, 0.48 (0.48)	0.15, 0.15 (0.15)	0.15, 0.14 (0.15)	0.10, 0.09 (0.10)	0.12, 0.13 (0.13)
10×, mean	0.94	0.96	0.27	0.52	0.16	0.17	0.12	0.15

APPRAISAL

N-{3-[(6-Chloro-3-pyridinyl)methyl]-1,3-thiazolan-2-yliden}cyanamide



Residues and analytical aspects of thiacloprid were considered for the first time by the present Meeting.

Thiacloprid is a non-systemic insecticide with registered uses in many countries. Thiacloprid causes disruption of the insect nervous system by acting as an inhibitor at nicotinic acetylcholine receptors.

The following abbreviations are used for the metabolites discussed below:

thiacloprid-amide	{3-[(6-chloro-3-pyridinyl)methyl]-2-thiazolidinylidene}urea (M02)
6-CNA	6-chloro-3-pyridinecarboxylic acid (M03)
thiacloprid-sulfoxide	N-[(6-chloro-3-pyridinyl)methyl]-N'-cyano-N-[2-(methylsulfinyl)-ethyl]urea (M08)
M09	N-{[6-(methylthio)-3-pyridinyl]-carbonyl}glycine
M12	Glucuronic acid conjugate of {3-[(6-chloro-3-pyridinyl)methyl]-4(or 5)-hydroxy-2-thiazolidinylidene}=
thiacloprid sulfonic acid	Sodium 2-[[[(aminocarbonyl)amino]-carbonyl][(6-chloro-3-pyridinyl)-methyl]amino]ethanesulfonate (M30)

Animal metabolism

The Meeting received results of animal metabolism studies in lactating goats and laying hens.

Goats

One lactating goat was dosed with [methylene-^{14}C]-thiacloprid at a rate of 10 mg/kg body weight for three consecutive days. Approximately 53.7% of the total radioactivity administered was excreted until sacrifice. A portion of about 48.3% was eliminated with urine and 4.5% with faeces. Due to the short period between the last dose and sacrifice, 40% of the dose was not recovered in the excreta. A low amount (0.93%) was secreted with the milk. At sacrifice 6 hours after the last dose, the total radioactive residues (TRR) in the edible tissues and organs accounted for 5.6% of the administered radioactivity. The major portion and the highest equivalent concentration were observed in the kidney and the liver.

The metabolism of thiacloprid in goats is comparable to the metabolism in rats.

The unchanged parent compound was found in all goat tissues and ranged from 28% of the TRR (equiv. to 7 mg/kg) in kidney, 61% (equiv. to 1.5 mg/kg) in milk, 83% (equiv. to 14.5 mg/kg) in liver, 90% (equiv. to 1.6 mg/kg) in fat to 92% (equiv. to 3.5 mg/kg) in muscle.

Further main metabolites were identified in kidney. Thiacloprid-sulfoxide was found at levels of 12.3% of the TRR (equiv. to 3.1 mg/kg) and M12 at 10% of the TRR (equiv. to 2.5 mg/kg). Except for thiacloprid-sulfoxide in milk (8.7% of the TRR) no other relevant metabolites in concentrations above 8% of the TRR were identified.

Hens

A group of six laying hens were fed with [methylene-^{14}C]-thiacloprid for three consecutive days at a dose rate of 10 mg/kg body weight each. Until sacrifice the excretion amounted on average to 75.4% of the total radioactivity administered. About 29.4% and 29.6% of the radioactivity eliminated during the test period was excreted within 24 hours of the first and the second doses, respectively. Another 16.4% was excreted between the final dose and sacrifice. On average, only 0.06% (equivalent to 0.4 mg/kg) of the total dose was determined in the eggs. Residue levels in liver, kidney, muscle (leg), muscle (breast) and skin (without fat) were 3.1, 2.4, 0.15, 0.13 and 0.30 mg/kg TRR, respectively.

The metabolism of thiacloprid in laying hens is comparable to the metabolism in rats.

The unchanged parent compound was found in all hen tissues and ranged from 17% of the TRR (equiv. to 0.54 mg/kg) in liver, 19% (equiv. to 0.03 mg/kg) in muscle, 48% (equiv. to 0.06 mg/kg) in eggs to 72% (equiv. to 0.08 mg/kg) in fat.

Further main metabolites were identified in muscle only. M9 was found at levels of 10.9% of the TRR (equiv. to 0.016 mg/kg). Except for thiacloprid-sulfoxide in fat (8.9% of the TRR) no other relevant metabolites in concentrations above 8% of the TRR were identified.

Thiacloprid is only moderately metabolized by goats and hens with 5.6% (goats) and 0.7% (hens) of the applied dose remaining in tissues after three days. The proposed metabolic pathway was via hydroxylation and the formation of glucoronide and cysteine conjugates, resulting in a large variety of metabolites in small amounts.

Plant metabolism

The Meeting received plant metabolism studies for thiacloprid on apples, tomatoes, cotton and wheat. In all studies [methylene-^{14}C]-thiacloprid was applied as a spray.

All plant metabolism studies demonstrated that the metabolic pathway of thiacloprid is comparable in all crops investigated. The main metabolic reactions are:

the hydroxylation of the parent compound at the thiazolidine ring

the oxidative cleavage at the methylene bridge leading to the partially and fully oxidised products 6-chloropicolyl alcohol (M36), 6-chloronicotinic acid (M03)

conjugation of these two aglycones with sugars, phosphate/sulfate and endogenous plant components.

Uptake of soil metabolites followed by further metabolisation also took place. However, these metabolic reactions occurred only to a limited extent, the majority of residue remained on the surface of the fruits as unchanged parent compound exceeding 90% of the total residue. The major metabolites identified were the monohydroxylated derivative of thiacloprid (M01; apples) and the oxidation product 6-chloronicotinic acid (M03; cotton seed, wheat) as well as various conjugates thereof or of its precursor 6-chloropicolyl alcohol (M36; cotton, tomatoes, wheat).

In translocation experiments with tomatoes it was shown that less than 0.1% of the radioactivity in the soil was transported into the fruits after uptake via the roots.

In cotton seeds a different metabolic pattern with 6-chloronicotinic acid (M03), being the main residue (46%), was observed, which might be the result of partitioning and selective transport effects. In treated cotton leaves the metabolism followed the same steps found in the other plants investigated:

hydroxylation of the parent compound at the thiazolidine ring;

cleavage at the methylene bridge leading to the partially and fully oxidised products 6-chloropicolyl alcohol (M36) and 6-chloronicotinic acid (M03);

conjugation of these two aglycones with sugars, phosphate/sulfate and endogenous plant components.

In each crop tested except cotton seeds, unchanged thiacloprid was found to be relevant residue with amount > 80% of the TRR.

Environmental fate

The Meeting received information on the environmental fate of thiacloprid in soil, including aerobic soil metabolism, field dissipation and crop rotational studies.

The soil photolysis study conducted with [methylene-^{14}C]-thiacloprid gave evidence that no accelerated degradation occurs under irradiation. Thiacloprid-amide could be identified as the main degradation byproduct. The calculated environmental half-life for thiacloprid was 74 days during midday and midsummer at 40° of latitude. No additional metabolites were identified in the samples.

In confined rotational crops studies, soil was treated with [pyridinyl-^{14}C-methyl]-thiacloprid. Turnips, lettuce and wheat were sown into the treated soil at intervals of 30, 170 and 354 days after treatment and were grown to maturity and harvested for analysis. In all matrices radioactivity above 0.01 mg/kg was found. After 354 days the residues measured ranged from 0.005 mg/kg in turnip bulbs up to 0.322 mg/kg in wheat straw. Thiacloprid-amide and thiacloprid sulfonic acid could be identified as relevant metabolites accounting for 15 – 35% of the TRR each. No parent thiacloprid was found.

The Meeting concluded that thiacloprid residues from the use of thiacloprid do not occur in concentrations above 0.01 mg/kg.

Methods of Analysis

The Meeting received descriptions and validation data for analytical methods for thiacloprid in plant and animal matrices. The method for enforcement purposes is based on extraction with acetone/water (3:1; v:v) and a subsequent clean-up by column chromatography on Florisil and elution with acetonitrile. The residues of thiacloprid parent compound are quantified by reversed phase HPLC with UV detection at 242 nm. Validation data for apples, tomatoes, cucumbers, peaches, citrus fruits, cotton seed, potatoes and tobacco was presented. In general a LOQ of 0.02 – 0.05 mg/kg was achieved, the recoveries were in the range of 72% to 105%.

Animal matrices were extracted with a mixture of acetonitrile/water or methanol. For milk samples, partitioning of the extracts against n-hexane was performed to remove fat. The aqueous remainder is partitioned against cyclohexane/ethyl acetate using a ChemElut column. Further clean-up is performed by column chromatography on Florisil and elution with acetonitrile. The residues are quantified by reversed phase HPLC with UV-detection at 242 nm. The method was validated by conducting recovery tests with muscle, milk and eggs. An LOQ of 0.01 mg/kg in milk and 0.02 mg/kg in muscle and egg was achieved, the recoveries were in the range of 75% to 104%

In addition the meeting received information on various specialized methods, mainly based on HPLC-MS/MS techniques with modification in the extraction and clean-up procedure. These methods for plant and animal matrices detect thiacloprid and possible metabolites with LOQs ranging from 0.01 mg/kg to 0.5 mg/kg (rice), depending on the matrix. In general an LOQ of 0.02 mg/kg could be achieved for all matrices except rice.

For thiacloprid, additional methods for the determination of all moieties containing 6-CNA were available. Thiacloprid and its metabolites were extracted from plant matrices with an acidic methanol / water mixture. After the clean-up thiacloprid and all metabolites containing the 6-chloropicolyl moiety were oxidized with alkaline potassium permanganate solution to yield 6-chloronicotinic acid. This was followed by acidification and reduction of the excess permanganate and the developed manganese dioxide with sodium bisulfite. The 6-CNA was converted to the corresponding trimethylsilyl ester with MSTFA (N-methyl-trimethylsilyltrifluoroacetamide) prior to quantitation by gas chromatography with mass selective detection in the single-ion monitoring mode

(GC-MS). Validation data for pome fruits, tomatoes, cotton seed, rape seed, sunflower seed, milk, muscle, liver, kidney and fat was presented. In general a LOQ of 0.05 mg/kg for plant matrices and 0.01 – 0.02 mg/kg for animal matrices was achieved, the recoveries were in the range of 66% to 102%.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the stability of thiacloprid in apples, currants, tomatoes, melons, peas, potatoes, cotton seed, rape seed, wheat and tobacco. All samples were fortified at 0.2 mg/kg (except tobacco with 2 mg/kg) and stored at -20°C for between 540 and 730 days. In all matrices the remaining thiacloprid residues levels were above 80% of the initial fortification concentrations.

No stability study was submitted to the Meeting on animal matrices.

Residue definition

The results of the radiolabeled thiacloprid plant metabolism studies on apples, tomatoes, cotton and wheat indicate that thiacloprid metabolizes or degrades slowly under typical foliar application conditions. Greater than 80% of the TRR is recovered as thiacloprid and no significant metabolites or degradates were found in crops treated directly.

In rotational crop studies significant total radioactive residues were found in lettuce and wheat. Most of the residue consisted of the metabolites thiacloprid amide (M02) and thiacloprid sulfonic acid (M30). Unchanged thiacloprid was not identified. These metabolites are not considered toxicologically significant and need not be considered for the residue definition.

In ruminants, orally administered radiolabeled thiacloprid undergoes limited metabolism to glucoronide and cysteine conjugates after hydroxylation. The major component in all matrices was unchanged thiacloprid (> 80% TRR in liver, fat and muscle, 61% TRR in milk and 28% TRR in kidney). Further metabolites were identified in kidney at levels below 12% of the TRR. In poultry orally administered (dosed at 10 mg/kg body weight) thiacloprid was moderately metabolised. In all matrices thiacloprid was identified as the major component (17% TRR liver, 19% TRR muscle, 48% TRR in eggs, 72% TRR in fat). Further metabolite found in muscle, was only M9 which accounted for 10.9% of the TRR.

The log P_{ow} of thiacloprid is 1.26. As no accumulation in fat was observed in animal metabolism studies the Meeting concluded that thiacloprid is not fat-soluble.

The analytical methods determine thiacloprid, possible metabolites or the total residue determined as 6-CNA.

Based on the results of the metabolism studies the Meeting concluded that the residue definition for enforcement and dietary intake calculations in plant and animal commodities is thiacloprid. The residue is not fat-soluble.

Results of supervised trials on crops

Citrus fruit

The Meeting received information on supervised residue trials on lemons and oranges from Brazil, New Zealand and South Africa.

In Brazil thiacloprid can be applied to citrus at 0.0048 kg ai/hL with a PHI of 21 days. In two Brazilian trials on lemons three applications were made at a rate of 0.0048 kg ai/hL and 0.0096 kg ai/hL with a PHI of 21 days. No whole fruit residue data was submitted.

One trial on lemons was submitted from New Zealand where a single spray application of 0.0096 kg ai/hL was made. Residues on whole lemon fruit were found to decline from 0.19 mg/kg 1 day after treatment to 0.07 mg/kg by day 14. A GAP for New Zealand was not submitted.

In South Africa thiacloprid can be applied to citrus at a rate of 0.0067 kg ai/hL. Corresponding number of applications or PHI was not stated. In two residue trials on oranges one treatment was conducted with spray concentrations of 0.014 kg ai/hL to 0.029 kg ai/hL with the PHI ranging from 44 to 190 days. No residues above the LOQ of 0.02 mg/kg were found in all samples.

The Meeting concluded that there was insufficient data available to support a recommendation for citrus fruit.

Pome fruit

The Meeting received information on supervised residue trials on apples from Australia, Belgium, France, Germany, Italy, Japan, the Netherlands, South Africa, Spain, United Kingdom and the USA.

Thiacloprid is registered for use on apples or pome fruits in some European countries as a pre-harvest foliar spray treatment. Residue trials were carried out in Belgium, France, Germany, Italy, the Netherlands, Spain and the United Kingdom. The GAPs from Austria, Belgium, Cyprus, Czech Republic, Greece, Hungary, Italy, the Netherlands, Russia and the United Kingdom consisted of two to three spray applications at 0.012 – 0.014 kg ai/hL with a PHI of 14 days. The residues matching this GAP in the whole fruits were: 0.04, 0.05, 0.1 (2), 0.11, 0.13, 0.14, 0.16, 0.21 and 0.36 mg/kg.

In Croatia, Germany, Latvia, Lithuania, Portugal, Romania, Slovakia, Slovenia and Spain the GAP consists of two to three spray application at a rate of 0.0096 kg ai/hL with a PHI of 14 days. The residues matching this GAP in the whole fruits were: 0.02, 0.04, 0.07, 0.08, 0.1, 0.11 and 0.12 mg/kg.

GAP in USA for apples consists of up to six applications at 0.01 kg ai/hL and a PHI of 30 days. The residues from 14 supervised trials in the USA, matching the US GAP (± 30%), were: 0.02, 0.04, 0.05, 0.06 (3), 0.07 (2), 0.09 (2), 0.1, 0.11, 0.14 and 0.28 mg/kg.

GAP in South Africa for apples consists of up to four applications at 0.0072 kg ai/hL and a PHI of 14 days. Of the four supervised trials provided from South Africa none matching South African GAP.

GAP in Japan for apples consists of up to three applications at 0.015 kg ai/hL and a PHI of seven days. The residues from two supervised trials in Japan matching GAP were 0.11 and 0.30 mg/kg.

GAP in Australia for apples consists of up to three applications at 0.018 kg ai/hL and a PHI of 14 days. The residue from one trial in Australia, matching GAP (± 30%), was 0.37 mg/kg.

The Meeting decided to pool the data from Australia, Europe, Japan and the USA. The combined results (n = 34) for apples were: 0.02 (2), 0.04 (3), 0.05 (2), 0.06 (3), 0.07 (3), 0.08, 0.09 (2), 0.1 (4), 0.11 (4), 0.12, 0.13, 0.14, 0.14, 0.16, 0.21, 0.28, 0.30, 0.36 and 0.37 mg/kg.

Field trials involving thiacloprid on pears were provided from Australia, South Africa and USA.

GAP in USA for pears consists of up to six applications at 0.01 kg ai/hL and a PHI of 30 days. The residues from 14 supervised trials in the USA, matching GAP (± 30%), in ranked order were :0.05, 0.06, 0.1, 0.14, 0.14, 0.23, 0.24 and 0.27 mg/kg.

GAP in South Africa for pears consists of up to four applications at 0.0072 kg ai/hL and a PHI of 14 days. Of the four supervised trials provided from South Africa none matched GAP.

GAP in Japan for pears consists of up to three applications at 0.015 kg ai/hL and a PHI of seven days. The residues from two supervised trials in Japan matching GAP were: 0.61 and 0.87 mg/kg.

GAP in Australia for pears is up to three applications with 0.018 kg ai/hL each and a PHI of 14 days. The residues from two supervised trials in Australia, matching the GAP, were 0.37 and 0.38 mg/kg.

The Mann-Whitney-U test indicated that the medians of the residues from the Japanese and the combined Australian and US data set for pears were not similar. The Meeting decided to pool only the data from Australia and the USA. The combined results (n = 10) for pears were 0.05, 0.06, 0.1, 0.14, 0.14, 0.23, 0.24, 0.27, 0.37 and 0.38 mg/kg.

The Meeting decided to make a recommendation for the crop group of pome fruits based on the combined data for apples and pears.

For apples and pears the combined results were 0.02, 0.02, 0.04(3), 0.05(3), 0.06(4), 0.07(3), 0.08, 0.09, 0.09, 0.1(5), 0.11(4), 0.12, 0.13, 0.14(4), 0.16, 0.21, 0.23, 0.24, 0.27, 0.28, 0.30, 0.36, 0.37, 0.37 and 0.38 mg/kg.

Based on residue data for apples and pears the Meeting decided to recommend a maximum residue level of 0.7 mg/kg, a STMR of 0.11 mg/kg and a HR of 0.38 mg/kg for pome fruits.

Stone fruits

The Meeting received information on supervised residue trials on Japanese apricots from Japan.

GAP in Japan for Japanese apricots consists of up to two applications at 0.0075 kg ai/hL with a PHI of seven days. The residue trials from Japan were conducted with an application rate of 0.015 kg ai/hL, which did not correspond to the submitted GAP.

Field trials on peaches were available from France, Italy, Japan and Spain.

GAP in Cyprus, Greece, Italy and Slovenia for peaches/nectarines consists of up to two applications at 0.0096 to 0.012 kg ai/hL and a PHI of 14 days. The residues in whole fruits from nine supervised trials in Europe matching the GAP were: 0.03(3), 0.06, 0.08, 0.09, 0.13, 0.13 and 0.19 mg/kg.

GAP in Japan for peaches is up to three applications at 0.015 kg ai/hL and a PHI of 7 days. The residues from two supervised trials from Japan matching the GAP ± 30% were 0.27 and 0.40 mg/kg.

The Mann-Whitney-U test for the data from Japan and the residue data from Europe suggested a similar median for both distributions. The combined residue data was 0.03(3), 0.06, 0.08, 0.09, 0.13, 0.13, 0.19, 0.27 and 0.40 mg/kg.

Field trials on cherries were provided from Belgium, France, Germany, Italy, Japan, Spain and USA.

GAP in Croatia, Cyprus, Czech Republic, the Netherlands, Romania, Slovenia and the United Kingdom for cherries is up to two applications with 0.0096 to 0.015 kg ai/hL each and a PHI of 14 days. The residues in whole fruits from 12 supervised trials in Europe matching the GAP were for sour cherries < 0.02, 0.02, 0.03, 0.04 mg/kg and for sweet cherries 0.02, 0.06, 0.06, 0.07, 0.08, 0.1, 0.11 and 0.15 mg/kg.

GAP in Japan for cherries is up to two applications with 0.015 kg ai/hL each and a PHI of one day. The residues from two supervised trials in Japan matching the GAP were 1.4 and 2.4 mg/kg. The Mann-Whitney-U test indicated that the medians of residues, resulting from applications according to the Japanese and European GAP for cherries, were not similar. Therefore only the data from the European trials were considered for further evaluation.

The Mann-Whitney-U test gave evidence that a similar distribution for sweet and sour cherries were not similar. Therefore only the data for sweet cherries were used for further evaluation.

In Northern America thiacloprid is not registered for use in cherries. Therefore the available supervised residue trials from USA were not considered.

Field trials on plums were provided from France, Germany, Spain and USA.

GAP in the Czech Republic, the Netherlands and Romania for plums is up to two applications at 0.0096 to 0.012 kg ai/hL and a PHI of 14 days. The residues in whole fruits from 14 supervised trials in Europe matching the GAP were: < 0.02(6), 0.02(5), 0.03, 0.03 and 0.05 mg/kg.

GAP in Japan for plums is up to three applications with 0.0075 kg ai/hL each and a PHI of seven days. The residue trials from Japan were conducted with an application rate of 0.015 kg ai/hL, which does not correspond to the submitted GAP.

In Northern America thiacloprid is not registered for use in plums. Therefore the available supervised residue trials from USA are not considered for evaluation.

The Meeting decided to make a recommendation for the stone fruits crop group, based on the combined data for peaches and sweet cherries.

For peaches and sweet cherries the combined results were 0.02, 0.03(3), 0.06(3), 0.07, 0.08, 0.08, 0.09, 0.1, 0.11, 0.13, 0.13, 0.15, 0.19, 0.27 and 0.40 mg/kg.

Based on residue data for peaches and sweet cherries the Meeting recommends a maximum residue level of 0.5 mg/kg, a STMR of 0.08 mg/kg and a HR of 0.4 mg/kg for for thiacloprid in stone fruits.

Grapes

Field trials on grapes were provided from Japan.

GAP in Japan for grapes consists of up to two applications at 0.53 kg ai/ha each and a PHI of 21 days. The residues from 4 supervised trials in Japan matching the GAP (± 30%) were: 0.12, 0.44, 0.80 and 1.6 mg/kg. The Meeting decided that four residue trials were not sufficient for a recommendation for grapes.

Berries and other small fruits except grapes

Field and glasshouse trials on strawberries were provided from Belgium, France, Germany, Japan, the Netherlands, Italy, Spain and the United Kingdom.

The GAP for field use in the Netherlands and the United Kingdom for strawberries consists of up to two applications at 0.12 kg ai/ha each and a PHI of three days. The residues from eight supervised trials in Europe matching the GAP were: 0.02, 0.03, 0.04, 0.07, 0.07, 0.08, 0.08 and 0.09 mg/kg.

GAP for glasshouse use in the Netherlands and the United Kingdom for strawberries is up to two applications at 0.12 to 0.14 kg ai/ha each and a PHI of one day. The residues from eight supervised trials in Europe matching the GAP (± 30%) were 0.04, 0.05, 0.13, 0.22, 0.31(3) and 0.33 mg/kg.

GAP in Japan for protected strawberries is up to three applications with 0.23 kg ai/ha each and a PHI of one day. The residue trials from Japan were conducted with an application rate of 0.15 kg ai/ha, which does not correspond to the submitted GAP.

Field trials on currants were provided from Belgium, Germany and the United Kingdom. GAP in Germany, Latvia, the Netherlands and the United Kingdom for currants is up to three applications with 0.072 to 0.14 kg ai/ha each and a PHI of three days. The residues from eight supervised trials in Europe matching the GAP (± 30%) were: 0.08, 0.16, 0.21, 0.21, 0.28, 0.35, 0.37 and 0.59 mg/kg.

Field trials on raspberries were provided from Germany and the United Kingdom. GAP in Germany, the Netherlands and the United Kingdom for raspberries is up to three applications with 0.096 to 0.14 kg ai/ha each and a PHI of three days. The residues from eight supervised trials in Europe matching the GAP (± 30%) were: 0.1, 0.15, 0.15, 0.27, 0.31, 0.34, 0.34 and 0.62 mg/kg.

Various GAPs in Germany, Latvia, the Netherlands, Poland, Switzerland and the United Kingdom for small fruits and berries is up to three applications with 0.12 to 0.14 kg ai/ha each and a PHI of three days. The Meeting decided to make a recommendation for the whole group of berries and other small fruits except grapes based on the combined data for protected strawberries, currants and raspberries.

For protected strawberries, currants and raspberries the combined results were 0.04, 0.05, 0.08, 0.1, 0.13, 0.15, 0.15, 0.16, 0.21, 0.21, 0.22, 0.27, 0.28, 0.31(4), 0.33, 0.34, 0.34, 0.35, 0.37, 0.59 and 0.62 mg/kg.

Based on residue data for protected strawberries, currants and raspberries the Meeting recommends a maximum residue level of 1 mg/kg, a STMR of 0.275 mg/kg and a HR of 0.62 mg/kg for thiacloprid in berries and other small fruits except grapes.

Kiwi fruits

The Meeting received information on supervised residue trials on kiwi fruits from New Zealand.

GAP in New Zealand for kiwi fruit is up to two applications with 0.0096 kg ai/hL each before the flowering. The residues from nine supervised trials in New Zealand matching the GAP (± 30%) were: < 0.02 (5), 0.03, 0.04, 0.06 and 0.1 mg/kg.

The Meeting recommended a maximum residue level of 0.2 mg/kg, an STMR value of 0.02 mg/kg and a HR of 0.1 mg/kg for thiacloprid in kiwi fruits.

Onions

The Meeting received information on supervised residue trials on onions from Brazil and Germany.

GAP in Belize, Brazil, Costa Rica, Dominican Republic, El Salvador, Guatemala. Honduras, Nicaragua and Panama for onions is up to 0.1 kg ai/ha and a PHI of 21 days. Only one supervised residue trial from Brazil matched this GAP. The corresponding residue was < 0.02 mg/kg in bulb onion. From Germany two additional trials were provided with residues of < 0.01 and < 0.01 mg/kg.

The Meeting concluded that the data available for onions was not sufficient to support an STMR or MRL recommendation.

Garlic

The Meeting received information on supervised residue trials on garlic from Brazil.

GAP in Belize, Brazil, Costa Rica, Dominican Republic, El Salvador, Guatemala. Honduras, Nicaragua and Panama for garlic is up to 0.1 kg ai/ha and a PHI of 21 days. Neither of the two supervised residues trials matched the GAP for garlic within ± 30%.

The Meeting concluded that the data available for garlic was not sufficient to support a recommendation.

Cucumbers

The Meeting received information on supervised residue trials on field and glasshouse grown cucumbers from Belgium, France, Germany, Greece, the Netherlands, Italy and Spain.

The GAP for field use in Croatia, Cyprus, Georgia, Greece, Italy, the Netherlands and Spain for cucumbers is up to four applications at 0.12 to 0.15 kg ai/ha each and a PHI of one to three days. The residues from eight supervised trials in Europe matching the GAP (± 30%) were: 0.02, 0.03(3), 0.04, 0.1, 0.11 and 0.14 mg/kg.

The GAP for glasshouse use in the United Kingdom for cucumbers, which reflects the critical GAP, is up to three applications at 0.21 kg ai/ha each and a PHI of three days. The residues from 12

supervised trials in Europe matching the GAP (± 30%) were 0.04, 0.04, 0.07, 0.07, 0.08(4), 0.12, 0.15, 0.15 and 0.18 mg/kg.

The Meeting decided to pool the data from outdoor and indoor residues trials for a recommendation on cucumbers. The combined results are 0.02, 0.03(3), 0.04(3), 0.07, 0.07, 0.08(4), 0.1, 0.11, 0.12, 0.14, 0.15, 0.15 and 0.18 mg/kg.

For gherkins, GAPs from Greece and the Netherlands were available, which correspond to the GAPs for cucumber. The Meeting concluded that an extrapolation of the data from cucumbers to gherkins is not possible, because of the different surface area-to-mass ratio for gherkins, from higher residues can be expected than in cucumbers.

Based on the combined data for cucumbers the Meeting recommended a maximum residue level of 0.3 mg/kg, an STMR value of 0.08 mg/kg and a HR of 0.18 mg/kg for cucumbers.

Squash, summer

The Meeting received GAPs for courgettes and squash corresponding to the uses in cucumbers and gherkins. The treatment methods cover foliar spraying as well as drip application. The Meeting concluded that the residue data for cucumber can be extrapolated to summer squash.

Based on an extrapolation from cucumbers the Meeting recommended a maximum residue level of 0.3 mg/kg, an STMR value of 0.08 mg/kg and a HR of 0.18 mg/kg for thiacloprid in summer squash.

Melons and watermelons

The Meeting received information on supervised residue trials on melons from France, Greece and Italy. Data on protected melons was also received from Japan.

GAP in Croatia, Italy and Spain for melons and watermelons is up to three applications at 0.14 kg ai/ha each and a PHI of three to four days. The residues for whole melon fruits from six supervised trials in Europe matching the GAP (± 30%) were < 0.02, 0.02, 0.03, 0.05, 0.06 and 0.06 mg/kg. In melon pulp all residues were < 0.02(6) mg/kg.

GAP in Japan for protected melons is up to three applications at 0.45 kg ai/ha each and a PHI of one day. The residues from two supervised trials in Japan matching the GAP (±30%) were < 0.005 and < 0.005 mg/kg in the pulp.

Field trials on watermelons were available from Greece and Spain. Data on protected watermelons was also available from Japan.

GAP in Croatia, Italy and Spain for watermelons is up to three applications at 0.14 kg ai/ha each and a PHI of three to four days. The residues for whole watermelon from four supervised trials in Europe matching the GAP (± 30%) were < 0.02(3) and 0.06 mg/kg. In watermelon pulp all residues were < 0.02(4) mg/kg.

GAP in Japan for protected watermelons is up to three applications at 0.45 kg ai/ha each and a PHI of one day. The residue trials from Japan were conducted with an application rate of 0.3 kg ai/ha, which did not correspond to the submitted GAP.

The Mann-Whitney-U test for melons and watermelons indicated that a similar distribution for melons and watermelons can be assumed. The combined residues for whole melons and watermelons were < 0.02(4), 0.02, 0.03, 0.05, 0.06, 0.06 mg/kg.

The Meeting decided to pool the data for melons and watermelons for mutual support and recommended a maximum residue level of 0.2 mg/kg for thiacloprid in melons and watermelons and an STMR of 0.02 mg/kg and HR value of 0.02 mg/kg for melon and watermelon pulp.

Squash, winter

GAP in Cyprus and the Netherlands for squash, field and glasshouse grown, is up to four applications at 0.014 kg ai/hL and a PHI of one to three days. This use corresponds to the GAP available for melons and watermelons in field. The Meeting concluded that the residue data for melon and watermelon can be extrapolated for use in winter squash.

Based on an extrapolation from melon and watermelon the Meeting recommends a maximum residue level of 0.2 mg/kg for thiacloprid in winter squash and an STMR of 0.02 mg/kg and HR value of 0.02 mg/kg for winter squash pulp.

Tomatoes

The Meeting received information on supervised residue trials on field and glasshouse grown tomatoes. Supervised trials were provided for field use from France and Italy and for glasshouse use from Germany, France, Japan and Spain. In addition, residue trials with drip application in glasshouse were conducted in Belgium and the Netherlands.

Supervised residue trials of field were conducted with two applications of 0.14 up to 0.22 kg ai/ha each and PHIs from zero to eight days. Corresponding GAPs from Greece and Slovenia were available with a PHI of three days. The residues for tomatoes from seven trials in Europe, matching the GAP (± 30%), in ranked order were: 0.02, 0.03, 0.03, 0.04, 0.05, 0.09 and 0.16 mg/kg.

For foliar use in glasshouses, data from eight supervised residue trials were provided corresponding to the GAP of the United Kingdom (three applications at 0.22 kg ai/ha each and a PHI of three days). The residues from protected tomatoes from eight trials in Europe matching the UK GAP (± 30%) in ranked order were: 0.07, 0.12, 0.12, 0.15, 0.18, 0.19, 0.25 and 0.29 mg/kg.

GAP in Japan for protected tomatoes is up to three applications with 0.23 kg ai/ha each and a PHI of one day. The residue trials from Japan were conducted with an application rate of 0.38 kg ai/ha, which does not correspond to the submitted GAP.

In the Netherlands drip application to glasshouse tomatoes is registered at an application rate of 0.0096 kg ai per 1000 plants and a PHI of three days. The corresponding residues from eight trials on protected tomatoes in Europe matching the GAP (± 30%) were: < 0.02(3), 0.02(3), 0.03 and 0.03 mg/kg.

Based on the glasshouse foliar spray GAP for tomatoes the Meeting recommended a maximum residue level of 0.5 mg/kg, an STMR value of 0.165 mg/kg and a HR value of 0.29 mg/kg for thiacloprid in tomatoes.

Peppers, sweet

Supervised residue field trials were provided from France, Italy and Spain. Data for glasshouse use as foliar spray was generated in France, the Netherlands and Spain. In addition, residue trials with drip application in glasshouse were conducted in Belgium and the Netherlands.

Supervised residue trials in field use with thiacloprid were conducted with two applications of 0.14 up to 0.22 kg ai/ha each and PHIs from zero to seven days. Corresponding GAPs from Greece and Slovenia are available with a PHI of three days. The residues for peppers from seven trials in Europe matching the GAP (± 30%) in ranked order were: 0.05, 0.06, 0.08, 0.1, 0.11, 0.21 and 0.45 mg/kg.

For the use as a foliar spray in glasshouse eight supervised residue trials were conducted, corresponding to the UK GAP (three applications at 0.22 kg ai/ha and a PHI of three days). The residues for protected peppers from eight trials in Europe matching GAP (± 30%) were: 0.07, 0.08, 0.1, 0.11, 0.33, 0.37, 0.37 and 0.38 mg/kg.

GAP in Japan for protected peppers is up to three applications with 0.23 kg ai/ha each and a PHI of one day. The residues from two supervised trials in Japan matching the GAP ±30% were 1.1 and 2.0 mg/kg. The Meeting compared the data sets for Japan and Europe using the Mann-Whitney-U test and decided that they belonged to different populations and could not be combined. Therefore only data from European trials was used for further evaluation.

In the Netherlands drip application in glasshouses is registered for peppers with an application rate of 0.0096 kg ai per 1000 plants and a PHI of three days. The corresponding residues from eight trials on protected peppers in Europe matching GAP (± 30%) were: 0.04, 0.04, 0.05(4), 0.06, 0.07 mg/kg.

For chili peppers GAPs are available, which correspond to the GAPs for sweet peppers. The Meeting concluded that an extrapolation of the data from sweet peppers to chili pepper is not possible, because of the different surface area to mass ratio for chili peppers, for which higher residues than in sweet peppers can be expected.

Based on the glasshouse foliar spray GAP for peppers the Meeting recommended a maximum residue level of 1 mg/kg, an STMR value of 0.22 mg/kg and a HR value of 0.38 mg/kg for thiacloprid in sweet peppers.

Eggplants

Field trials on protected aubergines were provided from Japan.

GAP in Japan for eggplants consists of up to three applications at 0.23 kg ai/ha each and a PHI of one day. The residues from two supervised trials in Japan matching the GAP (± 30%) were 0.28 and 0.38 mg/kg.

The Meeting received GAPs for eggplants from the Netherlands, Japan, the United Kingdom and various other countries corresponding to the GAO for field and glasshouse tomatoes. The treatment methods cover foliar spraying as well as drip application. The Meeting concluded that the residue data for tomatoes can be extrapolated to support the use in eggplants.

The Meeting compared the data sets eggplant from Japan and for protected tomatoes using the Mann-Whitney-U test and decided that they belonged to the same population and could be combined. The combined eggplant and protected tomato residues were: 0.07, 0.12, 0.12, 0.15, 0.18, 0.19, 0.25, 0.28, 0.29 and 0.38 mg/kg.

Based on an extrapolation from the critical glasshouse foliar spray GAP for tomatoes and residue trials for eggplants from Japan the Meeting recommended a maximum residue level of 0.7 mg/kg, an STMR value of 0.185 mg/kg and a HR value of 0.38 mg/kg for thiacloprid in eggplants.

Potatoes

The Meeting received information on supervised field trials on potatoes from Belgium, Brazil, France, Germany, Italy, Japan, Spain and the United Kingdom.

The 16 supervised trials available from Europe for potatoes were conducted with up to three applications at 0.096 kg ai/ha each and a PHI of 21 days. This corresponds to the GAP from Austria, Cyprus, Greece, Portugal, Romania, Spain and the United Kingdom. The residues in potato tuber were < 0.02(16) mg/kg.

GAP in Japan for potatoes is up to three applications with 0.23 kg ai/ha each and a PHI of seven days. The residues from two supervised trials in Japan matching GAP (± 30%) were: < 0.005 and < 0.005 mg/kg in the tubers.

In addition the Meeting received information from two supervised residue trials on potatoes from Brazil. The application rates were 0.14 and 0.29 kg ai/ha with a PHI of 21 days. No residue above the LOQ of 0.02 mg/kg was found in potato tubers.

The Meeting recommended a maximum residue level of 0.02 (*) mg/kg and an STMR value and HR value of 0 mg/kg for thiacloprid in potatoes.

Wheat

Field trials on wheat were provided from France and Germany.

Thiacloprid is registered for use on wheat in Romania and Lithuania. The application rates are 0.048 kg ai/ha and 0.034 kg ai/ha respectively with a PHI of 21 days for Lithuania and an undefined PHI for Romania. The Meeting received supervised residue trials on wheat with application rates of 0.05 up to 0.062 kg ai/ha, which corresponds to + 29% of the GAP. Residues in wheat grain were < 0.02 (5), 0.03 (3), 0.04 and 0.04 mg/kg.

The Meeting recommended a maximum residue level of 0.1 mg/kg, an STMR value of 0.025 mg/kg and a highest residue value of 0.04 mg/kg for thiacloprid in wheat grain.

Barley

The Meeting received information from supervised residue trials on barley from France and Germany.

Thiacloprid is registered for on barley in Romania. The application rate is 0.048 kg ai/ha with an undefined PHI. The Meeting received supervised residue trials on barley with application rates of 0.062 kg ai/ha, which corresponds to + 29% of the GAP. Residues in barley grain were < 0.02, 0.05, 0.06, 0.11 and 0.12 mg/kg.

The Meeting decided that there was insufficient data from which to recommend a maximum residue level for thiacloprid on barley.

Rice

Field trials on rice were provided from India and Japan. GAP in India for foliar spraying of rice is 0.12 kg ai/ha each and a PHI of 30 days. All supervised residue trials were performed, with application rates of 0.18 up to 0.36 kg ai/ha which were up to 3 × GAP. In addition, only the limit of detection of 0.001 mg/kg was reported for thiacloprid in rice. Nevertheless no residues above this LOD were detected in rice grain without husks or in the husks in any of the six supervised residue trials.

GAP in Japan for rice consists of up to three applications with 0.15 kg ai/ha without a PHI. In two residue trials with an application rate of 1.5 kg ai/ha no residue above the LOQ of 0.005 mg/kg could be detected in the grain after 117, and up to 152 days.

The Meeting concluded that the LOQ of the monitoring method (0.02 mg/kg) is an appropriate estimate for MRL values in rice.

The Meeting recommended a maximum residue level of 0.02 (*) mg/kg, an STMR value of 0 mg/kg and a highest residue of 0 mg/kg for thiacloprid in rice husks.

Maize

Field trials on maize were provided from France, Germany, Greece and Italy. In Europe GAP is available from Romania (an application rate of 0.048 kg ai/ha and no PHI). Eight supervised residue trials were conducted with two treatments of 0.075 kg ai/ha each and a PHI of 28 – 31 days. These trials did not match any of the GAPs provided to the Meeting.

The Meeting concluded that the residue data on maize was not sufficient for recommending MRL, STMR or HR values.

Tree nuts

The Meeting received information on supervised field trials on walnuts from Italy. GAP in Argentina, Chile, Italy and the United Kingdom consists of up to two applications at 0.0096 – 0.018 kg ai/hL and a PHI of 1 to 14 days. The four trials provided were performed at above GAP rate (0.03 kg ai/hL), but no residue could be detected in thenut kernel above the trial specific LOQ of 0.005 mg/kg.

Field trials were provided on almonds from USA.

GAP in Italy and the United Kingdom is up to two applications with 0.012 – 0.018 kg ai/hL and a PHI of 14 days. The residues for almond kernel from 14 trials in the USA matching the GAP (± 30%) were: < 0.01(13), 0.01 mg/kg.

Field trials were provided on pecan from USA.

GAP in Italy is up to 0.018 kg ai/hL and a PHI of 14 days. The residues for pecan kernel from 14 trials in USA matching the GAP (± 30%) were < 0.01(14) mg/kg.

Various GAPs in Germany, Italy and Turkey for tree nuts consist of up to two applications at rates of 0.0096 to 0.012 kg ai/hL each and a PHI of 21 days. The Meeting concluded that an extrapolation from almonds, walnuts and pecan to the whole group of tree nuts is possible. As thiacloprid is non-systemic, it was concluded that residues in nuts were comparable from different areas in the world. The combined thiacloprid residues in nuts were: < 0.01 (31), 0.01 mg/kg.

Because the analytical methods for enforcement are validated with a LOQ of 0.02 mg/kg, this value is used for the maximum residue level proposal for tree nuts.

The Meeting recommended a maximum residue level of 0.02 mg/kg and an STMR and HR value of 0.01 mg/kg for thiacloprid in tree nuts.

Oilseed rape and white mustard

Field trials on oilseed rape were provided from France, Hungary, Germany, Spain and Sweden.

Various GAPs in Czech Republic, Slovakia, Switzerland and the United Kingdom are up to two applications with 0.0096 up to 0.14 kg ai/ha. The residues in rapeseeds from 14 supervised trials matching the GAP ± 30% were < 0.02(3), 0.02, 0.03, 0.05, 0.06, 0.07(3), 0.09, 0.1, 0.22, 0.33 mg/kg.

The GAP in Czech Republic for white mustard is up to two applications with 0.096 kg ai/ha each and no PHI reported. The Meeting concluded that residue trials for rapeseed can be extrapolated to white mustard seed.

The Meeting recommended a maximum residue level of 0.5 mg/kg, an STMR value of 0.065 mg/kg and a HR value of 0.33 mg/kg for thiacloprid in rapeseed and white mustard seeds.

Cotton seeds

Field trials on cotton were provided from Greece, Spain and the USA.

For cotton, two sets of supervised residue trials from Europe and USA were made available. The trials conducted in the USA were analyzed using a total residue method measuring 6-CNA. In the European trials total thiacloprid residue, determined as 6-CNA, and thiacloprid only, were analyzed. This data shows clear differences in the residue levels. Therefore the Meeting concluded that the residue data from USA for cotton would not be considered for further evaluation. The residue trials from Europe were conducted with three applications of 0.096 kg ai/ha each and a PHI of 21 days. This use pattern corresponded to GAPs from Greece, Guatemala, Spain and Turkey. The residues in cotton seed from eight supervised trials matching the GAP (± 30%) were < 0.02(8) mg/kg.

The Meeting recommended a maximum residue level of 0.02 (*) mg/kg and an STMR and HR value of 0.02 mg/kg for thiacloprid in cotton seeds.

Sunflower seeds

The Meeting received information from one field trial on sunflowers from Hungary.

Registered uses of thiacloprid on sunflowers are available from Hungary and Slovakia. The application rates are 0.036–0.048 kg ai/ha and an undefined PHI and a PHI of 30 days, respectively. The one supervised trial on sunflowers (application rate of 0.097 kg ai/ha) did not correspond to any available GAP.

The Meeting concluded that the available residue data on sunflowers was not sufficient for a recommendation of MRL, STMR of HR values.

Green tea

Field trials on green tea were made available from Japan.

GAP in Japan for green tea is one applications at 0.6 kg ai/ha and a PHI of seven days. The residue trials from Japan were conducted with an application rate of 0.3 kg ai/ha, which did not correspond to the submitted GAP. The Meeting concluded that a recommendation of maximum residue levels for green tea was not possible.

Wheat forage

Field trials on wheat were provided from France and Germany.

Registered uses of thiacloprid on wheat are available from Romania and Lithuania. The application rates are 0.048 kg ai/ha and 0.034 kg ai/ha respectively with a PHI of 21 days for Lithuania and an undefined PHI for Romania. The Meeting received supervised residue trials on wheat with application rates of 0.05 up to 0.062 kg ai/ha, which corresponds to + 29% of the GAP. Residues in wheat forage were: 1.2, 1.2, 1.3(3), 1.7, 1.8, 1.8, 1.9 and 2.2 mg/kg.

The Meeting estimated an STMR value of 1.5 mg/kg and a highest residue value of 2.2 mg/kg for thiacloprid in wheat forage.

Wheat straw

Field trials on wheat straw were available from France and Germany.

Registered uses of thiacloprid on wheat straw are available from Romania and Lithuania with application rates of 0.048 kg ai/ha and 0.034 kg ai/ha respectively, with a PHI of 21 days for Lithuania and an undefined PHI for Romania. The Meeting received supervised residue trials on wheat with application rates of 0.05 up to 0.062 kg ai/ha, which corresponded to + 29% of the GAP. Residues in wheat straw were 0.06, 0.07, 0.07, 0.14, 0.53, 0.89, 0.97, 1.2, 1.6 and 1.7 mg/kg.

The Meeting estimated an STMR value of 0.71 mg/kg and a highest residue value of 1.7 mg/kg for thiacloprid in wheat straw.

Based on 88% dry weight matter the residues in wheat straw (dry matter) were 0.07, 0.08, 0.08, 0.16, 0.6, 1.0, 1.1, 1.3, 1.8, 1.9 mg/kg. The Meeting estimated a MRL of 5 mg/kg for wheat straw (dry matter based).

Almond hulls

Field trials on almonds were made available from the USA.

GAP in Italy and the United Kingdom is up to two applications with 0.012 – 0.018 kg ai/hL and a PHI of 1 to 14 days. The residues for almond hulls from 14 trials in the USA matching the European GAP (± 30%) were 0.99, 1.3, 1.4, 1.5, 1.8, 1.8, 2.0, 2.1, 3.2, 3.3, 3.3, 3.4, 4.5, 4.9 mg/kg.

The Meeting estimated an STMR value of 2.05 mg/kg and a highest residue of 4.9 mg/kg for thiacloprid in almond hulls (fresh weight).

Based on 90% dry weight matter the residues in almond hulls were 1.1, 1.4, 1.6, 1.7, 2.0, 2.0, 2.2, 2.3, 3.5, 3.6, 3.6, 3.7, 5.0 and 5.4 mg/kg. The Meeting estimated a MRL of 10 mg/kg for almond hulls (dry matter based).

Rape forage

The Meeting received information on supervised residue trials on oilseed rape from France, Hungary, Germany, Spain and Sweden.

Various GAPs in the Czech Republic, Slovakia, Switzerland and the United Kingdom consist of up to two applications at 0.0096 to 0.14 kg ai/ha with PHI between zero and 30 days. The residues in rape forage from 12 supervised trials matching the GAP (± 30%) were 1.0, 1.1(4), 1.2, 1.4, 1.5, 1.6, 1.7, 1.9 and 2.2 mg/kg.

The Meeting estimated an STMR value of 1.3 mg/kg and a highest residue of 2.2 mg/kg for thiacloprid in rape forage (fresh weight).

Cotton gin by-products

Field trials on cotton gin by-products were provided from Greece, Spain and the USA.

For cotton two sets of supervised residue trials from Europe and USA were made available. The residue trials conducted in the USA were analyzed using a total residue method measuring 6-CNA. In the European trials total thiacloprid residue, determined as 6-CNA, and thiacloprid only, were analyzed. Residues analyzed with the total residue method are much higher than thiacloprid only residues and can not be extrapolated to evaluate the residue situation. In the supervised residue trials according to the residue definition "thiacloprid only" no gin trash samples were analyzed. A recommendation for a STMR or highest residue value for cotton gin by-products was not possible.

Fate of residues during processing

Thiacloprid was generally stable to hydrolysis during pasteurization, baking and boiling conditions.

Information on the fate of thiacloprid residues during food processing was available for melons and watermelons, apples, peaches, cherries and tomatoes.

Calculated processing factors and the mean or best estimate are summarized in the following table.

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factor	Estimate of the processing factor
Apples	Apple, dried	0.3, 0.7	0.5
	Apple, juice	0.2, 0.29	0.25
	Apple, sauce	0.6, 0.86	0.73
	Apple, pomace dry	4.3, 8.7	6.5
Peaches without stone	Peach, preserve	0.22, 0.66, 0.66	0.66
Tomatoes	Tomatoes, peeled	0.25, 0.43	0.34
	Tomato, paste	2, 3.1	2.6
	Tomato, juice	0.42, 0.71	0.615
	Tomato, preserve	0.33, 0.71	0.52

For apples the estimated processing factors are applied to the STMR value of 0.11 mg/kg for pome fruits. The Meeting estimated STMR-P values for dried apple of 0.055 mg/kg, for apple juice of 0.0275 mg/kg, for apple sauce of 0.077 mg/kg and for apple pomace dry of 0.71 mg/kg.

For peaches the estimated processing factors are applied to the STMR value of 0.08 mg/kg for stone fruits. The Meeting estimated STMR-P values of 0.05 mg/kg for preserved peaches.

For cherries it was not possible to calculate processing factors as residues in the RAC were below the limit of quantification.

For tomatoes the estimated processing factors are applied to the STMR value of 0.165 mg/kg. The Meeting estimated STMR-P values for peeled tomatoes of 0.056 mg/kg, for tomato paste of 0.429 mg/kg, for tomato juice of 0.1 mg/kg and for tomato preserve of 0.086 mg/kg.

Farm animal dietary burden

The Meeting estimated the dietary burden of thiacloprid residues for ruminants based on STMR and highest residue values obtained from the submitted supervised residue trials. The diets are described in Appendix IX of the *FAO Manual* (FAO, 2002).

Estimated maximum dietary burden of farm animals

Crop	Residue (mg/kg)	Basis	Group	Dry matter (%)	Residue/ Dry matter (mg/kg)	Dietary content (%)			Residue contribution (mg/kg)		
						Beef cattle	Dairy cows	Poultry	Beef cattle	Dairy cows	Poultry
Apple, dry pomace	0.72	STMR-P	AB	100	0.72	*40*	*20*		0.29	0.14	0
Rape, forage	2.2	HR	AM	30	7.33	*30*	*30*		2.20	2.20	0
Cottonseed (meal)	0.02	HR	-	89	0.02			*20*	0.00	0.00	0.004
Wheat, forage	2.2	HR	AF	25	8.80	*25*	*50*		2.20	4.40	0
Wheat, grain	0.04	HR	GC	89	0.045	*5*		*80*	0.002	0.000	0.036
Total						*100*	*100*	*100*	4.7	6.7	0.04

Estimated median dietary burden of farm animals

Crop	Residue (mg/kg)	Basis	Group	Dry matter (%)	Residue/ Dry matter (mg/kg)	*Dietary content (%)*			Residue contribution (mg/kg)		
						Beef cattle	*Dairy cows*	*Poultry*	Beef cattle	Dairy cows	Poultry
Apple, dry pomace	0.72	STMR-P	AB	100	0.65	*40*	*20*		0.29	0.14	0
Rape, forage	1.3	STMR	AM	30	4.33	*30*	*30*		1.3	1.3	0
Cottonseed (meal)	0.02	STMR	-	89	0.02			*20*	0	0	0.004
Wheat, forage	1.5	STMR	AF	25	6.00	*25*	*50*		1.5	3	0
Wheat, grain	0.025	STMR	GC	89	0.03	*5*		*80*	0.001	0	0.022
Total						*100*	*100*	*100*	3.1	4.4	0.03

The dietary burdens of thiacloprid for estimation of MRL and STMR values for animal commodities are for beef cattle 4.7 and 3.1 mg/kg and for dairy cows 6.7 and 4.4 mg/kg respectively. For poultry a dietary burden of 0.04 and 0.03 mg/kg was calculated.

Farm animal feeding studies

The Meeting received animal feeding studies on ruminants. No study on poultry feeding was available.

Three groups of cows were dosed at levels equivalent to 2.1 (0.07 mg/kg bw) (1×), 6.2 (0.213 mg/kg bw) (3×) and 20.6 ppm (0.655 mg/kg bw) (10×) of thiacloprid in the diet together with a control group. On average from the cows treated at the 1× dose level, the liver contained the highest thiacloprid residue levels (0.10 mg/kg) followed by kidney (0.03 mg/kg), milk and muscle (0.02 mg/kg) and fat (0.01 mg/kg). Maximum levels for tissues were 0.02 mg/kg for fat, 0.02 mg/kg for muscle, 0.04 mg/kg for kidney and 0.11 mg/kg for liver.

In the second dose group thiacloprid residues increased to an average value of 0.04 mg/kg in milk and fat (highest value 0.04 mg/kg), 0.05 mg/kg in muscle (highest value 0.06 mg/kg), 0.1 mg/kg in kidney (highest value 0.11 mg/kg) and 0.29 mg/kg in liver (highest value 0.32 mg/kg). In the high dose group the residues found were 0.17 mg/kg in milk, 0.12 mg/kg in fat (highest value 0.16 mg/kg), 0.16 mg/kg in muscle (highest value 0.18 mg/kg), 0.27 mg/kg in kidney (highest value 0.32 mg/kg) and 0.94 mg/kg in liver (highest value 1.1 mg/kg).

A linear relation between the dose levels and the residue concentrations was observed.

In milk, residues reached a plateau level within five days and no accumulation was observed.

For poultry no feeding studies were provided. In the metabolism study based on a feeding level of 10 mg/kg bw (corresponding to 124 ppm in feed, based on dry weight) thiacloprid residues of 0.06 mg/kg in eggs, 0.03 mg/kg in muscle, 0.08 mg/kg in fat and 0.54 mg/kg in liver were found.

Animal commodity maximum residue levels

The dietary burden for beef and diary cattle was estimated at a maximum level 4.7 and 6.7 mg/kg respectively. The maximum residue level to be expected in tissues can be obtained from the results of feeding at a level of 6.2 ppm.

Dietary burden (mg/kg)[1]	Feeding level [ppm][2]	Thiacloprid residue level (mg/kg)[3]				
		Milk (mean)	Fat (high)	Muscle (high)	Liver (high)	Kidney (high)
MRL dairy cattle	(6.7)	*(0.04)*	*(0.04)*	*(0.06)*	*(0.32)*	*(0.11)*
	[6.2]	*0.04*	*0.04*	*0.06*	*0.34*	*0.11*
		Milk (mean)	Fat (mean)	Muscle (mean)	Liver (mean)	Kidney (mean)
STMR dairy cattle	(4.4)	*(0.02, 0.04)*	*(0.02, 0.04)*	*(0.02, 0.05)*	*(0.1, 0.29)*	*(0.04, 0.1)*
	[2.1,6.2]	*0.03*	*0.03*	*0.035*	*0.21*	*0.07*

1 In parentheses, estimated dietary burden

2 In square brackets, actual feeding level in transfer studies

3 Values in parentheses in italics are derived from the dietary burden, feeding levels and residue levels found in the transfer studies ´. "high" is the highest residue level in an individual tissue in the relevant feeding group. "mean" is the mean residue level in milk in the relevant feeding group.

The median dietary burdens were 3.1 mg/kg for beef cattle and 4.4 mg/kg for dairy cattle. The burden for dairy cows is between the dose levels of 2.1and 6.2 mg/kg of the animal feeding study. Therefore the mean value for each dose group and each commodity is taken for STMR estimation. The values are 0.03 mg/kg for milk, 0.03 mg/kg for mammalian fat, 0.035 mg/kg for mammalian meat and 0.21 mg/kg for edible offal, mammalian. For HR the calculated residues based on the maximum

estimated dietary burden were 0.04 mg/kg for mammalian fat, 0.06 mg/kg for mammalian meat and 0.34 for mammalian edible offal.

Based on the highest residues found in the feeding study (3× dose) the Meeting estimated maximum residue levels of 0.1 mg/kg for mammalian meat and 0.5 mg/kg for mammalian edible offal. Based on the mean value the Meeting estimated a maximum residue level of 0.05 mg/kg for milk.

For poultry no feeding studies are available. When the calculated maximum dietary burden for poultry is extrapolated from the results of the poultry metabolism study the resulting residue levels are far below 0.01 mg/kg. The Meeting estimated an STMR value and a highest residue of 0 mg/kg for thiacloprid in poultry products.

The Meeting estimated a MRL of 0.02 (*) mg/kg for poultry meat, poultry edible offal and eggs.

RECOMMENDATIONS

The Meeting estimated the STMR, HR and MRL values shown below.

The definition for the residue in plant and animals (enforcement and risk assessment) is: *thiacloprid.*

The residue is not fat soluble.

Commodity		**MRL, mg/kg**		**HR, mg/kg**	**STMR or STMR-P, mg/kg**
CCN	**Name**	**New**	**Previous**		
AM 0660	Almond hulls	10		5.4	2.05
DF 0226	Apple, dried			0.19	0.055
JF 0226	Apple, juice				0.0275
FB0018	Berries and other small fruits except grapes	1		0.62	0.275
SO 0691	Cotton seed	0.02 (*)		0.02	0.02
VC 0424	Cucumbers	0.3		0.18	0.08
MO 0105	Edible offal, mammalian	0.5		0.34	0.21
VO 0440	Eggplants	0.7		0.38	0.185
PE 0112	Eggs	0.02 (*)		0	0
FI 0341	Kiwi fruits	0.2		0.1	0.02
MF 0100	Mammalian fats, except milk fats			0.04	0.03
MM 0095	Meat, mammalian	0.05		0.06	0.03
VC 0046	Melons	0.2		0.02	0.02
ML 0106	Milks	0.05		-	0.03
SO 0495	Oilseed rape	0.5		0.33	0.065
VO 0445	Pepper, sweet	1		0.38	0.22
FP 0009	Pome fruits	0.7		0.38	0.11
VR 0589	Potatoes	0.02 (*)		0	0
PM 0110	Poultry meat	0.02 (*)		0	0
PO 0111	Poultry, edible offal of	0.02 (*)		0	0
	Rape forage (fresh weight)			2.2	1.3
GC 0649	Rice	0.02 (*)		0	0
VC 4207	Squash, summer	0.3		0.18	0.08

VC 0431	Squash, winter	0.2		0.02	0.02
FS0012	Stone fruits	0.5		0.40	0.08
JF 0448	Tomato, juice				0.1
	Tomato, paste				0.429
	Tomato, peeled				0.056
VO 0448	Tomatoes	0.5		0.29	0.165
TN 0085	Treenuts	0.02		0.01	0.01
VC 0432	Watermelons	0.2		0.02	0.02
GC 0654	Wheat	0.1		0.04	0.025
	Wheat, forage	-		2.2	1.5
AS 0654	Wheat, straw	5		1.7 (fresh matter)	0.71 (fresh matter)
SO 0485	White mustard	0.5		0.33	0.065

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDI) of thiacloprid based on 13 GEMS/Food regional diets were in the range of 1–10% of the maximum ADI of 0.01 mg/kg bw. The Meeting concluded that the long-term intake of residues of thiacloprid from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short Term Intake (IESTI) of thiacloprid on the basis of the recommendations made by the JMPR represented 0–90% of the ARfD (0.03 mg/kg bw) for children and 0–30% for the general population. The Meeting concluded that the short-term intake of residues of thiacloprid resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

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The annual Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and WHO Core Assessment Group on Pesticide Residues was held in Rome, Italy, from 3 to 12 October 2006. The FAO Panel of Experts had met in Preparatory Sessions from 28 September to 2 October. The meeting was held in pursuance of recommendations made by previous meetings and accepted by the governing bodies of FAO and WHO that studies should be undertaken jointly by experts to evaluate possible hazards to humans arising from the occurrence of pesticide residues in foods. During the meeting, the FAO Panel of Experts was responsible for reviewing pesticide use patterns (good agricultural practices), data on the chemistry and composition of the pesticides and methods of analysis for pesticide residues and for estimating the maximum residue levels that might occur as a result of the use of the pesticides according to good agricultural practices. The WHO Core Assessment Group was responsible for reviewing toxicological and related data and for estimating, where possible, acceptable daily intakes (ADIs) of the pesticides for humans. This report contains information on ADIs, maximum residue levels and general principles for the evaluation of pesticides. The recommendations of the joint meeting, including further research and information, are proposed for use by Member Governments of the respective agencies and other interested parties.

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