PIRIMICARB (101)

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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

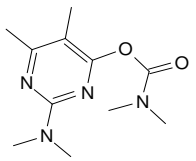
CA: 2-(dimethylamino)-5,6-dimethyl-4-pirimidinyl dimethylcarbamate

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.

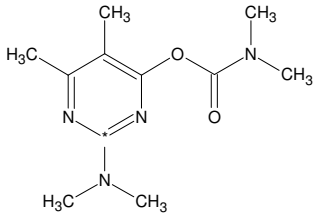


Figure 1. Label position (*) in ^{14}C labelled pirimicarb used in metabolism studies.

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.

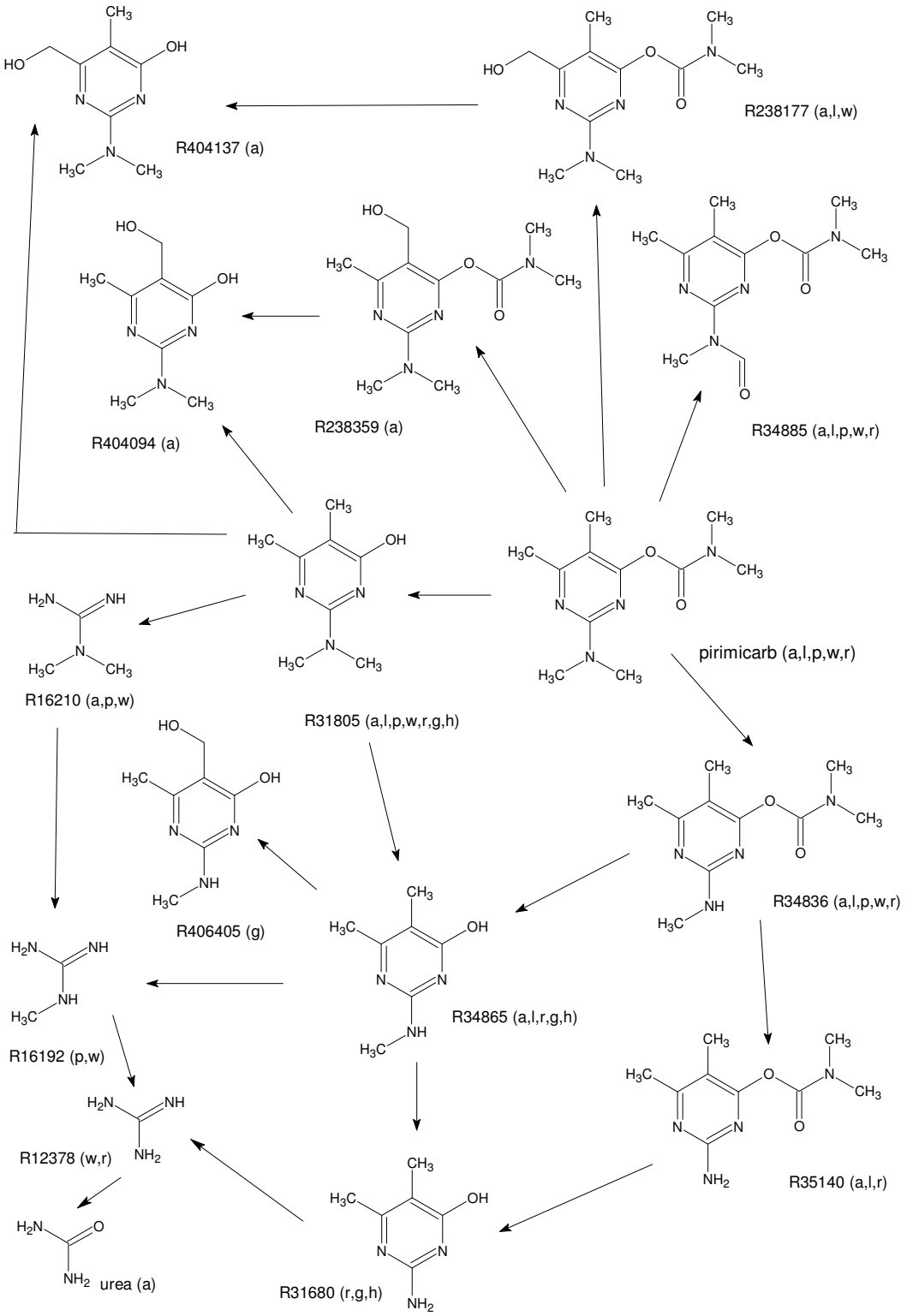


Figure 2. Metabolism scheme for plants and livestock (a= apple, l = lettuce, p = potato, w = wheat, r= rotational crops, g = goat, h= hen).

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			PHI, (days)	Residues (mg/kg)			Reference & Comments
	kg ai/ha	kg ai/hL	no		Pirimicarb	R34836 +R34885[1]	R238177	
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		0.05	1	-0	< 0.01	< 0.01	< 0.01	PP62/1099
(Escrito)			2	0	0.02	< 0.01	< 0.01	(AF/5966/SY/1)
				3	0.02	≤ 0.01	< 0.01	
				7	< 0.01	< 0.01	< 0.01	field crop
France (S), 2001		0.05	1	-0	< 0.01	< 0.01	< 0.01	PP62/1099
(Matisse)			2	0	0.03	< 0.01	< 0.01	(AF/5966/SY/2)
				3	0.08	0.01	< 0.01	
				7	0.04	0.01	< 0.01	field crop
Italy, 2001		0.05	1	-0	0.01	0.02	< 0.01	PP62/1099
(Tamaris)			2	0	0.05	< 0.01	< 0.01	(AF/5966/SY/3)
				3	0.02	0.01	< 0.01	
				7	< 0.01	< 0.01	< 0.01	field crop
Italy, 2001		0.05	1	-0	< 0.01	< 0.01	< 0.01	PP62/1099
(Pamir)			2	0	< 0.01	< 0.01	< 0.01	(AF/5966/SY/4)
				3	0.05	≤ 0.01	< 0.01	
				7	< 0.01	< 0.01	< 0.01	field crop
France (N), 2001		0.025	1	-0	< 0.01	< 0.01	< 0.01	PP62/1181
(Buffalo)			2	0	0.01	< 0.01	< 0.01	(AF/5967/SY/2)
				3	0.02	< 0.01	< 0.01	
				7	0.02	< 0.01	< 0.01	protected crop
France (N), 2002		0.025	1	-0	< 0.01	0.01	< 0.01	PP62/1301
(Cezanne)			2	0	0.03	< 0.01	< 0.01	(AF/6541/SY/1)
				3	0.03	0.26	< 0.01	
				7	0.02	0.01	< 0.01	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	0.25	0.031	1	-0	< 0.01	< 0.01	< 0.01	PP62/1201
(Paola)			2	0	0.31	0.02	< 0.01	(AF/5961/SY/4)
				3	0.02	0.02	< 0.01	
				7	0.02	< 0.01	< 0.01	
France (N), 2001	0.25	0.032	1	-0	< 0.01	< 0.01	< 0.01	PP62/1201
(Picnic)			2	0	0.07	< 0.01	< 0.01	(AF/5961/SY/1)
				3	0.01	< 0.01	< 0.01	
				7	< 0.01	< 0.01	< 0.01	
France (N), 2001	0.25	0.032	1	-0	< 0.01	< 0.01	< 0.01	PP62/1201
(Valina)			2	0	0.15	< 0.01	< 0.01	(AF/5961/SY/2)
				3	0.05	< 0.01	< 0.01	
				7	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	
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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