FIPRONIL (202)

Fipronil belongs to a new class of insecticides known as phenylpyrazoles. It was first reviewed by the 1997 JMPR for toxicology only, and was identified as a candidate for residue evaluation by the 2000 JMPR by the 1998 CCPR (ALINORM 99/24). The evaluation was postponed to the 2001 JMPR.

Information was reported to the Meeting by the manufacturer Aventis CropScience on metabolism in animals and plants, environmental fate in soil and water, methods of residue analysis and stability of residues in stored analytical samples, registered uses, residues in supervised trials, fate during processing and national MRLs. Information on national GAP was provided by the governments of Australia and Poland. The Meeting was informed that no authorized uses exist in Germany or The Netherlands.

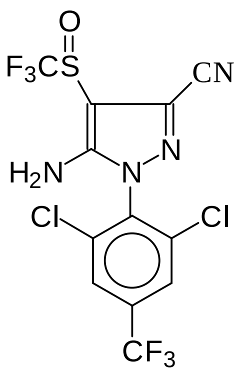
IDENTITY

BSI common name: fipronil

Chemical name:

IUPAC	(±)-5-amino-1-(2,6-dichloro-α,α,α-trifluoro-*p*-tolyl)-4-trifluoromethylsulfinylpyrazole-3-carbonitrile
CA	(±)5-amino-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-[trifluoromethyl)sulfinyl]-1*H*-pyrazole-3-carbonitrile

CAS No: 120068-37-3
CIPAC No: 581
Synonyms: MB 46030
Structural formula:



Molecular formula: $C_{12}H_4Cl_2F_6N_4OS$
Molecular weight: 437.1

Physical and chemical properties

Pure active ingredient

Appearance:	White powder	(Chabert and Lecourt, 1996)
Melting point:	203°C	(Chabert and Lecourt, 1996)

Octanol/water partition coefficient:		
HPLC method	log P_{OW} 3.5 at 20°C	(Cousin, 1997a)
Shake-flask method	log P_{OW} 4.0 at 20°C	(Chabassol and Reynaud, 1991a)
Hydrolysis:		
pH 5 (buffered)	Stable	(Corgier and Plewa, 1992a)
pH 7 (buffered)	Nearly stable (2% loss on 30 days)	
pH 9 (buffered)	DT-50 approximately 28 days	
Photolysis:		
	DT-50 0.33 days (k=-0.0176 $days^{-1}$)	(Corgier and Plewa, 1992b)
	Quantum yield (Φ_{300}) at 300 nm, 1.99×10^{-1}	(Boinay, 1997)
Dissociation constant:	Not determinable due to low water solubility	

Technical material

Physical and chemical properties:

Dry technical:[1]		
Minimum purity	95% (950 g/kg)	
Wet technical:[1]		
Minimum purity	87% (870 g/kg)	
Water	6% (6.0 g/kg)	
Vapour pressure:	3.7×10^{-9} h Pa at 25°C	((Chabassol and Reynaud, 1991b)
Solubility:		
Water, distilled (20°C)	1.9 mg/l	(Chabassol and Reynaud, 1991c)
buffered (pH 5, 20°C)	2.4 mg/l	((Chabassol and Reynaud, 1991c)
buffered (pH 7, 25°C)	3 mg/l	(Buddle, 1991)
buffered (pH 9, 20°C)	2.2 mg/l	(Chabassol and Reynaud, 1991c)
Organic solvents, g/100 ml		(Chabassol and Reynaud, 1991d)
acetone	54.6	
dichloromethane	2.2	
ethyl acetate	26.5	
hexane	0.003	
methanol	13.8	
1-octanol	1.2	
2-propanol	3.6	
toluene	0.3	
Relative density:	1.48-1.63 (20°C)	(Chabassol and Hunt, 1991a)
Stability:		
Thermal	No degradation at 30-150°C	(Chabassol, 1992)
Flammability	Not highly flammable; not autoflammable	(Fillion, 1996)
Oxidizing potential	Unreactive in water, ammonium dihydrogen phosphate, metallic zinc, diute neutral potassium permanganate	(Chabassol and Hunt, 1991b; Tran Thahn Phong, 1999)
Explosivity:	Not explosive	

<u>Pure metabolites</u>

Vapour pressure:		
MB 46136	7.6×10^{-7} Pa at 25°C	(Cousin, 1996a)
MB 45950	2.3×10^{-6} Pa at 25°C	(Cousin, 1996b)
MB 46513	0.4×10^{-5} Pa at 25°C	(Cousin, 1995)
Water solubility: (distilled, 20°C)		
MB 46136	0.16 mg/l	(Cousin, 1997c)
MB 45950	1.1 mg/l	(Cousin, 1998a)
MB 46513	0.95 mg/l	(Cousin, 1997e)
Octanol/water partition coefficient:		
MB 46136	Log P_{OW} 3.8, 20°C	(Cousin, 1997b)
MB 45950	Log P_{OW} 3.7, 20°C	(Cousin, 1998b)
MB 46513	Log P_{OW} 3.4, 20°C	(Cousin, 1997d)

Formulations

The following list includes the main formulations developed for crop uses:

Formulation type	ai content	Principal formulation names
Suspension concentrate (SC)	50, 200, 400, 750 g/l	Regent®, Ascend®, Klap®
Seed treatment suspension concentrate (FS)	10, 20, 50,	LeSak®
	250, 500, 750 g/l	Regent, Cosmos®, Icon®
Water-dispersible granule (WG)	800 g/kg	Regent, Cazador®, Schuss®
Microgranule (GR), sand, Biodac® or clay based	3, 5, 10, 15, 20 g/kg	Regent, Prince®
Ultra low volume (UL)	Range of 2-25 g/l	Adonis®
Emulsifiable concentrate (EC)	25, 300 g/l	Regent, Adonis
Granular bait	0.03 g/kg	Blitz®

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METABOLISM AND ENVIRONMENTAL FATE

Compounds are identified by code numbers as shown below. The chemical names do not conform to either IUPAC or CA nomenclature, but have been used to emphasize the relation between the compounds.

Code	Chemical name
fiponil (MB 46030)	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulfinylpyrazole
MB 45950	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylthiopyrazole
MB 45897	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)pyrazole
MB 46136	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulfonylpyrazole
fipronil-desulfinyl (MB 46513)	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylpyrazole
MB 46400	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)pyrazole-4-carboxylic acid
RPA 106889	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)pyrazole-3,4-dicarboxylic acid
RPA 104615	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)pyrazole-4-sulfonic acid
RPA 105320	5-amino-3-carbamoyl-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulfonylpyrazole

Code	Chemical name
RPA 105048	5-amino-3-carbamoyl-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylpyrazole
RPA 200761	5-amino-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulfinylpyrazole-3-carboxylic acid
RPA 200766	5-amino-3-carbamoyl-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulfinylpyrazole

Animal metabolism

Animal metabolism studies (Powles, 1992; Totis and Fisher, 1994; Steward, 1994a,b) were reviewed by the JMPR in 1997 for toxicology.

Rats. In an ADME (absorption, distribution, metabolism and excretion) study (Powles, 1992), rats were dosed orally with ^{14}C-phenyl ring-labelled fipronil in aqueous methyl cellulose (0.5% w/v) containing Tween 80 (0.01% w/v). Groups of 5 males and 5 females were treated as follows.

- Group A: single oral doses of 4 mg/kg bw [^{14}C]fipronil
- Group B: 14 daily oral doses of unlabelled fipronil followed by single labelled doses, all 4 mg/kg bw
- Group C: single oral doses of 150 mg/kg bw [^{14}C]fipronil.

After treatment the rats were placed in metabolism cages and urine, faeces and blood were collected over 7 days. Expired air was passed through organic traps to ensure the recovery of all radioactive residues. After seven days, the rats were killed and blood and selected tissues were sampled. There were no significant differences in the disposition of radiolabelled materials between the sexes within any treatment group. Recoveries of ^{14}C were all greater than 95%.

Table 1. Percentage recoveries of radioactivity from rats after dosing with [^{14}C]fipronil, group mean values (Powles, 1992).

Group	Sex	Urine	Faeces	Cage washes	Cage debris	Tissues	Total
A	Male	5.6	45.6	0.88	0.022	46.1	98.2
A	Female	5.6	46.0	1.2	ND	45.8	98.6
B	Male	16.2	56.1	1.6	0.03	23.7	97.6
B	Female	13.8	61.4	2.9	0.22	20.2	98.5
C	Male	29.3	66.9	3.8	0.68	2.9	103.6
C	Female	22.0	75.1	3.0	1.02	5.3	106.4

Absorption/kinetics

The amount of dose absorbed appeared to be dependent on dosage and regimen when the urine and tissue results were combined. Group A absorbed approximately 50%, group B about 40% and group C about 30% of the radioactivity. The absorbed fipronil was readily metabolized. No unconjugated [^{14}C]fipronil was detected in the urine or tissues.

In Groups A and C the rate of decrease of radioactivity in the blood was similar for both sexes. The half-lives in Group A for males were 149 ± 11 h and for females 200 ± 59 h, which may be due to the slow release of radioactivity from a tissue such as fat. In Group C residues decreased more rapidly than in group A, with half-lives of 54.4 ± 20 h for males and 51 ± 10.5 h for females.

Excretion

Most of the radioactivity was eliminated in the faeces in all groups. Proportions varied with the dosage, but were the same for males and females. Metabolites were selectively cleared by renal and/or hepatic mechanisms. The presence of metabolites in faeces would suggest biliary excretion.

Table 2. Elimination as % of applied radioactivity at 168 h - group mean values (Powles, 1992).

Group	Sex	Faeces	Urine	Total excreted
A	Male	45.6	5.6	51.2
A	Female	46.0	5.6	51.6
B	Male	56.1	16.2	72.3
B	Female	61.4	13.8	75.2
C	Male	66.9	29.3	96.2
C	Female	75.1	22.0	97.1

Distribution

Seven days after exposure to [^{14}C]fipronil residues were highest in the fat, with moderate levels in the adrenal gland, pancreas, skin, liver, kidney, muscle, thyroid, and ovaries and uterus in females. The levels were lower in other tissues.

Table 3. Concentrations of radioactivity in the tissues of rats after oral administration of [^{14}C]fipronil - group mean values (Powles, 1992).

Sample	^{14}C, µg/g as fipronil					
	A male	A female	B male	B female	C male	C female
Abdominal fat	14.7	18.8	5.8	5.8	29.4	54.5
Adrenals	4.3	4.7	1.5	1.4	7.6	14.6
Kidney	1.3	1.5	0.5	0.5	4.1	6.6
Liver	2.5	2.7	1.1	0.97	6.5	11.2
Muscle	0.83	0.98	0.39	0.31	1.8	3.2
Pancreas	3.6	6.0	2.1	1.98	8.9	15.0
Skin	2.5	3.7	1.3	1.1	7.9	17.5
Thyroids	2.3	3.5	0.88	1.5	1.4	7.7
Ovaries	-	5.1	-	1.7	-	15.6
Uterus	-	2.3	-	1.1	-	10.5

Biotransformation

Analysis by HPLC of fat, liver, kidney, muscle and uterus samples containing moderate to high levels of radioactivity indicated that the same main component was present in all tissues. This was characterized by co-chromatography with an authentic standard as the sulfonyl analogue MB 46136 and confirmed by mass spectrometry.

At least 11 radiolabelled metabolites from faeces extracts were resolved by HPLC. The main metabolites apart from polar components (probably conjugates) were identified. At early samplings, the main compound was unchanged fipronil with lesser amounts of MB 46136 and the trifluoromethylthio reduction product MB 45950, with the amide RPA 200766 identified in some samples. At later samplings the main metabolite was MB 46136.

High levels of very polar radiolabelled material were found by HPLC in unextracted undiluted urine samples. After deconjugation with enzyme preparations specific for cleavage of glucuronide and sulfate conjugates and chromatographic separation using a more polar solvent system, 14 compounds were resolved of which seven were characterized by chromatography and mass spectrometry. The two main components in the urine were evidently pyrazole-ring-opened compounds retaining two nitrogen atoms and the nitrile ligand. The five other identified compounds were the parent compound, MB 46136, MB 45950, MB 45897 formed by loss of the trifluoromethylsulfinyl group, and the amide RPA 200766. All were probably excreted as *N*-glucuronides in the urine since the pyrazole or pyrazole-derived moieties of the aglycones possess more than one possible site for the formation of glucuronide adducts.

In summary, when single doses of [^{14}C]fipronil were given to male and female rats at 4 or 150 mg/kg/bw or after exposure to 4 mg/kg/bw after pre-treatment with 14 daily unlabelled doses the ^{14}C was quantitatively recovered. The proportion of dose absorbed appeared to depend on dosage and regimen for both sexes with the highest absorption after the single low doses. Metabolism was rapid. No unmetabolized fipronil was detected in any tissues or urine. Most of the radioactivity was excreted in the faeces which contained unchanged [^{14}C]fipronil and metabolites, suggesting bilary elimination of absorbed and metabolized fipronil, and direct elimination of unabsorbed fipronil. This implies some excretion in the bile. Tissue concentrations were high 7 days after dosing, with the highest levels in the fat. The main residue in the fat and other tissues was MB 46136.

Kinetics

In another study by Totis and Fisher (1994) radioactivity was measured in the blood and tissues of Charles River CD strain (Sprague-Dawley) male and female rats after single oral doses of 4 or 40 mg/kg of [^{14}C]fipronil.

Blood radioactivity was determined in groups of 5 rats of each sex over 336 hours. Absorption was rapid for the 4 mg/kg group (mean T_{max} 5.5 h) but elimination was relatively slow. Absorption was much slower for the 40 mg/kg group (mean T_{max} 36 h) in which an initial period of rapidly falling levels in the blood was followed by much slower elimination. Half-lives were similar at both doses (40 mg/kg group half-life: 135 $\pm$ 16 h (males), 171 $\pm$ 27 h (females); 4 mg/kg group half-life: 185 $\pm$ 22 h (males), 245 $\pm$ 35 h (females).

Tissue samples from groups of 3 male and 3 female rats were analysed for total radioactivity by LSC at 4 sampling times. The results are shown in Table 4. Concentrations in the tissues peaked at the blood T_{max} for males and females except in the stomach and gastrointestinal tract which were involved in absorption.

Table 4. Radioactivity in the tissues of rats - group mean values (Totis and Fisher, 1994).

Sample	^{14}C, µg equivalents as fipronil							
	4 mg/kg bw				40 mg/kg bw			
	0.75h	4.8h	96h	168h	3h	33.6h	77h	168h
Males								
Stomach and contents	147	0.5	0.33	0.56	381	64	10	0.88
Abdominal fat	11	31	24	16	69	229	115	32
Adrenals	9.0	11	8.3	5.2	34	54	20	16
Kidney	3.3	3.5	2.1	1.5	14	17	8.8	3.3
Liver	9.2	6.8	3.4	2.4	31	36	17	5.8
Muscle	1.8	3.0	0.91	0.76	7.6	10	4.6	1.5
Pancreas	5.2	6.7	6.5	4.5	31	38	13	6.2
Skin and fur	1.9	5.1	4.1	3.3	17	30	16	6.4
Thyroids	3.7	5.1	2.7	2.2	25	29	17	10
Females								
	0.83h	6.2h	94h	168h	3h	38.4h	78h	168h
Stomach and contents	53	0.73	0.40	0.57	185	148	8.7	1.3
Abdominal fat	13	31	25	22	80	201	135	39
Adrenals	10	9.7	5.1	3.9	39	47	29	14
Kidney	4.1	3.4	1.8	1.6	16	16	11	1.1
Liver	12	7.7	3.2	2.9	32	32	20	6.3
Muscle	1.8	2.1	0.99	1.3	7.4	8.8	5.9	2.0
Pancreas	6.1	5.3	3.2	2.6	26	32	20	5.6
Skin & fur	2.4	5.4	3.8	3.9	20	29	19	6.2
Thyroids	4.2	4.1	2.7	2.9	16	16	23	13

Sample	^{14}C, µg equivalents as fipronil							
	4 mg/kg bw				40 mg/kg bw			
	0.75h	4.8h	96h	168h	3h	33.6h	77h	168h
Ovaries	5.9	5.6	5.4	4.6	20	44	20	9.9
Uterus	2.1	3.9	3.3	2.5	18	30	11	7.2

Goats. In a study by Stewart (1994b) repeated daily oral doses of [^{14}C]fipronil in capsules were given to three dairy goats at 0.05, 2 or 10 ppm in the diet (dry matter basis) for 7 days. The doses were given in the morning and afternoon before feeding after milk and excreta collections. The radiolabelled fipronil-derived material retained in tissues or excreta was characterized by a combination of chromatography and spectrometry.

83% of the total dose was recovered from the 0.05 ppm dose, 64% of which was in the faeces (Table 5). 18% was estimated to have been retained in the tissues, none was detected in the urine, and 0.86% was recovered from the milk. At 2 ppm 50% was recovered, 25% in tissues with 2.5%, 18% and 4.6% in the urine, faeces and milk respectively. At 10 ppm 77% was recovered: 61% in the faeces, 6.6% in urine, 1.3% in milk and 7.4% in tissues. It was suggested that the unrecovered radioactivity was retained in the carcase.

Table 5. Recovery of applied radioactivity after oral administration of [^{14}C]fipronil to lactating goats (Stewart, 1994b).

Sample	^{14}C, % of dose		
	0.05 ppm	2 ppm	10 ppm
Urine	ND	2.45	6.58
Faeces	64.16	17.8	61.28
Whole milk	0.86	4.64	1.33
Cage washes	ND	0.04	0.14
Cage debris	ND	ND	0.54
Tissues	18.31	25.41	7.44
Total	83.32	50.32	77.3

ND: not detectable

After day 1, over 40% of the daily radioactive dose was recovered from the low- and high-dose groups. From days 2 to 6, daily recoveries were approximately 58% and 74% respectively and excretion appeared to reach a plateau after dosing at 0.05 ppm. At 10 ppm recoveries were erratic, but on days 2 and 3, this animal received only half the daily dose which may have influenced the pattern of elimination. Excretion of the daily dose increased over the study at 2 ppm, with a maximum of 47% recovered on day 7.

No radioactivity was detected in blood or plasma from the 0.05 ppm dose, and at 2 ppm levels were 0.023 mg/kg fipronil equivalents in the blood and 0.034 mg/kg in plasma. Concentrations increased during the study. A similar pattern was observed in the blood at 10 ppm where concentrations increased from 0.016 to 0.052 mg/kg before the afternoon dose on days 1 and 7 respectively, but in the plasma no definite pattern was identified: concentrations varied from 0.011 to 0.086 mg/kg during the study.

At all intervals levels of radioactivity from the low dose in the milk were <0.001 mg/kg as fipronil. At 2 and 10 ppm however, residues in the milk increased from 0.02 to 0.11 mg/kg and from 0.052 to 0.17 mg/kg respectively from days 1 to 7.

23.5 hours after the last dose at 0.05 ppm, the TRR (total radioactive residue) in the tissues was <0.01 mg/kg as fipronil. At 2 and 10 ppm, the highest residues were in fat: 1.3 and 1.9 mg/kg in omental and 1.3 and 1.95 mg/kg in renal respectively. At 2 ppm, residues were 0.07, 0.1 and 0.4 mg/kg in muscle, kidney and liver respectively and at 10 ppm 0.08 mg/kg in muscle, 0.15 mg/kg in kidney and 0.86 mg/kg in liver. As in milk, residues at 2 ppm were 24 to 165 times higher than at 0.05 ppm, but after a fivefold increase in the dose from 2 to 10 ppm, the residues in tissues rose only about twofold.

Extracts of urine, faeces and milk (day 7), muscle, kidney, liver and omental and renal fat from the intermediate and high-dose groups were analysed by HPLC. Samples from the high-dose group were subjected to GC-MS and/or LC-MS. The combination of techniques produced strong evidence that fipronil was the main residue in the high-dose milk and fat: 0.1 and about 1.4 mg/kg respectively. Metabolites MB 45950 and MB 46136 were also present. Although the individual components comprising the TRR in kidney and muscle represented <0.05 mg/kg fipronil equivalents, the parent compound plus MB 46136 were confirmed to be present. In the liver, the main metabolite was MB 46136 (0.46 mg/kg as fipronil) representing approximately 53% of the TRR; minor metabolites identified were RPA 200766 (0.098 mg/kg fipronil equivalents) and fipronil (0.013 mg/kg). Several minor metabolites in liver were not identified, but none were present at >0.052 mg/kg fipronil equivalents. HPLC analysis of equivalent extracts after the intermediate dose confirmed MB 46136 to be the main component of the TRR in all samples.

At both doses, most of the radioactivity was associated with polar compounds. Deconjugation with an enzyme preparation specific for cleavage of glucuronide and sulphate conjugates appeared not to change the elution profile, although this does not necessarily indicate the absence of glucuronide and sulfate conjugates. At the high dose, MB 46136 was a minor constituent in deconjugated and original urine. HPLC and GC-MS analysis confirmed that the main component present in the faeces was MB 46136. Parent compound, MB 45950 and a polar component were also identified as constituents of the residue. Although not verified by GC-MS, these components also apparently contributed to the total radioactivity at 2 ppm. The results are shown in Table 6.

In conclusion, after seven daily oral doses of [^{14}C]fipronil to dairy goats at nominal levels of 0.05 and 10 ppm, the administered radioactivity was extensively excreted, mainly in the faeces. At 2 ppm a greater proportion of the administered dose was retained in the animal. Recoveries from urine, milk and tissues indicated a minimum absorption of approximately 19%, 33% and 15% at 0.05, 2 and 10 ppm respectively. At 0.05 ppm the percentage of the TRR in the milk was negligible. At 2 and 10 ppm, the TRR in milk increased during the study. At the intermediate dose, the levels in milk attained a steady state. Consistently with the lipophilic nature of the compound and its metabolites, the main residues were in fat, providing supportive evidence that unrecovered radioactivity was retained in the animal. The parent compound and the metabolites MB 46136, MB 45950 and RPA 200766 were the principal compounds, although the proportions varied with the dose.

Table 6. Compounds in the milk and tissues of a dairy goat dosed with fipronil at a level equivalent to 10 ppm in the diet (Stewart, 1994b).

Sample	Peak identified	Retention time (min)	Compound	Residues	
				% of TRR	mg/kg fipronil equivalents
Milk	M1.10	46.0	fipronil	59.8	0.099
	M2.10	55.5	MB 45950	11.7	0.019
	M3.10	60.0	MB 46136	22.5	0.037
	M4.10	64.5	Unknown	1.53	0.003
			Remainder	4.5	
Kidney	K1.10	22.0	Unknown	1.51	0.002
	K2.10	24.0	Unknown	3.22	0.005
	K3.10	25.0	Unknown	3.40	0.005
	K4.10	26.0	fipronil	3.21	0.005
	K5.10	28.3	MB 46136	75.06	0.113
	K6.10	32.3	Unknown	0.39	0.001
			Remainder	13.21	
Liver	L1.10	19.1	Mwt 330[1]	4.49	0.039
	L2.10	21.2	RPA 200766	11.32	0.098

Sample	Peak identified	Retention time (min)	Compound	Residues	
				% of TRR	mg/kg fipronil equivalents
	L3.10	21.3	Unknown	2.67	0.023
	L4.10	22.3	Mwt 355[1]	6.0	0.052
	L5.10	22.5	Unknown	2.13	0.018
	L6.10	24.4	Mwt 353[1]	10.7	0.092
	L7.10	25.2	Unknown	2.42	0.021
	L8.10	25.4	Unknown	0.53	0.005
	L9.10	26.3	Mwt 313[1]	1.12	0.010
	L10.10	27.3	fipronil	1.54	0.013
	L11.10	29.1	MB 46136	52.93	0.456
	L12.10	41.2	Unknown	2.22	0.019
			Remainder	1.95	
Muscle	MU1.10	13.0	RPA 200766	7.22	0.006
	MU2.10	46.0	fipronil	60.76	0.048
	MU3.10	54.5	MB 45950	8.26	0.007
	MU4.10	59.5	MB 46136	20.51	0.016
	MU5.10	61.0	Unknown	1.79	0.001
			Remainder	1.45	
Fat, omental	OM1.10	12.5	RPA 200766	0.64	0.012
	OM2.10	45.5	fipronil	73.19	1.405
	OM3.10	54.0	MB 45950	5.47	0.105
	OM4.10	59.0	MB 46136	16.85	0.323
	OM5.10	62.0	Unknown	0.94	0.018
			Remainder	2.91	
Fat, renal	RF1.10	13.0	RPA 200766	0.74	0.014
	RF2.10	45.5	fipronil	72.72	1.414
	RF3.10	54.5	MB 45950	6.04	0.117
	RF4.10	59.5	MB 46136	17.95	0.349
	RF5.10	62.0	Unknown	0.94	0.018
			Remainder	1.60	

[1]Proposed molecular ions

Hens. In a study by Stewart (1994a) oral capsules containing levels equivalent to 0.05, 2 or 10 ppm in the diet (dry matter basis) were given to three groups of five laying hens daily for 28 days. The birds were dosed in the morning before feeding after egg and excreta collections, with an additional hen as a control. Residues were determined by chromatography and spectometry.

At 0.05 ppm a mean of 52% of the radioactivity was recovered within 23.5 hours of the last dose, and at 2 and 10 ppm mean recoveries were 55% and 58% respectively. At all doses the highest recoveries were from excreta, and egg yolks and whites (Table 7).

Table 7. Total recoveries of applied radioactivity from laying hens (Stewart, 1994a).

Sample	^{14}C, % of total administered dose		
	0.05 ppm	2 ppm	10 ppm
Excreta	28.35	36.28	41.47
Egg yolk	16.11	15.11	13.26
Egg white	1.99	1.68	1.44
Tissues (skin, fat, muscle, liver)	5.40	0.82	0.65
Cage wash	ND	0.06	0.07
Cage debris	ND	0.57	0.43
Total (mean)	51.9	54.53	57.53

ND: not detectable

Mean daily recoveries of radioactivity in excreta and eggs (expressed as a percentage of the daily dose) increased throughout the study.

Group mean maximum levels of 0.18, 7.02 and 30 mg/kg as fipronil were determined in egg yolks from the low-, intermediate- and high-dose groups respectively, and towards the end of the study almost reached a plateau. In all groups the residues in the egg yolks were higher than in the whites (low dose up to 78 times, intermediate and high up to 20 and 24 times respectively) consistent with the lipophilic nature of the compound. Concentrations of radioactivity in the whites were 0.011 mg/kg fipronil equivalents at the low dose, 0.3 mg/kg at the intermediate and 1.1 mg/kg at the high dose. The residues in the yolks at all doses displayed apparent dose proportionality, and in the whites only at the intermediate and high doses.

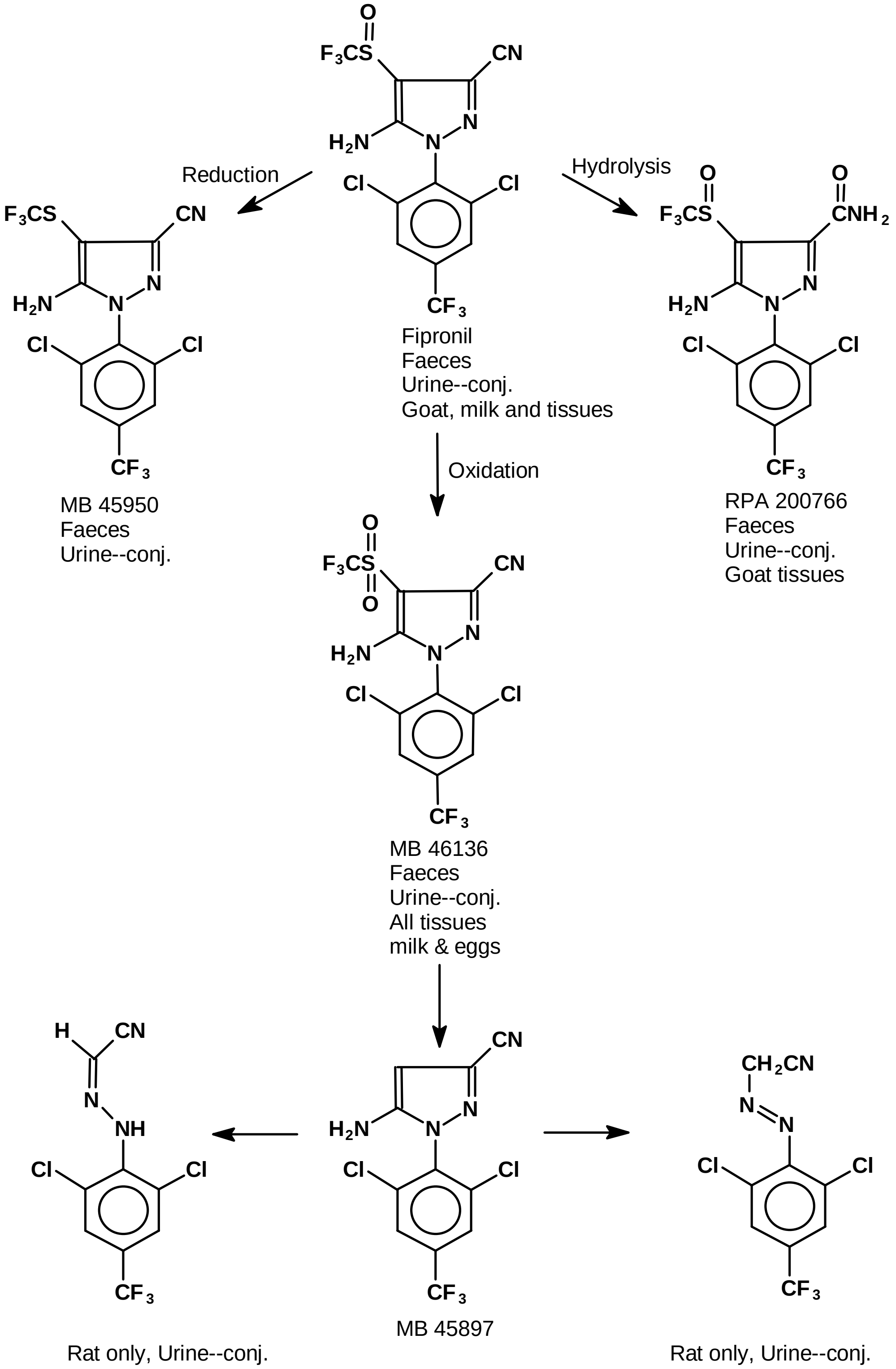
At the end of the study, 23.5 hours after the last dose, residues were highest in the skin, 0.1, 3.9 and 17 mg/kg as fipronil and in the peritoneal fat, 0.29, 12 and 56 mg/kg, at 0.05, 2 and 10 ppm respectively. Mean concentrations in liver and muscle were 0.03 mg/kg (low dose), 1.2 mg/kg (intermediate dose) and 4.9 mg/kg (high dose). In all tissues, residues were proportional to dose.

Extracts of egg yolk and white at day 27, and skin, peritoneal fat, liver and muscle from all groups were analysed by HPLC, and samples from the high-dose group by GC-MS. There was strong evidence that MB 46136 was the main component in egg yolks and whites and tissues of the high-dose group. Fipronil was a minor component in egg yolk, skin, fat and liver. Although samples from the other two groups were not analysed by GC-MS, the similar chromatography of these extracts strongly indicated that MB 46136 was also the main metabolite in the eggs and tissues at all doses.

Analysis of excreta by the same methods at day 27 confirmed that the main components were MB 46136 and fipronil. At the low and intermediate doses, the main residue was MB 46136, and at the high dose fipronil was the main component which may indicate that absorption of fipronil was incomplete at this dosage.

In summary approximately 52 to 58% of the administered radioactivity was eliminated, mainly in the excreta, at all doses and radioactive residues in both excreta and egg yolks and whites were close to reaching plateau levels. The elevated residues in egg yolk, skin and fat are consistent with the lipophilic nature of the compound. The metabolite MB 46136 was identified as the main residue in eggs and tissues at all doses. Figure 1 shows the metabolic pathway of fipronil in animals.

Figure 1. Metabolic pathways of fipronil in animals.



Animal metabolism of fipronil-desulfinyl (MB 46513)

Rats. In the ADME study (Totis, 1996) reviewed by the 1997 JMPR groups of male and female Sprague Dawley rats were dosed with [^{14}C]fipronil-desulfinyl labelled in the phenyl ring as follows.

- Single oral low doses (SOLD) of 1 mg/kg bw [^{14}C]fipronil-desulfinyl
- Single oral high doses (SOHD) of 10 mg/kg bw [^{14}C]fipronil-desulfinyl
- Repeat oral low doses (ROLD): 14 daily oral doses of 1 mg/kg bw unlabelled fipronil-desulfinyl followed by a single dose of 1 mg/kg bw [^{14}C]fipronil-desulfinyl.

After treatment the rats were placed in cages and urine and faeces were collected for 7 days. Exhaled carbon dioxide was not trapped during the study as results from an earlier group dosed at 15 mg/kg/day had shown <0.1% of the radioactivity was eliminated via this route during the first 48 hours. The rats were killed and samples of blood and selected tissues were collected. Recoveries of ^{14}C during the seven days were essentially quantitative, ranging from 93 to 100% (mean 97%, SD ± 3.7%).

In addition rats given single doses at 1 and 10 mg/kg were housed in wire cages and blood samples were taken approximately 0.5, 1, 2, 3, 4, 6, 8, and 24 hours after dosing, and then at 24 hour intervals until c. 360 hours (low-dose group) or 408 hours (high-dose group) after dosing, and finally at 48-72 hour intervals until 648 hours after dosing.

Absorption/kinetics

In all three regimes the percentage of [^{14}C]fipronil-desulfinyl absorbed was very similar, with a higher proportion absorbed by the females. The mean estimated percentage absorbed was calculated from the radioactivity detected in the urine, cage washes and tissues: approximately 31/44% (male/female), 34/46% (male/female) and 35/45% (male/female) for the high-, low- and repeated-dose groups respectively. The pharmacokinetic experiments indicated a relatively slow absorption at both doses with mean maximum blood concentrations approximately 46-73 hours after dosing (Table 8). The maximum concentrations in the blood appear to be proportional to the administered dose.

Table 8. Blood pharmacokinetic parameters of fipronil-desulfinyl in rats (Totis, 1996).

Parameter	Males		Females	
	Mean	SD	Mean	SD
C_{max} (µg equiv/g)				
SOHD	2.03	0.47	2.3	0.9
SOLD	0.14	0.02	0.15	0.03
T_{max} (hours)				
SOHD	73	9.1	71	8.3
SOLD	46	13.6	61	17.1
Half-life (hours)				
SOHD	170	21	221	55
SOLD	156	18	210	14

SD: Standard deviation

Distribution

The radioassay of tissues sampled seven days after administration of [^{14}C]fipronil-desulfinyl indicated that radioactivity was widely distributed; no residues were below the minimum limit of detection (MLD) after 168 hours. Residues were highest in the fat in all groups, and moderate to low in the liver, kidney, adrenals, lungs, thyroid, skin and fur, pancreas and uterus. Mean percentages of the dosed ^{14}C remaining in the tissues after seven days were 20 and 30% for SOHD males and females respectively; 27 and 41% for SOLD males and females respectively; and 22 and 32% for ROLD males and females respectively.

In all three groups, levels of radioactivity after 168 hours were indicative of a relatively slow elimination rate.

Elimination

In the three groups, the faeces were the main route of elimination for fipronil-desulfinyl, accounting for approximately 46% to 70% of the administered dose, and the urine for approximately 4% to 11%. After 24 hours, elimination was at a steady rate until the end of the study. More than 70% of the total was excreted after 96-120 hours in both the faeces and the urine. The proportion eliminated in the faeces was similar in all three groups. Mean levels in the faeces of males were slightly higher than in females (64% and 52% respectively). This difference was not apparent in the urine (8.4% and 8.6% respectively) but corresponds to the slightly higher radioactivity found in the tissues of females at slaughter.

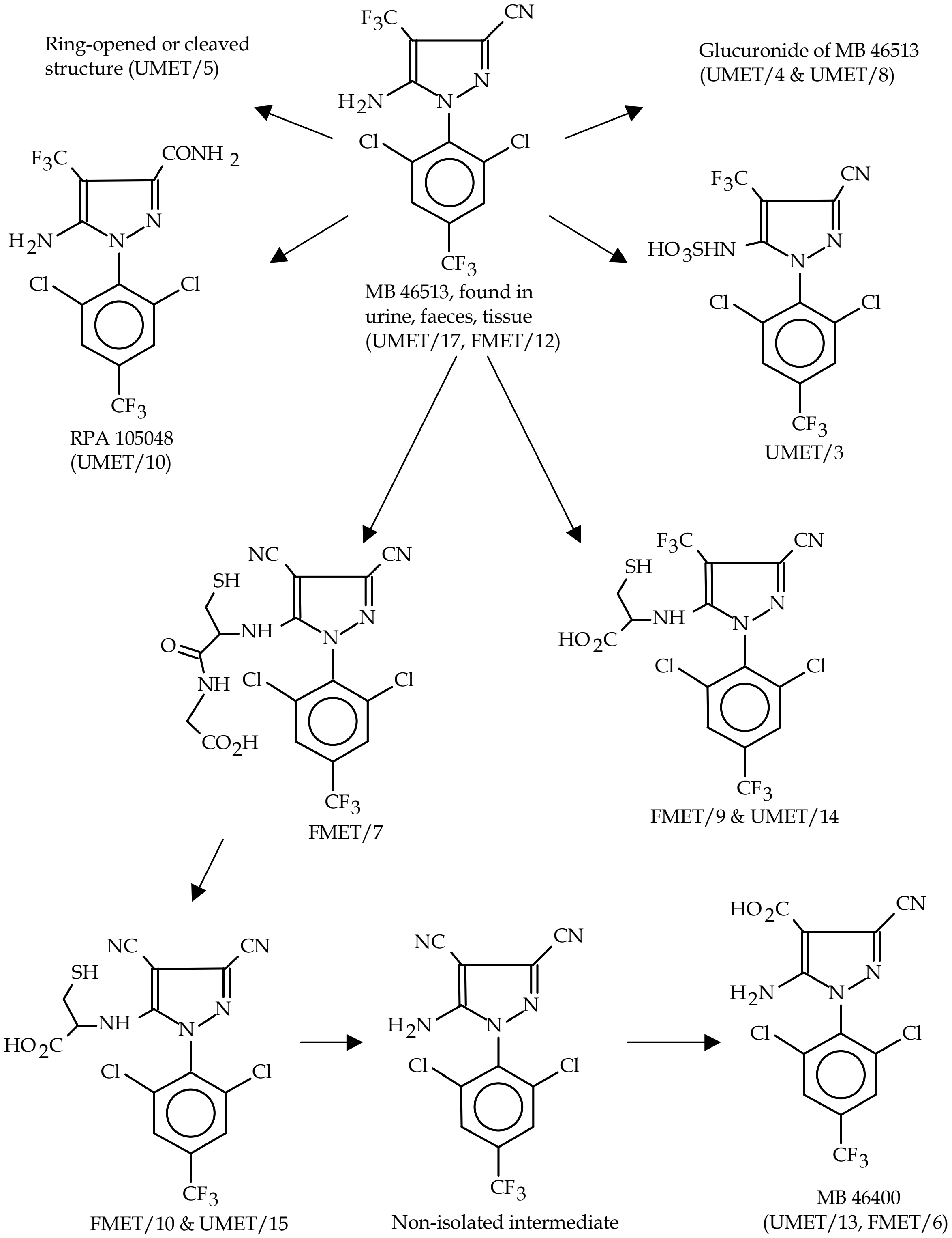
Metabolism

About 13 radiolabelled metabolites in the faeces and 17 in the urine extracts were resolved by HPLC, with identification by mass spectrometry (LC-MS, LC-MS-MS and/or GC-MS) and ^{19}F NMR. Trace levels of fipronil were detected in urine and it was the main component in the faeces in all the groups, which suggests that a large part of the administered dose was excreted in the faeces without absorption or as a conjugate via the bile which was de-conjugated by hydrolytic enzymes in the gut.

In the urine only the 4-carboxylic derivative MB 46400 (UMET/13. FMET/6. Figure 2) accounted for more than 5% of the dose. A second metabolite, UMET/3, accounted for over 2% in the animals from the repeat low-dose group; it was identified as a sulfate conjugate of fipronil-desulfinyl. Extracts from urine samples, including those from samples treated with enzyme preparations specific for cleavage of glucuronide and sulfate conjugates and those subjected to acid hydrolysis, were analysed by HPLC. Results obtained from incubations with the de-conjugating enzymes did not definitely demonstrate the presence of glucuronide or sulfate conjugates, but after acidic hydrolysis two other polar metabolites were thought to be amino acid conjugates of fipronil-desulfinyl. Structural identification using LC-MS indicated that UMET/8 was also a conjugate, tentatively identified as a 5-aminoglucuronide conjugate of the parent. UMET/10 was clearly identified as the amide RPA 105048. UMET/15 was proposed to be the 4-cyano-5-(*N*-cysteinyl) derivative of fipronil-desulfinyl. In faeces the second most abundant compound after the parent was FMET/10; after acidic hydrolysis, this metabolite was seen to decrease and therefore indicated to be a conjugate. LC-MS-MS and ^{19}F NMR showed that FMET/10 was the same compound as UMET/15, the 4-cyano-5-(*N*-cysteinyl) compound. FMET/6 was identified as the 4-carboxylic acid derivative of fipronil-desulfinyl. Acidic hydrolysis indicated that both FMET/9 and FMET/7 were conjugates; LC-MS-MS and ^{19}F NMR identified FMET/9 as the 5-(*N*-cysteinyl) conjugate of fipronil-desulfinyl and FMET/7 as a 4-cyano-5-(*N*-cysteinylglycine) conjugate linked through the cysteine residue. Polar metabolites FMET/1, FMET/2, and FMET/4 were found to be thermo-sensitive during hydrolysis experiments; LC-MS demonstrated that these were probably conjugates because of their high molecular weights.

Structures have been proposed for urinary metabolites which account for approximately 90% of the radioactivity eliminated in the urine and for faecal metabolites representing approximately 93% of that eliminated in the faeces. Only one radioactive residue was identified from tissue samples, this being the unchanged compound fipronil-desulfinyl. The proposed metabolic pathways of fipronil-desulfinyl in the rat are shown in Figure 2.

Figure 2. Proposed metabolic pathways of fipronil-desulfinyl in the rat.



Goats. In a study of distribution, metabolism, and elimination of radioactivity three diary goats were given repeated oral doses of [^{14}C]fipronil-desulfinyl in capsules at levels equivalent to 0.05, 2 and 10 ppm in the diet (dry matter basis) for seven days (Johnson *et al.*, 1996). Radioactive residue levels in the milk and selected tissues were determined.

The goats were dosed after milk and excreta collections before being fed in the morning and afternoon, and killed 23 hours after the last dose.

Recoveries were as follows. At 0.05 ppm, 72% of the total dose was recovered, with 19.5% in the faeces and 7.1% in urine, at 2 ppm, 52% with 26% and 4.7% in the faeces and urine respectively, and at 10 ppm, 69%, with 50% in faeces and 3.2% in urine. The highest residues in the tissues at all levels were in the liver and fat, with higher concentrations than in circulating plasma, and the lowest in muscle (Table 9).

Table 9. Mean recoveries of total radioactivity as % of administered dose after repeated oral dosing of lactating goats with [^{14}C]fipronil-desulfinyl (Johnson *et al.*, 1996).

Sample	^{14}C, % of total administered dose		
	0.05 ppm	2 ppm	10 ppm
Urine	7.1	4.7	3.2
Faeces	19.5	26	50
Cages wash	0.79	0.14	0.19
Milk	5.3	0.96	2.6
Total body fat	26	9.0	7.4
Kidneys	0.13	0.11	0.06
Liver	4.4	2.7	2.2
Skeletal Muscle	9.2	8.3	4.1

Concentrations of radioactivity in milk at each dose level approached a steady state about 104 hours after the first dose, and peaked at 0.008, 0.056, and 0.36 mg/kg fipronil-desulfinyl equivalents at the 0.05, 2, and 10 ppm dose levels respectively. Residues in the tissues 23 hours after the last dose are shown in Table 10.

Table 10. Total radioactivity in tissues after repeated oral dosing of lactating goats with [^{14}C]fipronil-desulfinyl (Johnson *et al.*, 1996).

Sample	TRR (mg/kg fipronil-desulfinyl equivalents)		
	0.05 ppm	2 ppm	10 ppm
Omental fat	0.078	0.57	2.7
Renal fat	0.066	0.53	2.2
Kidneys	0.0075	0.13	0.47
Liver	0.037	0.76	2.8
Skeletal muscle	0.0035	0.068	0.18

Samples from the 10 ppm goat were extracted for identification of residues. Extraction of radioactivity was essentially quantitative (>91%) from milk and all tissues except kidney (86%). Residues in urine were isolated by solid phase extraction and solvent elution. Extracts of milk, urine, faeces, liver, muscle, kidney and fat were examined using radio-HPLC and LC-MS. For metabolite profile comparison, liver and omental fat from the 0.05 ppm group and liver, omental fat, and milk from the 2 ppm group were analysed by HPLC. The chromatographic properties of components in these extracts were similar to those in the high-dose extracts indicating similar metabolism at all doses.

Analysis by radio-HPLC and LC-MS indicated that fipronil-desulfinyl was the only residue in faeces, renal fat, omental fat, and milk and also the main residue in liver, muscle, and kidney. Analysis of liver extracts by LC-MS indicated the presence of the deaminated ring-opened derivatives of RPA 106889 and RPA 105048, and ring-opened metabolite RPA 108058 (see Figure 3). One other unidentified metabolite, less than 10% of the TRR, was characterized by HPLC retention time as polar, possibly a derivative with the pyrazole ring intact. In muscle, one minor unidentified component (<0.02 mg/kg, 0.95% of the TRR) was detected in addition to fipronil-desulfinyl. In kidneys, all compounds except fipronil-desulfinyl were minor (0.82-5.07% of the TRR, 0.004 to 0.024

mg/kg. In addition to low levels of fipronil-desulfinyl, residues in urine were tentatively identified as 5-amino sulfate and glucuronide conjugates of fipronil-desulfinyl, its 5-(*N*-cysteine) conjugate and its *N*-oxide.

In conclusion, excretion occurred mainly in the faeces, the proportion excreted decreasing as the dose decreased. Residues in the milk appeared to indicate that a plateau was reached after 104 hours. The high residues in fat were consistent with the lipophilic nature of the compound. Fipronil-desulfinyl was the principal component of the total radioactive residue in milk and tissues at all doses.

Hens. McCorquodale *et al.* (1996) gave repeated oral doses of [^{14}C]fipronil-desulfinyl in capsules at nominal doses of 0.05, 2 and 10 ppm in the diet (dry matter basis) to three groups of five laying hens. After egg and excreta collection the hens were dosed for 14 days with [^{14}C]fipronil-desulfinyl in the morning before feeding. Three additional hens were the controls. The hens were killed 23 hours after the last dose. The residues in selected tissues and excreta were characterized by chromatography and spectometry, the initial chromatographic analysis being within 6 months. Storage stability was established by radio-HPLC analyses of liver and egg white and yolk extracts at intervals several months apart.

At 0.05, 2 and 10 ppm the mean recoveries of the total administered radioactivity within approximately 23 hours of the last dose were 68%, 79% and 81% respectively. At all doses most of the radioactivity was found in the excreta (Table 11).

Table 11. Mean recoveries of total radioactivity, % of administered dose, from laying hens after repeat oral doses of [^{14}C]fipronil-desulfinyl (McCorquodale *et al.*, 1996).

Sample	^{14}C, % of total administered dose		
	0.05 ppm	2 ppm	10 ppm
Excreta	53	69	71
Cages wash	1.6	1.4	1.2
Egg white	1.9	1.3	1.3
Egg yolk	4.8	2.9	3.6
Tissues	4.0	4.2	6.3

The distribution of the TRR between egg whites and yolks during the study was similar at all doses, and the residues appeared to reach a plateau. Maximum residues in the three groups were 0.005, 0.18, and 0.85 mg/kg fipronil-desulfinyl equivalents in the whites and 0.052, 1.55 and 7.5 mg/kg in the yolks. At the end of the study the highest levels were in omental fat, partially-formed eggs, liver, and skin plus fat (Table 12) and were higher than in plasma. In muscle the residues were lower than in plasma.

Table 12. Mean residues in the tissues and eggs of laying hens after repeat oral doses of [^{14}C]fipronil-desulfinyl (McCorquodale *et al.*, 1996).

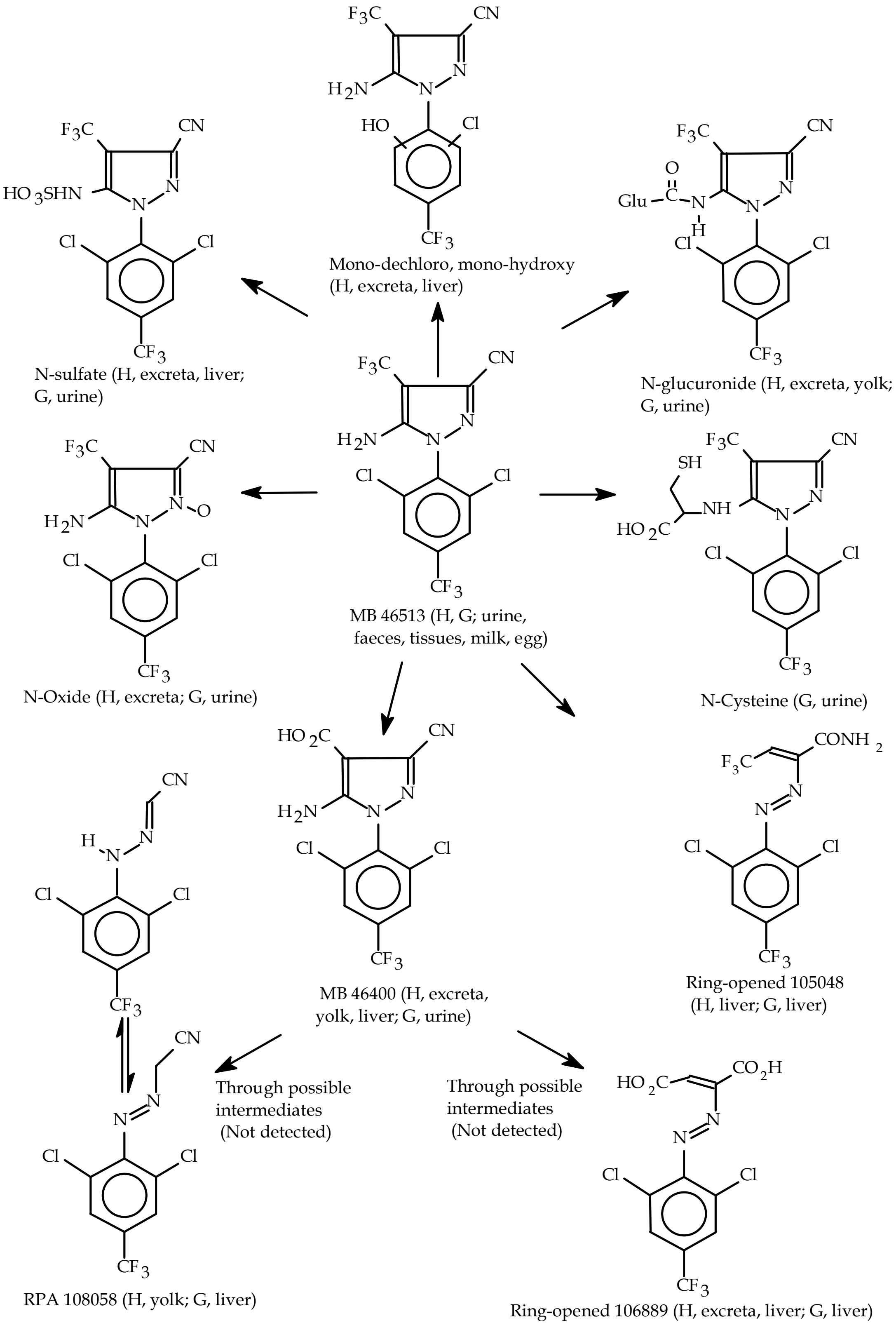
Sample	TRR (mg/kg fipronil-desulfinyl equivalents)		
	0.05 ppm	2 ppm	10 ppm
Eggs partially formed	0.058	1.55	8.7
Omental fat	0.058	1.61	8.8
Breast muscle	0.002	0.056	0.25
Thigh muscle	0.004	0.13	0.6
Liver	0.038	1.02	4.1
Skin plus fat	0.034	0.93	5.8

Samples from the 10 ppm group were extracted for identification of residues. Extraction of ^{14}C was essentially quantitative (>88%) except from one liver sample. Extracts of excreta, egg yolk and white, liver, muscle and fat were examined using radio-HPLC and LC-MS. For metabolite profile comparison, liver and omental fat from the 0.05 ppm group and liver, omental fat, and eggs from the 2 ppm group were analysed by HPLC. The similar chromatographic properties of the components in these extracts to those from the high-dose group indicate that metabolism was the same at all doses.

The combination of radio-HPLC and LC-MS analysis indicated that fipronil-desulfinyl was the only residue in skin and fat, omental fat, and egg white and the main component in excreta, liver, muscle, and egg yolk. Other residues in excreta were tentatively identified as 5-amino sulfate and glucuronide conjugates of fipronil-desulfinyl, MB 46400, a mono-dechloro, monohydroxy derivative of fipronil-desulfinyl, a deaminated ring-opened metabolite of RPA 106889, and the pyrazole *N*-oxide of the parent. In liver, LC-MS indicated the presence of the deaminated ring-opened derivatives of RPA 106889 and RPA 105048. Other unidentified metabolites present at much less than 10% of the TRR were characterized by HPLC retention time as being polar in nature, and thought to be derivatives or conjugates with the pyrazole ring intact. In muscle, three minor unidentified components (<0.02 mg/kg, 1.8-3.9% of the TRR) were detected in addition to fipronil-desulfinyl. In egg yolk, a minor component was tentatively identified as RPA 108058 and additional components characterized as more polar than the parent, probably derivatives or conjugates with the pyrazole ring intact. Proposed metabolic pathways are shown in Figure 3.

In conclusion, after repeated oral administration of [^{14}C]fipronil-desulfinyl to laying hens at doses of 0.05, 2 and 10 ppm, approximately 53 to 71% of the administered radioactivity was eliminated in the excreta. Residues seemed to reach a plateau by the end of the dosing period on the basis of radioactivity measurements in eggs. The elevated radioactive residues in egg yolk, omental fat, and skin plus fat were consistent with the lipophilic nature of the compound. Fipronil-desulfinyl was identified as the principal component of the total radioactive residue in eggs and tissues at all doses.

Figure 3. Proposed metabolic pathways of fipronil-desulfinyl in livestock.



Summary of animal metabolism

The fate of fipronil in mammals was similar in all species studied. It is relatively well absorbed, dependent on dose level and formulation, and extensively distributed in the tissues particularly those with high lipid content. Excretion is mainly via the faeces which contained both free (Phase I) and conjugated (Phase II) metabolites. Both biliary and direct intestinal excretion is involved. Much less is excreted in the urine and urine metabolites are almost exclusively conjugates.

The metabolic pathways of fipronil-desulfinyl in livestock are consistent with those in rats. Fipronil-desulfinyl is metabolized to more polar derivatives or forms polar conjugates which are excreted. The compound is unmetabolized fipronil-desulfinyl is distributed into eggs, milk, and/or tissues, with the highest levels in fat. This is consistent with the lipophilic nature of the molecule. On the basis of these results, it appears that only fipronil-desulfinyl could be transferred to animal substrates in measurable quantities.

Plant metabolism

Soil application

Metabolism by maize, sugar beet, cotton and sunflower was studied after soil application.

Maize. In the first of two trials Yenne and Stone (1994) applied phenyl-labelled [^{14}C]fipronil as a 1.5% granular formulation at 420 g ai/ha (granular proposed use rate 150 g ai/ha), and also at 10 times this rate, and as a stem injection. Both applications were intended to induce higher than normal metabolite levels to facilitate analysis. However results were only reported for plants treated at the lower rate.

The granular formulation was applied to the soil surface before planting, then seeds and granules were covered with approximately 3.8 cm of soil. Plants were harvested at the forage stage (whole green plants, 42 days after treatment) and at maturity (grain at 98 days after treatment and fodder at 106 days after treatment).

The forage contained 0.21 mg/kg fipronil equivalents, fodder 3.7 mg/kg and grain 0.16 mg/kg. The harvested samples contained less than 4.5% of the applied radioactivity (Table 13).

Table 13. Distribution of ^{14}C in field maize after soil treatment with fipronil (Yenne and Stone, 1994).

Sample	^{14}C % of applied	% of TRR	mg/kg as fipronil
Forage	4.0	100	0.21
Mature Plant	4.3		
Fodder	4.0	92.5	3.7
Grain	0.3	7.5	0.16

A series of solvents were used for extraction. Analysis was by ^{14}C-HPLC and MS. 76%, 106% and 99% of the TRR was accounted for in forage, fodder, and grain respectively, after the extractions (including mild acid hydrolysis), distributed as follows. Fipronil (0.08 mg/kg, 39.9% of the TRR), RPA 200766 (0.03 mg/kg, 12.7% of the TRR), and MB 46136 (0.02 mg/kg, 8.7% of the TRR) were in the forage. Two unidentified metabolites at <0.01 mg/kg were also observed. There were seven significant compounds (>0.05 mg/kg) in the fodder, five of which were identified: fipronil (0.45 mg/kg, 12.1% of the TRR), RPA 200761 (0.29 mg/kg, 7.7% of the TRR), RPA 200766 (0.94 mg/kg, 25.3% of the TRR), MB 45950 (0.06 mg/kg, 1.7% of the TRR), and MB 46136 (1.02 mg/kg, 27.6% of the TRR. There were five minor unidentified metabolites at levels of 0.01-0.02 mg/kg. Only one metabolite, a conjugate of RPA 200766, was found in the grain extracts at 0.14 mg/kg (87.5% of the TRR). The results are shown in Table 14.

Table 14. Distribution of fipronil and metabolites in maize tissues (Yenne and Stone, 1994).

Sample	Compound	Residues (mg/kg)	% of TRR[1]
Forage	Fipronil	0.08	39.9
	MB 46136	0.02	8.7
	RPA 200766	0.03	12.7
Fodder	Fipronil	0.45	12.1
	RPA 200761	0.29	7.7
	RPA 200766	0.94	25.3
	MB 45950	0.06	1.7
	MB 46136	1.02	27.6
	2 major unidentified metabolites	0.10 0.20	2.8 5.4
Grain	RPA 200766 conj.	0.14	87.5

[1] Calculated from total residues in individual samples

To establish that fipronil residues in maize had been stable throughout the 3 years of the study and to determine the distribution of residues in maize more clearly, a supplementary study (Yenne and Jesudason, 1995) was conducted. An acetonitrile solution of [^{14}C]fipronil was applied to soil at 146 g ai/ha, and seeds planted and covered with 3.8 cm of soil. Some plants were harvested at the forage stage (whole green plants, 35 days after treatment) and some at maturity (grain at 90 to 106 days after treatment and fodder at 106 days after treatment). Forage samples were cut into 30-cm segments, and plants harvested at maturity were divided into grain, cobs, and 60-cm stalk segments.

The bottom stalk segments of both forage and mature plants contained the highest percentages of radioactivity. Forage from all segments combined contained 0.11 mg/kg fipronil equivalents, fodder 0.51 mg/kg, the cob 0.025 and grain 0.013 mg/kg. At mature harvest the above-ground plant sections contained 0.81% of the radioactivity applied to the soil (Table 15).

Table 15. Distribution of radioactivity in maize plants (Yenne and Jesudason, 1995).

Sample	^{14}C, % of applied	% of TRR	mg/kg as fipronil
Forage	0.45	100	0.11
Mature plant	0.81		
Fodder	0.78	95.1	0.51
Cobs	0.01	1.84	0.025
Grain	0.02	3.09	0.013

Forage, fodder and grain samples were extracted in order to isolate metabolites for identification; cobs were not extracted because of the low level of uptake. Two extraction procedures were compared in order to provide radio-validation of the method of analysis for residue trials. In procedure 1 the first extraction solvent was 100% acetonitrile whereas in procedure 2 it was acetonitrile/water (75/25); subsequent extractants were identical. The second extraction procedure gave more than 96% recovery of the TRR from all tissues. Extracts were analysed by ^{14}C-HPLC followed by MS to identify and confirm metabolites. Fipronil at 0.044 mg/kg (39% of the TRR), RPA 200766 at 0.033 mg/kg (30% of the TRR), MB 46136 at 0.013 mg/kg (11.6% of the TRR), and RPA 200761 at 0.012 mg/kg (10% of the TRR) were identified in the forage. In fodder, fipronil (0.061 mg/kg, 12% of the TRR), RPA 200766 (0.19 mg/kg, 38% of the TRR), MB 46136 (0.082 mg/kg, 16% of the TRR), and RPA 200761 (0.008 mg/kg, 1.6% of the TRR) were also observed, together with an additional minor metabolite RPA 105320 (0.007 mg/kg, 1.4% of the TRR). In grain, only RPA 200766 was identified at 0.008 mg/kg (60% of the TRR).

Table 16. Distribution of ^{14}C residues in maize tissues (Yenne and Jesudason, 1995).

Sample	Compound	Residues (mg/kg)	% of TRR[1]
Forage	Fipronil	0.044	39.1
	MB 46136	0.013	11.6
	RPA 200766	0.033	29.9
	RPA 200761	0.012	10.3
	Unextracted	0.001	0.8
Fodder	Fipronil	0.061	12.1
	MB 46136	0.082	16.2
	RPA 200766	0.194	38.4
	RPA 200761	0.008	1.6
	RPA 105320	0.007	1.4
	Unextracted	0.017	3.4
Grain	RPA 200766	0.008	60.4
	Unextracted	<0.01	0

[1] Calculated from total residues in individual samples.

Fipronil, MB 46136, and RPA 200766 were the main metabolites found in forage and fodder in both the metabolism studies on maize. Only RPA 200766 or its conjugate was found in the grain.

Sugar beet. In a study by Oliver *et al.* (1993) investigating the distribution and metabolism of phenyl ring-labelled [^{14}C]fipronil, the compound was applied with seed at sowing as a 2% granular formulation at the equivalent of 200 g ai/ha and, for the identification of metabolites, at 10 times this rate.

After harvest the leaves and beets were analysed separately; two individual plants were analysed, and the remainder bulked and three replicate samples taken. Sufficient activity was present at 200 g ai/ha for analysis so only these results are reported.

Residues were isolated by sequential acetone, methanol/water, and Soxhlet extractions followed by acid digestion. The leaves contained residues equivalent to a mean of 92% of the TRR, of which 86-91% (0.57-0.59 mg/kg) was extractable. Mild and strong acid hydrolysis released 1.5% to 3.2% (0.01 to 0.021 mg/kg). In the root tissue, 80% to 91% of the activity (0.037 to 0.061 mg/kg) was released after extractions with acetone and methanol/water only. Metabolites were identified by co-chromatography against standards and GC-MS.

In the leaves, 85% to 88% of the extracted activity was in the methanol extract. The residue comprised MB 46136 (0.18 to 0.23 mg/kg) the main component, followed by RPA 105320 (0.11 to 0.12 mg/kg), MB 45950 (0.017 to 0.029 mg/kg), and MB 45897 (0.01 to 0.019 mg/kg). Low levels of fipronil were detected by GC-MS. Polar material (0.087 to 0.09 mg/kg) was shown to consist of at least four components by reverse phase TLC.

In the beets, organosoluble material accounted for 96.7% to 98% of the extracted activity. MB 46136 (0.028 to 0.053 mg/kg) was again the main component. Fipronil (0.005 to 0.012 mg/kg) and RPA 200766 (0.002 to 0.003 mg/kg) were also found. Low levels of MB 45950 were detected by GC-MS (Table 17).

Table 17. Distribution of ^{14}C residues in sugar beet (Oliver *et al.*, 1993).

Sample	Compound	mg/kg as fipronil[1]
Leaves	Fipronil	NA[2]
	MB 46136	0.19
	MB 45950	0.024[3]
	MB 45897	0.011
	RPA 105320	0.12

Sample	Compound	mg/kg as fipronil[1]
	RPA 200766	0.025
	4 polar components	total 0.088
Beet	Fipronil	0.009
	MB 46136	0.033
	MB 45950	NA[2]
	RPA 200766	0.003

[1] Mean of replicates
[2] NA: not analysed. Observed at low levels by GC-MS
[3] Not confirmed by GC-MS

Cotton. Yenne and Stone (1995) investigated the metabolism of phenyl ring-labelled [^{14}C]fipronil in and on cotton plants after soil incorporation (a further study on foliar application is described later).

An in-furrow treatment at planting of an acetonitrile solution of [^{14}C]fipronil was applied to the cotton at approximately 224 g ai/ha. Plants were harvested at maturity (plants dessicated, seeds hard, 140 days after planting) and the above-ground parts separated into seed, lint, bolls and foliage.

Only 2.3% of the applied radioactivity was found in the above-ground plant parts. Foliage contained 2.2 mg/kg fipronil equivalents (more than 97% of the TRR in the plant at harvest), bolls 0.16 mg/kg, lint 0.02 and seed <0.01 mg/kg respectively.

Table 18. Distribution of ^{14}C in cotton plants (Yenne and Stone, 1995).

Sample	% of applied ^{14}C	% of TRR	mg/kg as fipronil
Foliage	2.29	97.4	2.17
Bolls	0.05	2.1	0.16
Lint	0.3	0.3	0.02
Seed	0.1	0.1	<0.01

More than 76% of the TRR was extracted with a sequence of organic solvents followed by acid and base digestion from the foliage and bolls. Lint was digested with cellulase; insufficient radioactivity was present for identification of metabolites. Seed was not extracted, as it contained <0.01 mg/kg fipronil equivalents.

Extracts were analysed by radio-HPLC followed by MS. In the foliage fipronil was determined at 0.01 mg/kg (0.3% of the TRR), and the metabolites RPA 200766 at 0.9 mg/kg (41.5% of the TRR), MB 46136 at 0.15 mg/kg (6.9% of the TRR), and RPA 200761 at 0.17 mg/kg (8.1% of the TRR). Two unknown metabolites (<0.05 mg/kg) were more polar than fipronil and its metabolites on the basis of partitioning and retention time characteristics. An additional 12 metabolites were detected at <0.01 mg/kg. No MB 45950 was determined. In bolls, fipronil (0.02 mg/kg, 12% of the TRR) and RPA 200766 (0.03 mg/kg, 21% of the TRR) were identified; and two other metabolites, more polar than fipronil, at <0.01 mg/kg (Table 19). The results indicate that measurable residues in cotton seed would be unlikely.

Table 19. Radioactive residues in cotton plants (Yenne and Stone, 1995).

Sample	Compound	Residues (mg/kg as compound, not as fipronil)	% of TRR[1]
Foliage	Fipronil	0.01	0.3
	MB 46136	0.15	6.9
	RPA 200766	0.90	41.5
	RPA 200761	0.17	8.1
Bolls	Fipronil	0.02	12
	RPA 200766	0.03	21

[1] Calculated from total residues in individual samples

<u>Sunflower</u>. In a study by Bellet *et al.* (1993) [^{14}C]fipronil (phenyl-labelled) was applied in furrow with seed at sowing as a 2% granular formulation at 200 g ai/ha, the intended use rate. Plants were sampled at intervals and at harvest. The TRR was measured in the aerial parts of the pre-harvest samples, and the leaves, trunk, heads and seeds taken at harvest were analysed separately.

Samples one month after planting showed absorption of 1.3% of the applied ^{14}C (0.17 mg/kg fipronil equivalents) and at harvest contained 4.8% in total. The distribution of the ^{14}C within the plants at harvest is shown in Table 20. More than 80% of the radioactivity was in the leaves.

Table 20. Distribution of radioactivity in sunflowers at harvest (Bellet *et al.*, 1993).

Sample	% of applied ^{14}C	% of TRR	mg/kg as fipronil
Leaves	4.0	83	1.4
Trunk	0.6	13	0.13
Head	0.08	1.7	0.033
Seed	0.12	2.9	0.034

Residues were isolated by sequential acetone, methanol/water, and Soxhlet extractions followed by digestion of tissues with acid. Extractability of radioactivity from the four substrates ranged from 83 to 95%. Metabolites were identified by co-chromatography with standards and GC-MS.

The main residue in the leaves was unchanged fipronil (0.48 mg/kg; 30% of the TRR). MB 46136 was a major metabolite (0.22 mg/kg, 14% of the TRR) together with RPA 200766 (0.11 mg/kg; 7.1% of the TRR). Numerous minor metabolites were found but none exceeded 0.05 mg/kg. The trunk also contained unchanged fipronil and MB 46136 was a main metabolite. Again, no other metabolites were identified which exceeded 0.01 mg/kg. The head contained no components above 0.01 mg/kg. The metabolite pattern in the seeds (which contained 0.029 mg/kg extractable ^{14}C, 2.7% of the TRR) differed in that extracts contained 14 components, all at levels below 0.01 mg/kg. Fipronil and closely related metabolites were not present in the seeds (Table 21).

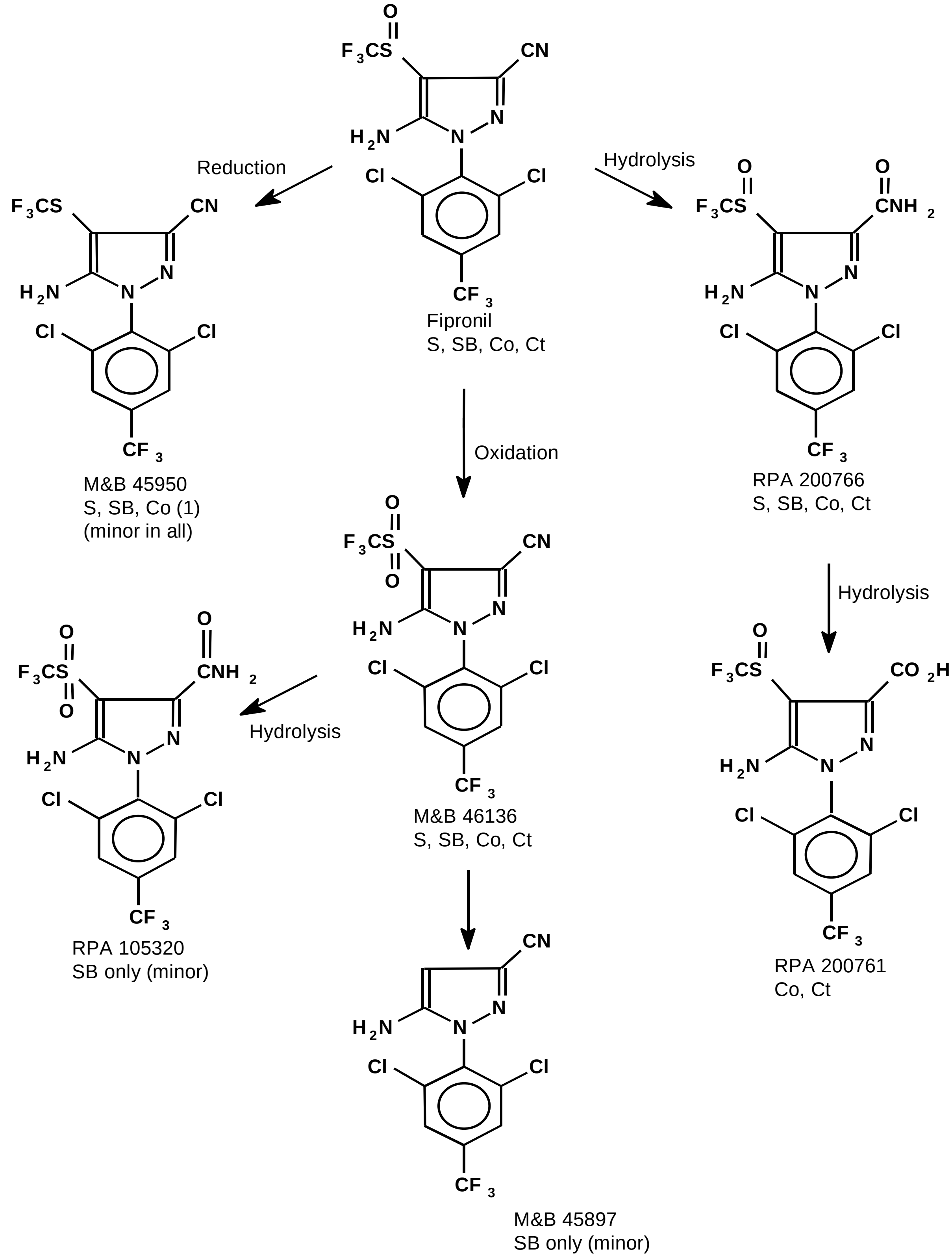
Table 21. Distribution of fipronil and metabolites in sunflower plants (Bellet *et al.*, 1993).

Sample	Compound	Residue (mg/kg)	% of total residue
Leaves			
Organic extract	Fipronil	0.48	30
	MB 46136	0.22	14
	MB 45950	0.04	2.6
	RPA 200766	0.11	7.1
	6 unknowns	0.015 to 0.045, total 0.16	9.9
Acid fraction	5 components	each <0.01, total 0.017	1.1
Trunk	Fipronil	0.034	4.3
	MB 46136	0.12	1.5
	2 unknowns	each <0.01, total 0.01	1.2
	polar (baseline) compound(s)	total 0.019	2.4
Head	multiple components	each <0.01, total 0.021	1.5
Seed	14 unknowns	each <0.01, total 0.029	2.7

In summary three pathways of metabolism of fipronil in plants after application by soil incorporation were identified:

(1) reduction to sulfide MB 45950, a minor pathway observed in some plants
(2) oxidation to MB 46136
(3) hydrolysis to amide RPA 200766. Further hydrolysis of metabolites RPA 200766 and MB 46136 to metabolites RPA 200761 and RPA 105320 also occurs in some plant tissues.

The proposed metabolic pathways are shown in Figure 4.



C: corn (maize), Co: cotton, S: sunflower, SB: sugar beet.

Figure 4. Plant metabolism after soil incorporated application.

Foliar application

Rice. In a study by Cooper *et al*. (1994) phenyl ring-labelled [^{14}C]fipronil was applied to transplanted rice plants as either a granular treatment or as a foliar spray.

Seedlings were transplanted into plots resembling paddy-fields (Thailand) with water levels between 9 and 15 cm. In two plots a 0.3% granule formulation was broadcast at 50 g ai/ha 20 days after the seedlings had been transplanted. In another two plots, the plants were sprayed twice with a 5% suspo-emulsion of fipronil at 20 and 50 days after being transplanted at 50 g ai/ha per treatment. There were also two control plots. The plots were maintained according to GAP for rice growing in Thailand.

Plants from all six plots were randomly sampled 51 days after transplanting (1 day after the second foliar spray and 31 days after the single granular treatment). At harvest, 92 days after transplanting, root, straw and ear samples were processed to provide pannicle, husk, brown rice, polished rice, and bran. The TRR was measured in all the samples. The results demonstrated that fipronil applied either as a granule or foliar application was absorbed and distributed throughout the whole plant. The residues at harvest after the granule application were lower than those in the sprayed plants (Table 22).

Table 22. Radiolabelled residues in rice plants at harvest, 72 days after granular treatment and 42 days after the second foliar treatment (Cooper *et al*., 1994).

Sample	^{14}C, mg/kg as fipronil					
	Foliar application			Granule application		
	Plot 1	Plot 3	Mean	Plot 2	Plot 5	Mean
Root	0.109	0.092	0.101	0.076	0.055	0.065
Straw	0.248	0.266	0.257	0.111	0.086	0.099
Pannicle	2.046	2.145	2.096	0.337	0.315	0.326
Husk	0.495	0.545	0.520	0.075	0.073	0.074
Bran	0.155	0.128	0.142	0.022	0.022	0.022
Brown Rice	0.025	0.023	0.024	0.005	0.006	0.006
Polished Rice	0.012	0.014	0.013	0.004	0.004	0.004
Total[1]	0.28	0.3	0.29	0.1	0.078	0.089

[1] Brown rice: polished rice + bran. Results for polished rice and bran, but not brown rice, have therefore been included in the total

74%-94% of the TRR was extractable. Extracts were analysed by ^{14}C-HPLC and by MS. Fipronil was present in all samples and no MB 46136 was detected in any. The photodegradation product fipronil-desulfinyl was detected only in samples from the foliar-treated plots (brown rice 0.45 μg/kg, polished rice 0.19 μg/kg), as was MB 45897. RPA 200766 was detected in all rice samples but at higher levels in samples from the granule-treated plots. Low levels of MB 45950 and RPA 104615 were present in all samples. The results are shown in Table 23.

Table 23. Residues in brown and polished rice (Cooper *et al*., 1994).

Indicated identity	Residue, μg/kg as compound, not as fipronil, and (% of TRR)			
	Brown rice		Polished rice	
	Granule	Foliar	Granule	Foliar
Fipronil	1.3 (25.4%)	12.3 (51.6%)	0.72 (17.6%)	4.97 (38%)
MB 46136	ND[2]	ND	ND	ND
MB 45950	0.21 (4.1%)	1.02 (4.3%)	0.21 (5.1%)	0.36 (2.8%)
fipronil-desulfinyl	ND	0.45 (1.9%)	ND	0.19 (1.5%)
MB 45897	ND	0.85 (3.6%)	ND	0.61 (4.7%)
RPA 200766	0.62 (12.1%)	2.27 (9.5%)	0.93 (22.8%)	1.81 (13.8%)

Indicated identity	Residue, µg/kg as compound, not as fipronil, and (% of TRR)			
	Brown rice		Polished rice	
	Granule	Foliar	Granule	Foliar
RPA 104615	0.11 (2.1%)	0.43 (1.8%)	0.1 (2.5%)	0.45 (3.4%)
Unascribed[1]	1.56 (30.5%)	2.18 (9.1%)	1.13 (27.7%)	2.46 (18.8%)
Unextracted	1.36 (26.6%)	4.6 (19.3%)	1.06 (26.0%)	2.54 (19.4%)

[1] Unascribed: radioactivity that could not be assigned to discrete peaks on the chromatogram
[2] ND: not detectable

To investigate the unextracted residue, brown rice samples were treated with methanol and ammonia, then refluxed with methanol and increasing concentrations of potassium hydroxide. Approximately 82% of the residual 20% was released, about 56% of which was partitioned into dichloromethane; the remaining 26% did not. It was concluded that most of the unextracted residue was not incorporated into natural products but consisted of fipronil and/or metabolites chemically bound to varying degrees.

A number of metabolites were detected in the granule- and foliar-treated green plant samples and harvested plant parts (excluding rice) (Table 24).

Table 24. Residues in granule- (GR) and foliar-treated plants (Cooper *et al*., 1994).

Interval	Sample and application		Residue, mg/kg as compound, not as fipronil					
			fipronil	MB 45950	MB 46136	Fipronil-desulfinyl	MB 45897	RPA 200766
51 DAT	Root	GR	0.01	0.013	0.004	0.008	0.001	0.001
		Foliar	0.096	0.015	0.005	0.016	0.001	0.001
	Plant	GR	0.008	0.003	0.003	0.01	<0.001	0.001
		Foliar	0.27	0.13	0.013	0.053	0.004	0.006
Harvest	Root	GR	0.004	0.016	0.010	0.01	ND	ND
		Foliar	0.025	0.013	0.015	0.019	ND	0.002
	Straw	GR	0.012	0.015	0.017	0.023	ND	0.005
		Foliar	0.1	0.023	0.029	0.047	ND	0.004
	Pannicle	GR	0.027	0.12	0.029	0.24	0.005	0.006
		Foliar	0.48	0.24	0.1	0.57	0.019	0.018
	Husk	GR	0.015	0.037	0.004	0.017	ND	0.006
		Foliar	0.11	0.027	0.021	0.053	0.006	0.013
	Bran	GR	0.004	0.003	ND	0.006	ND	<0.001
		Foliar	0.065	0.011	0.007	0.018	0.001	0.005

DAT: days after treatment
ND: not detectable

The number and nature of the metabolites demonstrated that rice plants are metabolized extensively by oxidative as well as reductive mechanisms, and the distribution throughout the plants also demonstrated that fipronil and its metabolites can be translocated.

Cabbage. In a study by Lowden and French (1995) HISPI cabbages grown from seed were foliar-sprayed twice with [^{14}C]fipronil (phenyl-labelled), once at the onset of heart formation and again 14 days later, at a rate equivalent to 200 g ai/ha per treatment.

Two treated plants were sampled 0, 17, 21, 24 and 28 days after the first spray, and four taken 14 days after the first dose (two before and two after the second dose: 14a and 14b). The remaining plants were harvested 35 days after the first spray. Control plants were sampled 0, 14, 28 and 35 days after the first spray. The TRR was measured in all samples, which were sequentially extracted with acetonitrile/water (1/1), acetonitrile, and hexane and the residues identified by HPLC, MS, and NMR.

A summary of the results is shown in terms of concentration in Table 25 and in percentage of the TRR in Table 26.

Table 25. Concentrations of fipronil and metabolites in cabbages, expressed as mg/kg fipronil equivalents (Lowden and French, 1995).

Component	Days after application									
								35 (Harvest)		
	0	14a	14b	17	21	24	28	Total	Leaves	Heart
Fipronil	1.98	0.53	1.65	0.91	0.83	0.54	0.57	0.66	1.08	0.38
MB 46136	0	0	0	0.01	0.01	0.01	0	0.01	0.03	0
MB 45950	0	0	0	0	0.03	0	0.01	0	0	0
fipronil-desulfinyl	0	0.14	0.3	0.21	0.22	0.21	0.2	0.17	0.35	0.03
RPA 200766	0	0	0	0.03	0.05	0.05	0	0.16	0.29	0.06
RPA 104615	0	0.05	0	0.05	0.04	0.04	0.15	0.21	0.4	0.08
Bound	0	0.06	0.1	0.08	0.08	0.09	0.1	0.07	0.13	0.03

Table 26. Fipronil and metabolites in cabbages, expressed as % of TRR (Lowden and French, 1995).

Component	Days after application									
								35 (Harvest)		
	0	14a	14b	17	21	24	28	Total	Leaves	Heart
Fipronil	100	68	81	71	66	57	55	52	47	66
MB 46136	0	0	0	0.8	0.8	1.1	0	0.8	1.3	0
MB 45950	0	0	0	0	2.4	0	1.0	0	0	0
fipronil-desulfinyl	0	18	15	16	17.5	22	19.4	13	15	5.2
RPA 200766	0	0	0	2.3	4.0	5.3	0	12.5	13	10
RPA 104615	0	6.4	0	3.9	3.2	4.3	15	16	18	14
Bound	0	7.7	4.9	6.2	6.3	9.6	9.7	5.5	5.7	5.2

This study demonstrated that the photodegradation products of fipronil, fipronil-desulfinyl and RPA 104615 are two of the main degradation products of fipronil found in cabbage plants after foliar application. At harvest (35 days after first application), these compounds accounted for 13% and 16% of the TRR respectively. RPA 200766, the amide metabolite, was also a major product (13% of the TRR at harvest).

Potatoes. In a study by Mislankar (1995) plants were sprayed twice with [^{14}C]fipronil at 112 g ai/ha/application (season rate) or 560 g ai/ha/application (5 times the season rate), the first 7 weeks and the second 10 weeks after planting, 28 days before harvest. Potatoes were harvested as they matured and tubers composited. The TRR was determined in the vines and potatoes at both rates.

Table 27. Total radioactive residues in potato plants (Mislankar, 1995).

Sample	Rate (g ai/ha)	% of TRR	mg/kg as fipronil
Potato	2 x 112	1.05	0.005
Vine	2 x 112	99	0.47
Potato	2 x 560	0.54	0.02
Vine	2 x 560	99.5	3.7

Only tubers, the edible portions of the plants, were extracted. As the TRR in the potatoes sprayed at the low rate was only 0.005 mg/kg as fipronil, only extracts from the potatoes sprayed at the high rate were analysed.

Sequential solvent extractions extracted almost 95% of the TRR (0.019 mg/kg) from the high-rate tubers. Analysis was by HPLC with LSC; all metabolites were below 0.005 mg/kg. Metabolites were identified by HPLC retention time compared to standards and confirmed by mass spectrometry.

Four metabolites were identified in the tubers: fipronil at 0.0034 mg/kg (18% of the TRR), MB 46136 at 0.0013 mg/kg (6.6% of the TRR), fipronil-desulfinyl at 0.003 mg/kg (16% of the TRR), MB 45897 at 0.0012 mg/kg (6.4% of the TRR), and RPA 104615 at 0.0018 mg/kg (9.6% of the TRR). Five unknown metabolites, all below 0.002 mg/kg, accounted for a total of 0.005 mg/kg. Trace amounts of MB 45950, RPA 200766, and RPA 200761 were detected by MS only.

Table 28. Fipronil and its metabolites in potato tubers (Mislankar, 1995).

Sample	Compound	Residue (mg/kg as compound, not as fipronil)	% of TRR[1]
Tuber (5 times rate)	Fipronil	0.0034	18
	MB 46136	0.0013	6.6
	fipronil-desulfinyl	0.003	15.8
	MB 45897	0.0012	6.4
	RPA 104615	0.0018	9.6
	5 compounds	all <0.002, total 0.005	26

[1] Calculated from total residues in individual samples

The residue levels at the fivefold rate indicate that fipronil and its metabolites are poorly translocated from the foliage to the tubers, and therefore there should not be measurable residues in tubers at the proposed rate.

After foliar application to potatoes, fipronil is oxidized to the sulfone MB 46136 or photolysized to fipronil-desulfinyl. The sulfone undergoes further transformation to the sulfonic acid RPA 104615 followed by loss of the sulfonic acid group to form MB 45897.

Cotton. In a study by Yenne and Stone (1995) an acetonitrile solution of [^{14}C]fipronil was sprayed twice on cotton foliage at 112 g ai/ha/application, the second time at the soft-boll stage 49 days before harvest. The mature dessicated plants with hard seeds were harvested 140 days after planting and the above-ground portion of the plants separated into seed, lint, bolls and foliage. The TRR was determined in all tissues.

The foliage contained 8.3 mg/kg fipronil equivalents (97% of the TRR in the plant at harvest), bolls 0.81 mg/kg, lint 0.1 and seed <0.01 mg/kg.

Table 29. Distribution of radioactivity in cotton plants (Yenne and Stone, 1995).

Sample	^{14}C, % of TRR	Residue (mg/kg as fipronil)
Foliage	97	8.3
Bolls	2.7	0.81
Lint	0.3	0.1
Seed	0	<0.01

Cotton foliage and bolls were extracted with a sequence of organic solvents followed by acid and base digestion. More than 90% of the TRR was extractable. Lint was digested with cellulase; seed was not extracted as it contained <0.01 mg/kg fipronil equivalents.

Foliage, boll extracts and digested lint were analysed by ^{14}C-HPLC and TLC followed by MS to identify and confirm metabolites. The compounds in the foliage were fipronil at 4.4 mg/kg (53% of the TRR), MB 46136 at 0.55 mg/kg fipronil equivalents (6.6% of the TRR), RPA 200761 at 1.3 mg/kg (15.5% of the TRR), and fipronil-desulfinyl at 0.01 mg/kg (0.1% of the TRR), two unknown significant metabolites (0.05 mg/kg) which were more polar than fipronil and the identified metabolites from their partitioning and retention time characteristics, and an additional 6 metabolites at 0.01 mg/kg. Trace amounts of MB 45950, RPA 105320, and RPA 200766 were detected by LC-MS only. In bolls, the same main metabolites were identified, but in different proportions: fipronil (0.52

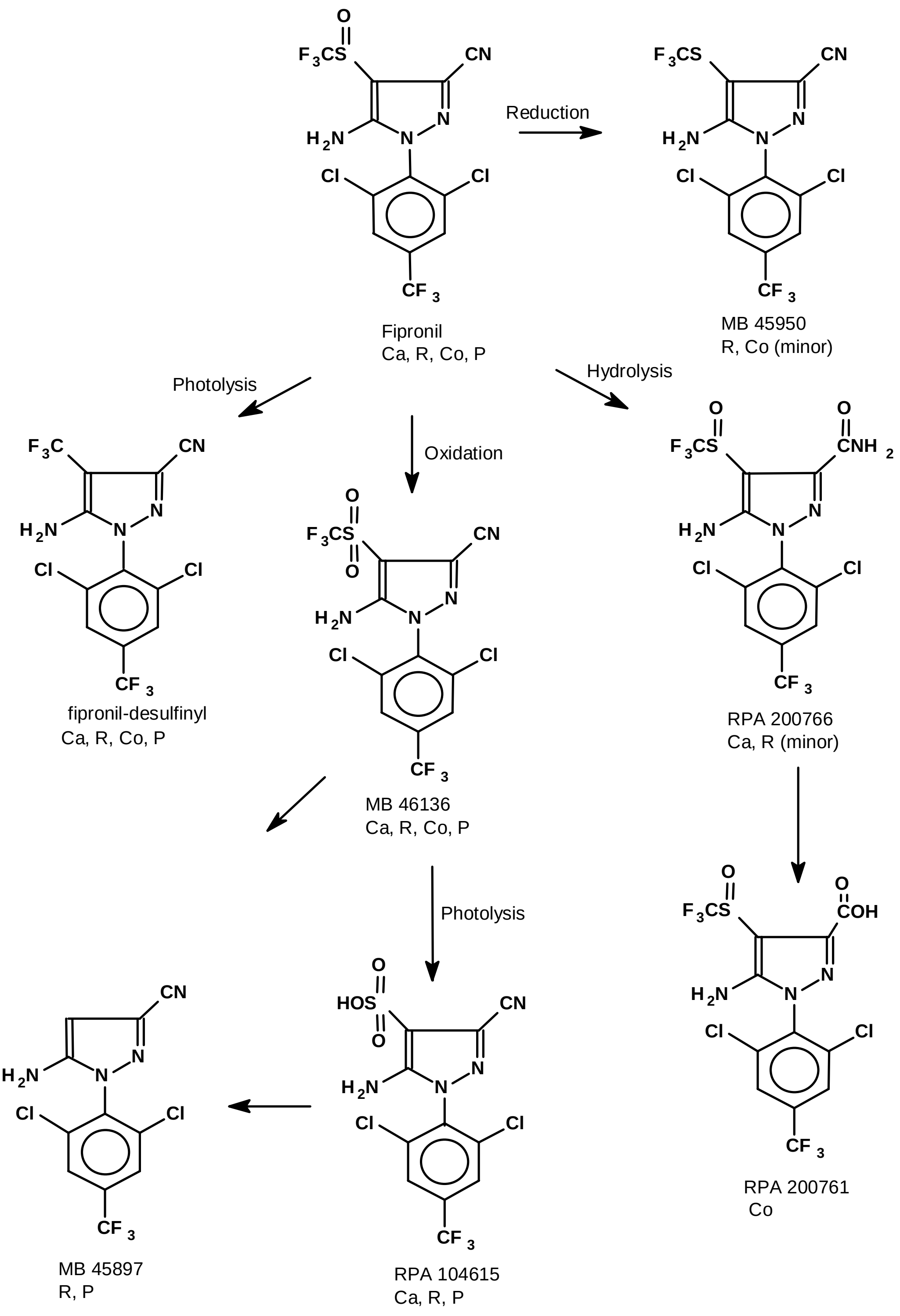
mg/kg, 64% of the TRR), MB 46136 (0.11 mg/kg, 14% of the TRR), RPA 200761 (<0.01 mg/kg, 0.3% of the TRR), and fipronil-desulfinyl (0.01 mg/kg, 0.9% of the TRR). Lint contained only 2 metabolites above 0.01 mg/kg, fipronil and MB 46136. Low levels (<0.01 mg/kg) of fipronil-desulfinyl, RPA 200761, and MB 45950 were also confirmed; 5 additional unknowns were observed, all at levels below 0.01 mg/kg (Table 30).

Table 30. Radioactive residues in cotton plants (Yenne and Stone, 1995).

Sample	Compound	mg/kg as fipronil	% of TRR
Foliage	Fipronil	4.4	53
	MB 46136	0.55	6.6
	RPA 200761	1.3	15.5
	fipronil-desulfinyl	0.01	0.1
	Polar unknowns	0.18	2.2
	6 compounds	all <0.01	total 0.8
Bolls	Fipronil	0.52	64
	MB 46136	0.11	14
	RPA 200761	<0.01	0.3
	fipronil-desulfinyl	0.01	0.9
Lint	Fipronil	0.05	48
	MB 46136	0.11	14
	RPA 200761	<0.01	1.2
	fipronil-desulfinyl	<0.01	2.0
	MB 45950	<0.01	4.2
	5 compounds	all <0.01	total 14

The metabolic pathways in plants after foliar application of fipronil may be summarized as follows. In addition to the formation of metabolites *via* oxidation (MB 46136), reduction (MB 45950) and hydrolysis (RPA 200766 and RPA 200761), the photodegradation products fipronil-desulfinyl and RPA 104615 have been shown to be terminal residues. Cleavage of the sulfonic acid group from RPA 104615 can also occur to give MB 45897 (minor). Fipronil, MB 46136 and fipronil-desulfinyl were the main compounds observed in all studies. Proposed metabolic pathways in foliar-treated plants are shown in Figure 5.

Figure 5. Metabolic pathways in plants after foliar applications.



Ca: Cabbage; R: Rice; Co: Cotton; P: Potato

Plant metabolism – summary of results

Soil incorporated applications. The metabolism of fipronil was studied in sunflowers, sugar beet, field maize, and cotton after the application of fipronil to soil with incorporation. Formulations, rates, and samples analysed are shown in Table 31.

Table 31. Summary details of soil-incorporated fipronil metabolism studies.

Crop	Method of application	Time of application	Rate (g ai/ha)	Sample analysed	Sampling time
Sunflower	Granule	at planting	200	Leaves, trunk, head, seeds	at harvest
Sugar beet	Granule	at planting	200	Beet, leaves	at harvest
Field maize	a) Granule b) Soil spray	at planting	a) 420 b) 146	Green forage Fodder, grain	a) 42 DAP[1] at harvest b) 35 DAP at harvest
Cotton	Soil spray	at planting	224	Foliage, bolls, lint, seed	at harvest

[1] Days after planting

In all studies the samples indicated in the above table were analysed quantitatively for radioactivity, and subjected to solvent extraction followed by extract analysis to give qualitative information. Methods of analysis included TLC, HPLC-MS and GC-MS.

Measurement of radioactivity showed that the uptake of soil-applied fipronil into plants is low (less than 5% based upon total radioactivity measured in whole plants at harvest). Analysis of extracts from maize forage samples revealed fipronil, the sulfone MB 46136 and the amide RPA 200766 as the main metabolites in both studies; RPA 200761 was also found in the second study. In harvest samples, sugar beet and sunflower leaves as well as maize fodder and cotton foliage contained the metabolites MB 46136 and RPA 200766 and varying amounts of the parent compound. RPA 105320 and MB 45897 were identified only in beet leaves; RPA 200761 was identified in maize fodder and cotton foliage.

In edible plant parts, MB 46136 and RPA 200766 were present in sugar beet. In maize grain only RPA 200766 was found. Sunflower seed extracts contained a complex mixture of substances dissimilar to those in the leaves; a number of components each representing <0.01 mg/kg were separated. Cotton seed was not analysed as it contained less than 0.01 mg/kg of TRR. The distribution of identified compounds in all the studies is shown in Table 32.

Table 32. Compounds identified in soil-applied fipronil metabolism.

TRR, mg/kg as fipronil	Fipronil	MB 45897	MB 45950	MB 46136	RPA 105320	RPA 200761	RPA 200766
Sunflower							
Leaves 1.43	M	--	vm	M	--	--	m
Seed 0.034 [1]	--	--	--	--	--	--	--
Sugar beet							
Leaves 0.45	MSO	vm	vm, NC	M	M	--	vm
Root 0.05	M	--	MSO	M	--	--	m
Maize-1							
Forage 0.21	M	--	--	m	--	--	M
Fodder 3.7	M	--	vm	M	--	m	M
Grain 0.16	--	--	--	--	--	--	M
Maize-2							
Forage 0.11	M	--	--	M	--	M	M
Fodder 0.51	M	--	--	M	vm	vm	M
Grain 0.01	--	--	--	--	--	--	M
Cotton							
Foliage [2] 2.33	vm	--	--	m	--	m	M
Lint 0.02 [3]	--	--	--	--	--	--	--

TRR, mg/kg as fipronil	Fipronil	MB 45897	MB 45950	MB 46136	RPA 105320	RPA 200761	RPA 200766
Seed <0.01 [3]	--	--	--	--	--	--	--

M: major, >10% of TRR; m: minor, 5-10% of TRR; vm: very minor, <5% of TRR
NC: not confirmed by MS; MSO: observed by MS only
[1] 14 components, all <0.01 mg/kg
[2] Including bolls
[3] Not analysed

In summary, for soil-incorporated uses of fipronil, identification of residues in plant tissues shows that the metabolism proceeds mainly by oxidation to sulfone MB 46136 and hydrolysis to amide RPA 200766. Further hydrolysis of these metabolites can also occur. Very small amounts of sulfide MB 45950 can occur by reduction, but in no case was it >5% of the total radioactive residue.

Foliar applications. The metabolism of [^{14}C]fipronil has been studied after foliar spray application to cabbages, rice, cotton, and potatoes. The timing rates of application, and samples analysed at harvest are shown in Table 33.

Table 33. Summary details of foliar-applied fipronil metabolism studies.

Crop	Time of application	Rate (g ai/ha)	Sample analysed	Sampling
Cabbage	First spray at onset of heart formation; 14 day spray interval	2 x 200	Leaves, heart	21 DALT[1]
Rice[2]	First spray 20 days after transplant; 30 day spray interval	2 x 50	Straw, panicle, husk, bran, brown rice, polished rice	42 DALT
Cotton	First spray pre-first bloom; second spray at soft boll stage	2 x 112	Foliage, bolls, lint, seed	49 DALT
Potato	First spray to immature foliage, 7 weeks after planting; 21 day spray interval	2 x 560[3]	Tuber	28 DALT

[1]Days after last treatment
[2]The study also included a granular application to the rice paddy 20 days after the transplant of rice; only data from the foliar portion of the study are reported.
[3]Rate applied is 5 times proposed use rate to allow identification of metabolites.

Radioactive residues were quantified in all samples. Organic solvents were used for extraction and extracts were used for identification. Methods of analysis were similar to those for the extracts after soil-incorporation.

In addition to the formation of previously known fipronil metabolites from oxidation (MB 46136), reduction (MB 45950) and hydrolysis (RPA 200766, RPA 200761), photodegradation products fipronil-desulfinyl and RPA 104615 have been identified as possible terminal residues after foliar application of fipronil. The main residues are consistently parent and fipronil-desulfinyl; lesser amounts of MB 45950 and MB 46136 can also form. Table 34 shows the distribution of identified compounds in all the studies.

Table 34. Compounds identified in foliar-applied fipronil metabolism.

Sample and TRR, mg/kg as fipronil	Compound							
	Fipronil	MB 45897	MB 45950	MB 46136	fipronil-desulfinyl	RPA 104615	RPA 200761	RPA 200766
Cabbage								
Whole plant 1.28	M	--	--	vm	M	M	--	M
Cotton								
Foliage[1] 9.13	M	--	--	m	vm	--	M	--
Lint 0.1	M	--	vm	M	vm	--	vm	--
Seed[2] <0.01	--	--	--	--	--	--	--	--

Sample and TRR, mg/kg as fipronil	Compound							
	Fipronil	MB 45897	MB 45950	MB 46136	fipronil-desulfinyl	RPA 104615	RPA 200761	RPA 200766
Potato								
Tuber 0.021[3]	M	m	MSO	m	M	m	MSO	--
Rice								
Straw 0.26	M	--	M	M	M	--	--	vm
Panicle 2.1	M	vm	M	m	M	--	--	vm
Husk 0.52	M	vm	M	m	M	--	--	m
Bran 0.14	M	vm	M	m	M	--	--	vm
Brown rice 0.024	M	vm	vm	--	vm	vm	--	m
Polished rice 0.013	M	vm	vm	--	vm	vm	--	M

M: major, >10% of TRR; m: minor, 5-10% of TRR; vm: very minor, <5% of TRR
MSO: observed by MS only
[1] Including bolls
[2] Not analysed, TRR <0.01 mg/kg
[3] 5-times rate; single rate TRR was only 0.005 mg/kg

Environmental fate in soil

Photolysis

The photolytic degradation of [^{14}C]fipronil after surface application to a clay-loam soil has been studied by Burr and Austin (1992). Soil at 75% of its 1/3 bar moisture holding capacity was treated with [^{14}C]fipronil at a rate equivalent to 0.25 kg ai/ha.

All soil samples were incubated under aerobic conditions. Control samples were maintained in the dark; test samples were irradiated with a xenon lamp, filtered to provide light in the wavelength range 290-800 nm. An 8/16 h light/dark irradiation cycle was used to simulate natural sunlight equivalent to a typical day in Florida, USA, and samples were taken after 0, 3, 7, 14, 21 and 30 days. The extracts were analysed by thin-layer and high-performance liquid chromatography.

The nature and number of degradation products found differed between the control and irradiated samples. In the control sample extracts, the metabolites MB 45950, MB 46136 and RPA 200766 were identified and by day 30 all were at approximately equal proportions of the applied nominal dose, about 10%. The remainder of the radioactivity was identified as being from fipronil.

In the irradiated samples the products MB 45950, MB 46136, RPA 200766, fipronil-desulfinyl and RPA 104615 were identified. The proportion of RPA 200766 was very similar in the control and irradiated samples, indicating that its production is not related to photolytic degradation. MB 45950 was only a very minor product under irradiation, <2% by day 30. The two photoproducts RPA 104615 and fipronil-desulfinyl each accounted for about 7% of the applied nominal dose and were not found in the control samples.

Fipronil was degraded rapidly with and without irradiation with half-lives of about 49 and 34 days respectively. Irradiation yielded RPA 104615 and fipronil-desulfinyl which were also observed in an aqueous photolysis study described below but not in the control samples in either study.

Aerobic degradation

The degradation of [^{14}C]fipronil applied at 200 g ai/ha to a sandy loam soil in Manningtree, UK, and Speyer 2.2 soil in Germany over a 336-day period was studied by Waring (1993). Key characteristics of the soil were as follows.

Soil	Textural class	Cation exchange capacity (me/100g)	Organic matter (%)	pH
Speyer 2.2	Sand	3.3	3.3	6.1
Manningtree Soil	Sandy loam	6.4	1.7	7.8

Aliquots of the soils (50 g dry weight equivalent) were incubated in crystallizing dishes housed in glass chambers. Moistened CO_2-free air was drawn through each chamber before being passed through various traps to collect polar and non-polar volatiles and $^{14}CO_2$. Duplicates of each soil were sampled at intervals of 0, 1, 3, 7, 14, 30, 41, 80, 149, 252 and 336 days after treatment.

Initially, [^{14}C]fipronil accounted for >98% of the applied radioactivity. Over the study period, the amount of parent compound decreased to 12-20% in the Manningtree and 44%-46% in the Speyer soil.

RPA 200766 and MB 46136 were the main degradation products, accounting for a maximum of 38% and 24% of the applied radioactivity respectively in Manningtree and 27% and 14% in Speyer 2.2 soil. Smaller quantities of MB 45950 (<5%) and the photodegradation product fipronil-desulfinyl (1%, assumed to be an artifact) were also detected in both soils, and MB 45897 (<1%) in Speyer 2.2 soil.

The initial half-lives determined by HPLC were 128 and 308 days in Manningtree and Speyer soils respectively.

A study was conducted to investigate the rate of degradation of phenyl ring-labelled [^{14}C]fipronil in four European soils incubated under aerobic conditions at 22°C and 10°C (Humphreys *et al.*, 1994). It used radiolabelled test material to determine the nature of the bound residues. The characteristics of the soils used are shown in Table 35.

Table 35. Soil characteristics (Humphreys *et al.*, 1994).

Soil	Origin	% Clay	% Silt	% Sand	% Organic matter	pH
Speyer 2.2	Germany	8	9	83	5.7	6.3
Sandy loam	UK	9	11	80	0.75	6.4
Sandy clay loam 1	France	26	27	47	1.2	6.16
Sandy clay loam 2	France	24	26	50	2.2	6.18

The soils were allowed to equilibrate for 14 days at 0.33 bar moisture holding capacity (MHC) and then raised to 0.1 bar MHC for dosing and incubation. Dishes containing 75 g of the appropriate soil (oven-dried equivalent) were dosed with [^{14}C]fipronil at a rate equivalent to 200 g ai/ha and incubated in the dark under aerobic conditions. Sample traps were used to retain any volatile products. Duplicate soil and trap samples were taken at 7 days and at 1, 3, 5 (sandy clay loam 1 only), 6, 9 and 12 months. Good radiochemical recoveries were achieved and most of the radioactivity was extracted. Radio-HPLC analysis showed a reduction of fipronil content with time for each soil. Half-lives from the Timme model (Timme *et al.*, 1986) are shown in Table 36.

Table 36. Estimated half-lives of fipronil in soil treated with [^{14}C]fipronil at 200 g ai/ha (Humphreys *et al.*, 1994).

Soil	Half-life (days), 10° C	Half-life (days), 22° C
Speyer 2.2 (Germany)	247	62
Sandy loam (UK)	163	117
Sandy clay loam 1 (France)	61	18
Sandy clay loam 2 (France)	62	40

The main degradation product in all cases was RPA 200766 (about 30-47%), with MB 46136 about 20%. MB 45950 was found at levels below 10% (below 5% in most cases), and RPA 105320 and MB 45897 at very low levels. These compounds accounted for more than 85% of the extractable radioactivity in the Speyer and sandy loam soils, and more than 60% in the sandy clay loam.

Polar products not previously detected were found in significant amounts (from 5.9% to 29% collectively) in the later stages of the study, at higher levels in the sandy clay loams than in the other soils. These were acid analogues of fipronil and its degradation products, the result of hydrolysis of nitrile to amide and thence to carboxylic acid, found at levels reflective of the abundance of their precursors. RPA 200761, the acid derived from amide RPA 200766, was detected at the highest level in all soils followed by RPA 106881, the acid of MB 46136. MB 46233, the acid of MB 45950, was observed at much lower concentrations. Of these acids, only RPA 200761 was found, in some samples, at levels above 10% of the applied dose.

Adsorption/desorption

The adsorption/desorption characteristics of fipronil, MB45950, MB 46136 and fipronil-desulfinyl have all been studied.

Fipronil. Godward *et al.* (1992) studied the soil adsorption and desorption of [^{14}C]fipronil in five European soils: Speyer 2.2 (Germany), sandy loam and loam (UK), and sandy clay loam 1 and 2 (France).

Recoveries of ^{14}C throughout the study were essentially quantitative for all soils and [^{14}C]fipronil was stable throughout. K_{oc} values calculated for each soil and the Freundlich isotherm constants for adsorption and desorption are shown in Table 37.

Table 37. Freundlich adsorption/desorption constants for fipronil (Godward *et al.*, 1992).

Soil	% organic C	K	1/n	K_{OC}
Adsorption				
German Speyer 2.2	3.35	14.32	0.947	427
UK sandy loam	0.34	4.19	0.950	1248
UK loam	4.25	20.69	0.938	486
French sandy-clay-loam-1	1.16	9.32	0.969	800
French sandy-clay-loam-2	1.59	10.73	0.949	673
Desorption				
German Speyer 2.2	3.35	13.35	0.905	398
UK sandy loam	0.34	7.25	0.986	2162
UK loam	4.25	21.51	0.910	506
French sandy-clay-loam-1	1.16	10.14	0.960	870
French sandy-clay-loam-2	1.59	12.88	0.948	808

Comparison of the adsorption and desorption isotherms indicates that the processes involved in adsorption and desorption are similar.

The results indicate that fipronil is unlikely to show much mobility in soil. According to McCall's designation, fipronil would be expected to be of medium to low mobility (McCall *et al.*, 1980).

MB 45950. The adsorption and desorption properties of [^{14}C]MB 45950 (phenyl-labelled), a soil degradation product of fipronil, have been investigated in four soils: silt and sandy loam (USA), loam and silt loam (UK) and a sediment sandy clay loam (UK) (McMillan, 1997b).

Recoveries throughout the study were essentially quantitative and ^{14}C-MB 45950 was stable. The K_{oc} values calculated for each soil and the Freundlich equation constants for adsorption and desorption are shown in Table 38.

Table 38. Freundlich adsorption/desorption constants for MB 45950 (McMillan, 1997b).

Soil	% organic C	K	1/n	K_{OC}
Adsorption				
US Silt loam	0.5	28.1	1.046	5621
US Sandy loam	1.2	42.36	0.950	3530
UK Loam	2.2	9.97	0.997	4362
UK Silt loam	1.9	32.2	0.932	1695
UK Sediment	2.3	100.02	0.970	4349
Desorption				
US Silt loam	0.5	27.87	0.958	5574
US Sandy loam	1.2	48.48	0.945	4040
UK Loam	2.2	94.59	0.968	4300
UK Silt loam	1.9	37.92	0.923	1996
UK Sediment	2.3	97.59	0.953	4243

Comparison of the isotherms derived from the adsorption and desorption data indicates that adsorption is reversible with substantial hysteresis in the adsorption/desorption curve. The results show that MB 45950 would not be expected to show any significant movement in soil. According to the McCall classification MB 45950 should be classified as having low mobility.

<u>MB 46136</u>. The adsorption and desorption of the degradation product [^{14}C]MB 46136 (phenyl-labelled) have been investigated in four soils, silt and sandy loam (USA), loam and silt loam (UK), and a sediment (UK) classified as sandy clay loam according to the USDA classification (McMillan, 1997a).

Because of the low solubility of MB 46136 in water only low concentrations could be used. This, and the fact that the compound is adsorbed to glass, contributed to some variability in recoveries, which occasionally fell outside the target range of 90-110%. [^{14}C]MB 46136 was stable throughout.

The K_{oc} values calculated for each soil and the Freundlich equation constants for adsorption and desorption are shown in Table 39.

Table 39. Freundlich adsorption/desorption constants for MB 46136 (McMillan, 1997a).

Soil	% organic C	K	1/n	K_{OC}
Adsorption				
US silt loam	0.5	26.55	1.141	5310
US sandy loam	1.2	48.64	0.996	4054
UK loam	2.2	148.4	1.054	6745
UK silt loam	1.9	27.51	0.947	1448
5 UK sediment	2.3	80.18	0.970	3486
Desorption				
US silt loam	0.5	659.1	1.696	131815
US sandy loam	1.2	72.1	1.024	6008
UK loam	2.2	231	1.085	10500
UK Silt loam	1.9	33.8	0.947	1777
UK Sediment	2.3	95.1	0.979	4136

The shape of the isotherms suggest a single adsorption mechanism. In all cases, the desorption mechanism is relatively independent of the treatment concentration.

The results indicate that MB 46136 would not be expected to show any significant movement in soil. According to McCall *et al.* (1980) MB 45950 should be classified as having low to negligible mobility.

<u>Fipronil-desulfinyl</u>. The soil adsorption and desorption properties of the photodegradation product [^{14}C]fipronil-desulfinyl (phenyl-labelled) were investigated in four soils and a sediment in the USA:

silt loam, clay, sand and loamy sand, and a sediment classified as loam according to the USDA classification (Feung and Mislankar, 1996).

Recoveries throughout the study were essentially quantitative for all soils, and [^{14}C]fipronil-desulfinyl was stable throughout. The K_{OC} values calculated for each soil and the Freundlich equation constants for adsorption and desorption are shown in Table 40.

Table 40. Freundlich adsorption/desorption constants for fipronil-desulfinyl (Feung and Mislankar, 1996).

Soil	% organic C	K	1/n	K_{OC}
Adsorption				
Silt loam	0.5	5.47	0.738	1094
Clay	1.2	15.2	1.183	1267
Sand	0.4	4.34	0.920	1085
Loamy sand	0.3	5.13	0.953	1710
Sediment	5.0	69.3	0.947	1386
Desorption				
Silt loam	0.5	6.21	0.793	1242
Clay	1.2	14.7	0.916	1225
Sand	0.4	5.77	0.932	1443
Loamy sand	0.3	5.93	0.951	1977
Sediment	5.0	66.2	0.913	1324

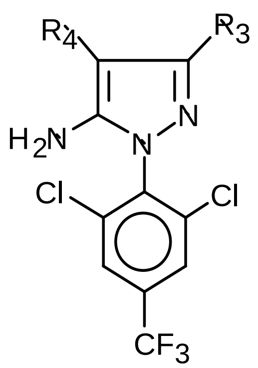
The desorption results demonstrated that fipronil-desulfinyl, once adsorbed, was tightly bound to soil. According to McCall *et al.* (1980) fipronil-desulfinyl should be classified as having low mobility.

<u>Accumulation of fipronil in confined rotational crops</u>.

The reference standards used in confined rotational crop studies are listed in Figure 6.

Figure 6. Structures of fipronil and standards representing potential metabolites.

General structure



Compound	R_3	R_4
Fipronil	CN	$SOCF_3$
MB 45950	CN	SCF_3
MB 46136	CN	SO_2CF_3
RPA 200766	$CONH_2$	$SOCF_3$
Fipronil-desulfinyl	CN	CF_3
MB 200761	COOH	$SOCF_3$
MB 45897	CN	H
RPA 105048	$CONH_2$	CF_3
RPA 104615	CN	SO_3H
RPA 105320	$CONH_2$	SO_2CF_3

Soil incorporation treatment

In a study by Jesudason and Mackie (1995) phenyl ring-labelled [^{14}C]fipronil was applied to a sandy loam soil at 157 g ai/ha and incorporated. Samples were collected after application to determine whether the soil had retained radioactivity approximating the theoretical application rate. The treated soil was then planted with carrots, radishes or lettuce, and sorghum or wheat at 30, 153 and 365 days after treatment (DAT). Lettuce leaf was harvested 36-196 days, radish leaf and root 36 to 51 days, carrot leaf and root 83 days, wheat forage 51 days, wheat straw and grain 231 days, sorghum forage 20-36 days and sorghum grain 107 to 112 days after planting. The total ^{14}C residues are shown in Table 41.

Table 41. TRR in rotational crops treated with [^{14}C]fipronil (Jesudason and Mackie, 1995).

Sample	TRR (DPM/g[1] and mg/kg [^{14}C]fipronil equivalents)					
	30 DAT		153 DAT		365 DAT[2]	
	DPM/g	mg/kg	DPM/g	mg/kg	DPM/g	mg/kg
Lettuce leaf	161	0.003[3]	274	0.006[3]	425	0.009[3]
Carrot leaf	1047	0.021	--	--	--	--
Carrot root	796	0.016	--	--	--	--
Radish leaf	--	--	199	0.004[3]	317	0.006[3]
Radish root	--	--	131	0.003[3]	171	0.003[3]
Wheat forage	--	--	837	0.017	--	--
Wheat straw	--	--	8495	0.172	--	--
Wheat grain	--	--	590	0.012	--	--
Sorghum forage	1380	0.028	--	--	667	0.014
Sorghum stover	1761	0.036	--	--	1169	0.024
Sorghum grain	410	0.008[3]	--	--	803	0.016

[1] DPM/g:-disintegrations per min/g tissue
[2] DAT: days after treatment
[3] <0.01 mg/kg, not analysed further

All crops that had TRR values at or above 0.01 mg/kg were extracted and the extracted residues characterized and identified. Total recoveries were more than 88% of the TRR in all samples at the three rotational intervals. All extracted residues were organosoluble in 30 DAT carrot leaf, carrot root and sorghum forage, 153 DAT wheat forage, and 365 DAT sorghum forage. Water-soluble residues in 30 DAT sorghum stover, 153 DAT wheat grain, and 365 DAT sorghum were below 0.01 mg/kg. Unextractable residues were below 0.01 mg/kg in all samples.

Organosoluble compounds were identified by two reverse-phase HPLC methods using a Spherisorb ODS column and a Hichrom spherisorb semi-preparative column with UV (280 nm) and radiometric detection. The LOQ ranged from 0.001 to 0.003 mg/kg. The distribution of organosoluble compounds found in the analysed rotational crops is shown in Table 42.

Table 42. Organosoluble residues identified in rotational crops (Jesudason and Mackie, 1995).

Residues in organosoluble extracts, mg/kg as compound, not ^{14}C as fipronil											
Sample	TRR	fipronil	RPA 200761	RPA 200766	MB 46136	Fipronil-desulfinyl	RPA 105320	MB 45950	RPA 104615	RPA 105048	un-known
30 DAT											
Carrot											
Leaf	0.021	0.005	0.001[1]	0.010	0.001	ND	0.001	ND	0.001[1]	ND	ND
Root	0.016	0.005	<LOQ	0.002	0.005	ND	ND	0.004	<LOQ	ND	ND

Residues in organosoluble extracts, mg/kg as compound, not ^{14}C as fipronil											
Sample	TRR	fipronil	RPA 200761	RPA 200766	MB 46136	Fipronil-desulfinyl	RPA 105320	MB 45950	RPA 104615	RPA 105048	un-known
Sorghum											
Forage	0.028	0.013	0.004[1]	0.003	0.003	ND	ND	ND	0.004[1]	ND	ND
Stover	0.036	0.003	0.003	0.004	0.008	0.001	ND	ND	0.003	ND	0.002
153 DAT											
Wheat											
Forage	0.017	0.003	ND	0.003	0.003	ND	ND	ND	ND	ND	0.006
Straw	0.172	0.020	0.015	0.067	0.044	0.019	0.012	ND	ND	0.003	0.007
Grain	0.012	ND	0.006	0.001	ND	ND	ND	ND	ND	ND	0.002
365 DAT											
Sorghum											
Forage	0.014	0.001	0.002	0.001	0.001	ND	ND	ND	0.003	ND	ND
Stover	0.024	0.001	0.003	0.005	0.004	ND	ND	ND	ND	ND	0.003
Grain	0.016	ND	0.009	ND	ND	ND	ND	ND	ND	ND	ND

[1] The concentration of RPA 200761 and RPA 104615 together, not individually
ND: not detectable

The results are consistent with established routes of environmental degradation and plant metabolism of fipronil and demonstrate common pathways in rotated crops, and indicate that:

- The uptake of the TRR is low, in this study less than 0.01 mg/kg in 8 of 18 samples analysed at the three rotational intervals and between 0.01 and 0.05 mg/kg in 9 of 18.
- Fipronil and its metabolites are not highly systemic. No significant residues were found in the edible substrates analysed (those with TRR >0.01 mg/kg). Only cereal straw and forage, both animal feed items, contained residues higher than 0.01 mg/kg.

Soil surface treatment

In a study by Jesudason and Mackie (1999) phenyl ring-labelled [^{14}C]fipronil was applied to the surface of a sandy loam soil at 369 g ai/ha, and the plots planted with lettuce (*Lactuca sativa*), and radish (*Raphanus sativus*), and sorghum (*Sorghum vulgare*) or wheat (*Triticum aestivum*) 30, 150 and 365 days after treatment. All crops were grown to maturity and sampled, and sorghum and wheat were also sampled at the forage stage. The crops were harvested at intervals after planting of 32-43 days for lettuce, 32-43 days for radish, 196 days for wheat forage, 259 days for wheat straw and grain, 33-74 days for sorghum forage and 118-171 days for sorghum grain and fodder. Recoveries of ^{14}C are shown in Table 43.

Table 43. TRR in rotational crops after surface treatment of soil with [^{14}C]fipronil (Jesudason and Mackie, 1999).

Sample	TRR (DPM/g and mg/kg [^{14}C]fipronil equivalents)					
	30 DAT		150 DAT		365 DAT	
	DPM/g	mg/kg	DPM/g	mg/kg	DPM/g	mg/kg
Lettuce leaf	2562	0.040	608	0.009	1624	0.025
Radish leaf	6812	0.11	631	0.010	1667	0.026
Radish root	1615	0.025	196	0.003[1]	362	0.006 [1]
Wheat forage	--	--	3007	0.047	--	--
Wheat straw	--	--	13345	0.21	--	--
Wheat grain	--	--	789	0.012	--	--
Sorghum forage	3320	0.052	--	--	3100	0.048
Sorghum fodder	7757	0.12	--	--	2350	0.037
Sorghum grain	1712	0.027	--	--	1006	0.016

DAT: days after treatment
DPM/g: disintegrations per min/g tissue
[1] Not analysed further

The recovery of ^{14}C was >97% from the extracted samples except radish leaves (70% of 0.001 mg/kg). Recoveries of extractable ^{14}C from samples with >0.01 mg/kg ranged from 86% to 110% of the TRR, and the total identified compounds in solvent-extractable fractions from 31% to 84%. The unidentified radioactive compounds (each $\geq$0.01 mg/kg) were characterized. The processed samples were extracted with an organic solvent, followed by a mildly acidified organo-aqueous mixture, and the combined extracts were turbo-evaporated and/or further cleaned up, if necessary, through a C-18-Sep-Pak cartridge before HPLC analysis. The unextractable residue was combusted and radioassayed to determine the bound residue. The LOQ for extracts counted by liquid scintillation ranged from 0.001 to 0.006 mg/kg. The metabolites were consistent with those found in earlier studies. The results are summarized in Table 44.

Table 44. Identified residues in rotational crops (Jesudason and Mackie, 1999).

Sample	Residues in organosoluble extracts (mg/kg of compound, not ^{14}C)											
	Solvent extrac-table (mg/kg)	Fipronil	MB 46136	MB 46513	MB 45950	RPA 200761	RPA 200766	RPA 105320	RPA 104615	RPA 105048	% of TRR Identi-fied	Un-known	Total as % TRR
30 DAT													
Lettuce leaf [1]	0.024	0.006	0.003	0.001	ND	<LOQ	0.006	0.001	0.002	0.003	56	0.002	61
Radish leaf	0.083	0.002	0.006	0.001	ND	0.002	0.011	ND	0.054	0.005[2]	77	0.002	79
Radish root	0.021	0.003	0.007	0.001	ND	ND	0.002	ND	0.008	ND	84	0.001	88
Sorghum forage	0.037	0.006	0.002	0.001	ND	0.004	0.002	ND	0.016	0.001[2]	62	0.005	72
Sorghum fodder	0.068	ND	0.013	ND	ND	0.010	ND	ND	0.045	ND	57	ND	57
Sorghum grain	0.014	ND	ND	ND	ND	0.006	ND	ND	0.005	ND	39	0.003	50
150 DAT													
Lettuce leaf	0.004	ND	ND	ND	ND	ND	0.002	ND	ND	ND	22	0.004	67
Radish leaf	0.004	ND	ND	ND	ND	0.002	ND	ND	ND	ND	23	0.002	45
Wheat forage	0.034	0.007	0.008	0.003	ND	ND	0.011	0.003[2]	ND	0.003[2]	77	ND	77
Wheat straw	0.152	0.007	0.051	0.012	ND	ND	0.039	ND	0.027	ND	65	0.016	73
Wheat grain	0.009	ND	ND	ND	ND	ND	ND	ND	0.001	ND	8	0.008	79
365 DAT													
Lettuce leaf	0.024	0.004	0.008	0.001	0.001	ND	0.004	0.002	ND	0.001	84	0.003	96
Radish leaf	0.02	ND	ND	ND	ND	0.012	0.004	ND	ND	ND	61	0.004	76
Sorghum forage	0.035	ND	ND	ND	ND	0.013	0.002	ND	ND	ND	31	0.021	74
Sorghum fodder[3]	0.031	ND	0.006	ND	ND	0.011	ND	ND	0.009	ND	83	0.005	70
Sorghum grain[4]	0.011	ND	ND	ND	ND	0.005	0.003	ND	ND	ND	37	0.003	69

ND: not detectable

[1] Metabolite MB 45897 was <LOQ

[2] Confirmed by LC-MS-MS in 30 DAT lettuce only

[3] Trace of parent detected by LC-MS-MS

[4] Trace of RPA 104615 detected by LC-MS-MS; presence of RPA200766 unconfirmed by LC-MS-MS

The sum of the residues of fipronil, MB 46136, fipronil-desulfinyl and MB 45950 ranged from 0.006 to 0.07 mg/kg. MB 45950 was detected in only one sample, at 0.001 mg/kg, at the last rotation. The study demonstrates that neither fipronil nor its significant metabolites are likely to be found in grain at any plant-back interval of a month or more, or in root crops planted 5 or more months after foliar applications at 369 g ai/ha.

Accumulation in field rotational crops

In a field rotational crop study at two sites, North Carolina and California, USA, one plot at each site was treated once at the maximum US proposed labelled rate of 0.34 kg ai/ha as a soil-surface, fallow-soil application with commercial equipment (Carringer, 1998b). The test and control plots were divided into 16 subplots, and examples of root, leafy and legume vegetables and small grain were planted 30, 120, 240 and 365 days after treatment. At each plant-back interval, four crops from the crop groups were sampled at normal harvest maturity. The minimum limit of detection (MLD) and LOQ were 0.002 and 0.005 mg/kg respectively. The results are summarized in Tables 45 and 46.

Residues of fipronil, fipronil-desulfinyl, MB 45950, and MB 46136 were below the MLD in all control samples except in one 119-day untreated green pea forage sample from the California site which had residues between the MLD and LOQ, and below the LOQ in treated samples from the North Carolina site, except in a 120-day treated winter wheat sample with residues of 0.006 mg/kg MB 46136 and 0.022 mg/kg fipronil-desulfinyl in the straw.

Significant residues were found only at the California site where they were exclusively in the vegetative portion of the crops not in the reproductive portion (i.e. grain). At 119, 239 and 367-day plant-back intervals residues from <0.005 to 0.026 mg/kg were found only in animal feedstuffs (small grain forage and straw, and legume forage). Only at the 31-day plant-back interval were quantifiable residues found in lettuce and radish tops, ranging from 0.008 to 0.016 mg/kg.

Table 45. Residues of fipronil, MB 45950, MB 46136 and fipronil-desulfinyl in treated rotational crops, California site (Carringer, 1998b).

Plant-back interval (DAT)	Sample	Days after planting	Residues (mg/kg)[1]			
			Fipronil	fipronil-desulfinyl	MB 45950	MB 46136
31	Radish top	32	ND, ND	0.011, 0.014	ND, ND [2]	<0.005, <0.005
	Radish root	32	ND, ND	ND, <0.005	ND, ND	ND, ND
	Cowpea forage	52	ND, ND	0.009, 0.009	ND, ND	ND, ND
	Cowpea grain	144	ND, ND	ND, ND	ND, ND	ND, ND
	Cowpea straw	144	ND, ND	ND, ND	ND, ND	ND, ND
	Red leaf lettuce	50	ND, ND	0.011, 0.016	ND, ND	0.008, 0.012
	Sorghum forage	33	ND, ND	0.01, 0.007	ND, ND	<0.005, <0.005
	Sorghum grain	122	ND, ND	ND, ND	ND, ND	ND, ND
	Sorghum straw	122	<0.005,ND	0.031, 0.032	ND, ND	0.024, 0.021
119	Radish top	33	ND, ND	<0.005, <0.005	ND, ND	ND, ND
	Radish root	33	ND, ND	ND, ND	ND, ND	ND, ND
	Green pea forage	57	<0.005, ND	ND, ND	ND, ND	<0.005,ND
	Green pea grain	140	ND, ND	ND, ND	ND, ND	ND, ND
	Green pea straw	140	ND, ND	<0.005, ND	ND, ND	<0.005, <0.005
	Green leaf lettuce	57	ND, ND	<0.005, <0.005	ND, ND	ND, ND
	W. wheat forage	57	ND, ND	<0.005, <0.005	ND, ND	ND, ND
	W. wheat grain	176	ND, ND	ND, ND	ND, ND	ND, ND
	W. wheat straw	176	ND, ND	0.022, 0.019	ND, ND	<0.005, <0.005
239	Radish top	55	NA[3]	NA	NA	NA
	Radish root	55	NA	NA	NA	NA
	Cowpea forage	132	ND, ND	0.006, 0.005	ND, ND	<0.005, <0.005
	Cowpea grain	191	NA	NA	NA	NA
	Cowpea straw	191	NA	NA	NA	NA
	Red leaf lettuce	90	NA	NA	NA	NA
	W. wheat forage	83	ND, ND	<0.005, <0.005	ND, ND	ND, ND
	W. wheat grain	132	NA	NA	NA	NA
	W. wheat straw	132	ND, ND	0.026, 0.022	ND, ND	0.016, 0.013
367	Radish top	36	NA	NA	NA	NA
	Radish root	36	NA	NA	NA	NA
	Cowpea forage	60	NA	NA	NA	NA
	Cowpea grain	105	NA	NA	NA	NA
	Cowpea straw	105	NA	NA	NA	NA
	Red leaf lettuce	60	NA	NA	NA	NA
	Sorghum forage	60	ND, ND	<0.005, <0.005	ND, ND	ND, <0.005
	Sorghum grain	142	NA	NA	NA	NA
	Sorghum straw	142	ND, ND	0.010, 0.018	ND, ND	0.013, 0.02

ND: not detectable W: winter

[1] Duplicate samples analysed

[2] Not detectable, MLD: 0.002 mg/kg

[3] NA: not analysed if residues not above the LOQ at the previous plant-back interval

Table 46. Residues of fipronil, MB 45950, MB 46136 and fipronil-desulfinyl in treated rotational crops, North Carolina site (Carringer, 1998b).

Plant-back Interval (DAT)	Crop sample	Age of crop at sampling (days)	Residues (mg/kg) [1]			
			Fipronil	fipronil-desulfinyl	MB 45950	MB 46136
31	Radish top	34	ND, ND [1,2]	ND, ND	ND, ND [2]	ND, ND
	Radish root	34	ND, ND	ND, ND	ND, ND	ND, ND
	Soya bean forage	34	ND, ND	<0.005, ND	ND, ND	ND, ND
	Soya bean grain	146	ND, ND	ND, ND	ND, ND	ND, ND
	Soya bean straw	146	ND, ND	<0.005, <0.005	ND, ND	<0.005, <0.005
	Collard foliage	125	ND, ND	ND, ND	ND, ND	ND, ND
	Sorghum forage	34	ND, ND	ND, ND	ND, ND	ND, ND
	Sorghum grain	124	ND, ND	ND, ND	ND, ND	ND, ND
	Sorghum straw	124	ND, ND	ND, ND	ND, ND	ND, ND
120	Radish top	57	ND, ND	ND, ND	ND, ND	ND, ND
	Radish root	57	ND, ND	ND, ND	ND, ND	ND, ND
	Wando pea forage	Not collected	--	--	--	--
	Wando pea grain	Not collected	--	--	--	--
	Wando pea straw	Not collected	--	--	--	--
	Mustard foliage	215	ND, ND	ND, ND	ND, ND	ND, ND
	W. wheat forage	201	ND, ND	ND, ND	ND, ND	ND, ND
	W. wheat grain	285	ND, ND	ND, ND	ND, ND	ND, ND
	W. wheat straw	286	ND, ND	0.022, 0.019	ND, ND	<0.005, 0.006
296	Radish top	60	NA[3]	NA	NA	NA
	Radish root	60	NA	NA	NA	NA
	Wando pea forage	60	NA	NA	NA	NA
	Wando pea grain	109	NA	NA	NA	NA
	Wando pea straw	110	NA	NA	NA	NA
	Mustard foliage	60	NA	NA	NA	NA
335	S. wheat forage	92	NA	NA	NA	NA
	S. wheat grain	Not collected	NA	NA	NA	NA
	S. wheat straw	Not collected	NA	NA	NA	NA
365	Radish top	40-41	NA	NA	NA	NA
	Radish root	40-41	NA	NA	NA	NA
	Soya bean forage	64	NA	NA	NA	NA
	Soya bean grain	177	NA	NA	NA	NA
	Soya bean straw	177	NA	NA	NA	NA
	Mustard foliage	64	NA	NA	NA	NA
	Sorghum forage	40-41	NA	NA	NA	NA
	Sorghum grain	141	NA	NA	NA	NA
	Sorghum straw	141	NA	NA	NA	NA

W.: winter
S.: spring
[1] Duplicate field samples analysed
[2] ND: not detectable, MLD: 0.002 mg/kg
[3] NA: not analysed if residues were not above the LOQ at the previous plant-back interval

The results indicated that no residues would be found in cereal grain at any interval. The low levels in animal feed supported a 31-day plant-back interval for soya beans and a 120-day plant-back interval for leafy vegetables and root crops after a foliar application of fipronil at a rate of 0.34 kg ai/ha.

<u>Summary</u>

When fipronil is applied to the soil at 157 g ai/ha uptake is low in rotational crops. Only two animal feed items from the small grain crops contained residues above 0.01 mg/kg, indicating that fipronil and its relevant metabolites are not highly systemic.

After surface application of fipronil at 369 g ai/ha to the soil neither fipronil nor its relevant metabolites are likely to be found in grain after 30 to 365 days, nor in root crops or leafy vegetables after 5 months.

A field crop rotational study confirmed that after application at 340 g ai/ha to the soil surface residues in the vegetative portions of crops are low and undetectable in grain. At plant-back intervals of 119-367 days after treatment residues ranged from <0.005 mg/kg to 0.026 mg/kg in animal feedstuff samples. Only at short plant-back intervals (30 DAT) were residues found in leafy and root crops (<0.002 to 0.032 mg/kg).

Environmental fate in water/sediment systems

Hydrolysis

The hydrolysis of [^{14}C]fipronil at 25°C was determined by Corgier and Plewa (1992a) in a study in the dark, under sterile conditions, at pH 5, 7, and 9 at an initial concentration of 0.89 mg/l of buffer solution. The results were as follows.

pH 5 (buffered)	stable
pH 7 (buffered)	nearly stable (2% loss in 30 days)
pH 9 (buffered)	DT-50 approximately 28 days

At pH 9 fipronil was converted exclusively to RPA 200766, according to pseudo-first-order kinetics, with a half-life of 28 days and rate constant k = -0.0243 day^{-1}. No volatile compounds were found at any pH. Recoveries of ^{14}C ranged from 96.4% to 101.6%.

Aqueous photolysis

Corgier and Plewa (1992b) determined a half-life of 0.33 days.

In another study by Boinay (1997) at pH 5 and 25°C under sterile conditions at an initial concentration of 0.9 mg/l with 1% acetonitrile as co-solvent, light was provided by a xenon lamp with radiation of less than 290 nm filtered out. Time under the lamp was converted to equivalents of ‘Florida summer days’. Irradiation was for 0, 1, 2, 4 or 6 hours. Control samples were maintained in darkness for 6 hours. Analyses of test and trap solutions showed good recoveries ranging from 99.8 to 103% of the applied radioactivity. Practically no volatile compounds were formed.

After 6 hours the main organoextractable photoproduct was fipronil-desulfinyl, with a minor unknown (HPLC RT = 2 min) accounting for 43% and 4% of the applied radioactivity respectively. The aqueous extract contained RPA 104615 and a minor unknown (HPLC RT = 3.3 min) accounting for 8.2% and 5.6% respectively. The remaining 32% was accounted for by the parent compound. Dark controls showed no appreciable degradation.

The kinetics of photolytic degradation were pseudo first order with a DT-50 of 0.33 days and k = -0.0176 days^{-1}. The quantum yield of the direct photolysis of fipronil in an aqueous solution was determined in a radiometer irradiation apparatus at 300 nm and was 0.199 (mean of two values).

Biodegradability

In a study to assess the biodegradability of fipronil in an aerobic aqueous medium (Mead, 1997) fipronil was exposed to activated sewage sludge micro-organisms at 19.05 mg/l with culture medium in sealed vessels stored in the dark at 21°C for 28 days. Degradation of the test material was assessed by measuring the carbon dioxide produced.

Preliminary assessment of the toxicity of fipronil to the micro-organisms was not possible because of the insolubility of the test material. Therefore fipronil and sodium benzoate were included as a toxicity control for validation, in addition to innoculum and standard sodium benzoate controls.

After 28 days, degradation of fipronil was 47% and of sodium benzoate 100% confirming the suitability of the inoculum and test conditions. The toxicity control achieved 42% degradation, confirming that the test substance was not toxic to the micro-organisms.

Anaerobic aquatic degradation

The anaerobic aquatic degradation of [^{14}C]fipronil after application at 10 µg/cm^2 to a flooded sandy loam soil, already incubated for 53 days to establish stable anaerobic conditions, was studied for a year (Waring, 1993a). Soil 5 cm deep was covered by 12 cm of deionized water and kept in the dark at 25 ± 1°C in glass cylinders. Moistened nitrogen gas was passed over the water surface of each cylinder and then through a variety of traps to collect radiolabelled volatiles. Duplicate samples were removed for analysis at 0, 1, 3, 7, 14, 30, 59, 120, 179, 269 and 365 days after application.

Recoveries from the surface water decreased from >93% initially to 13-21% after 365 days incubation. The soil contained 69% of the applied ^{14}C after 365 days. Most of the radioactivity in the soil (>62%) was extracted with acetonitrile. Less than 0.1% was collected in the traps.

Initially [^{14}C]fipronil accounted for >92% of the applied ^{14}C, but decreased during the course of the study to <16%. Decomposition resulted mainly in the formation of the reduced product MB 45950 and the amide RPA 200766, accounting for 32 and 47% of the applied radioactivity respectively, after 365 days. Several unidentified minor degradation products were detected in the soil samples by TLC or HPLC. The initial half-life of [^{14}C]fipronil in flooded Manningtree, UK, sandy loam soil under anaerobic conditions was about 123 days.

Degradation in water/sediment systems

The degradation of both fipronil and fipronil-desulfinyl has been studied.

Fipronil. The degradation of [^{14}C]fipronil under aerobic conditions in two systems each comprising an aerobic water phase and a sediment phase under reducing conditions (system 1 "Ongar, UK" and system 2 "Manningtree, UK") over a period of 121 days was studied by Ayliffe (1998). Glass flasks containing 4 cm of soil sediment covered by 11 cm of associated water were incubated in the dark at 20 ± 2°C. The ratio of water to oven-dry equivalent weight of sediment was approximately 1:4. The systems were acclimatized for 28 days before the single application of [^{14}C]fipronil at the equivalent of 200 g ai/ha to the surface water of each flask. Moistened air was supplied under positive pressure into the water surface and the effluent air passed through two 1 M potassium hydroxide traps to collect any liberated CO_2. Samples were analysed 0, 0.02, 0.25, 1, 2, 7, 14, 29, 58, 93 and 121 days after [^{14}C]fipronil application.

Mean recoveries were above 94% in the two systems. The radioactivity was gradually transferred from the water to the sediment. By the end of the study, ^{14}C levels in the water were about 12.4% and 2.5% of the total applied in systems 1 and 2 respectively, and in extracts of the two sediments c. 83% and 94% respectively. The unextractable residues recovered by combustion were generally below 5%. Volatiles accounted for less than 1% of the applied radioactivity in both systems.

The main degradation product was the sulfide MB 45950, with maximum concentrations of 88% in system 1 and 80% in system 2. Up to 4 minor products generally accounted for less than 2% of the applied ^{14}C at any time. These included RPA 200766 and the amide of MB 45950, MB 46126.

The half-lives of fipronil in the water phase, sediment and the total system were characterized by HPLC of extracts, and were less than 14 and 6 days in water, 48 and 75 days in sediment and 22 and 32 days in the total system for systems 1 and 2 respectively.

Fipronil-desulfinyl. Lowden and Mahay (2000) determined the route and rate of degradation of ^{14}C-labelled fipronil-desulfinyl in systems with “Manningtree-UK” and “Ongar-UK” water phases treated at the equivalent of 100 g ai/ha in the water phase and incubated in the dark at 20°C for up to 365 days, with sampling at 0 h and 6 h and 1, 2, 7, 14, 30, 61, 100, 152 and 365 days after treatment. The water was separated from the sediment, then water and sediments were radioassayed and examined by HPLC. The recoveries of applied radioactivity had an overall mean value of 94.5%. Analysis of the water phases showed that there was initially a relatively rapid transfer of radiolabelled material from the water phases to the sediments in both systems, with a reduction in the rate of transfer after about 30 days. There was simultaneous degradation in the water and sediment resulting in the formation of four minor products. No significant quantities of volatile products were formed, <0.5% applied radioactivity (a.r.).

Two minor products were MB 46400 and RPA 105048 which reached a maximum of 6.8% and 4.3% of the a.r. in the total systems respectively. Two other minor products were also detected. Compound A was detected in the sediment extracts only, accounting for only 1.07% of the a.r. at 61 days before disappearing. Compound B appeared in both the water and sediment phases reaching a maximum of 1.6% of the a.r. in the total systems at 365 days. At 30 days the products accounted for 1.6% of the 22% of the a.r. in the water of the Manningtree system and 3.6% of 32% of the a.r. in the water of the Ongar system. At 365 days however, the products accounted for 5.7% of totals of 11% and 10% of the a.r. in the waters of the Manningtree and Ongar systems respectively.

In the sediments, the main component of the extractable materials was fipronil-desulfinyl which was accompanied by small amounts of MB 46400, RPA 105048 and compound B (Manningtree sediment only).

These results indicate that in the environment, any fipronil-desulfinyl reaching or formed in the water of a water/sediment system from a fipronil application will move to the sediment at an initially rapid rate. The degradation (principally hydrolysis) of the compound is likely to proceed steadily in both the water and sediment phases. The movement of the compound from water to sediment plus the degradation resulted in DT-50 values of 4.2 days and 9.9 days and DT-90 values of 174 days and 146 days for the Manningtree and Ongar water phases respectively.

Bioaccumulation in fish

In studies by Chapleo and Hall (1992) and Roohi *et al.* (1992) bluegill sunfish were exposed to a continuous flow of water containing 850 ng/l [^{14}C]fipronil for 35 days (uptake phase). Daily water samples showed that the test concentration remained between 810 and 990 ng/l (mean 900 ng/l) fipronil equivalents.

The fish were then exposed to a continuous flow of dilution water alone for two weeks (depuration phase), as were a control group. The radioactivity in edible (muscle) and non-edible (viscera) tissues during the uptake and depuration phases of the control fish was low: all results were below the limit of reliable determination.

After 14 days’ exposure the residues in whole fish and edible and inedible portions appeared to be stable.

In whole fish, concentrations of the TRR increased to a mean maximum of 315 ng/g fipronil equivalents fresh weight, corresponding to a bioconcentration factor of 380 after 35 days exposure to [^{14}C]fipronil. The apparent steady-state bioconcentration factor was 321, corresponding to 273 ng/g fipronil equivalents fresh weight. Over 99% of the radioactivity accumulated at the steady state was eliminated during the depuration phase.

During the uptake phase, most of the radioactivity (>69%) was in the inedible fraction, where the apparent steady-state bioconcentration factor was 575 corresponding to 489 ng/g fipronil equivalents fresh weight. Over 97% of the radioactivity at steady-state was eliminated during the depuration phase.

In the edible fraction, the apparent steady-state bioconcentration factor was 164, corresponding to 139 ng/g fipronil equivalents fresh weight. Over 96% of the radioactivity at the steady state was eliminated during the depuration phase.

The muscle and viscera (edible and inedible respectively), were subjected to extraction procedures designed to isolate the radiolabelled components. These extracts were then analysed using chromatographic and spectrometric techniques for the identification of individual components.

The results demonstrate that absorbed fipronil is metabolized to MB 46136, MB 45950 and MB 45897. The parent compound and its metabolite MB 45950 each accounted for approximately 11% of the TRR, and MB 45897 and MB 46136 for 26 and 44% respectively. The amide RPA 200766, was also present in lesser amounts. The percentage of these metabolites in the inedible lipophilic fraction exceeded that in the less lipophilic edible fraction by a factor of 3 to 4, consistent with the octanol/water partition coefficients of the compounds.

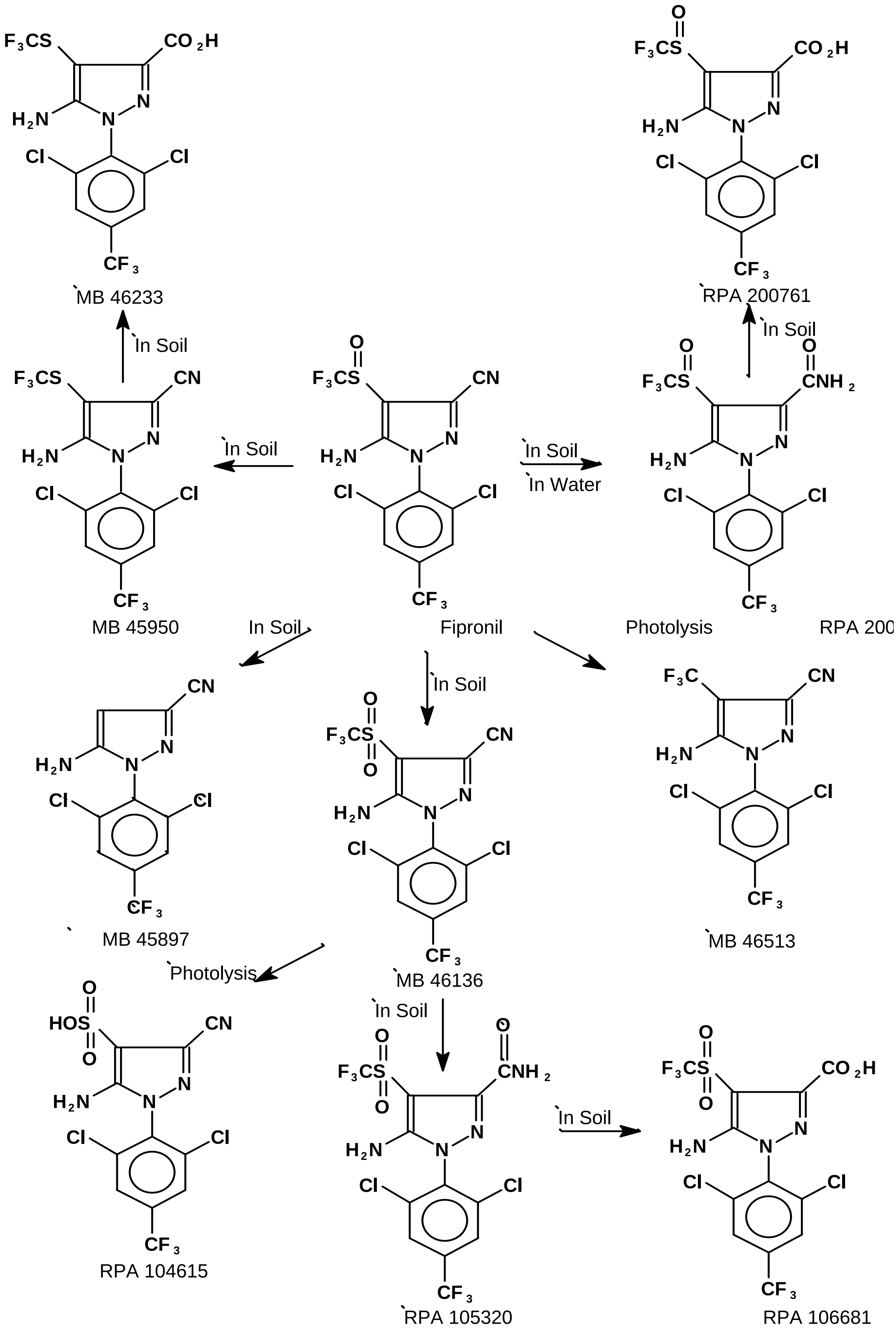
Analysis of the samples from days 1 to 3 of the depuration phase revealed the same compounds as at day 7, when approximately 90% of the TRR had been eliminated. In both cases and in common with the uptake phase, MB 46136 was the main metabolite, but results indicated that metabolism of the parent compound continued during the depuration phase and that no single metabolite was preferentially eliminated. The proportions of the metabolites in the edible and inedible fractions were found, in the initial phase of depuration, to be approximately the same as in the uptake phase, but the ratio decreased to 1.5 by day 7, consistent with the reasonably rapid elimination of the compounds from both fractions.

It is concluded that fipronil in fresh water is both accumulated by and eliminated from bluegill sunfish. The pattern of accumulation and elimination of [^{14}C]fipronil indicates that a simple, two-compartment model is approached. In all tissues and in whole fish, the accumulated radioactivity was almost completely eliminated (>96%) after 14 days depuration, with no indication of preferential elimination of any metabolite.

Summary of environmental fate

It has been demonstrated in laboratory studies that fipronil undergoes oxidation to the sulfone MB 46136 (a major degradation product in aerobic soil studies), reduction to the sulfide MB 45950 (a minor product in aerobic soil and a major product in aerobic and anaerobic aquatic studies), hydrolysis to the amide RPA 200766 (a major product in aerobic soil and hydrolysis studies and a minor product in the aquatic studies), and photolysis to the desulfinylated degradation product fipronil-desulfinyl (the major product in the aqueous photolysis study and minor in the soil photolysis study) and to a lesser extent the sulfonic acid RPA 104615 (only a minor product in aqueous degradation and soil photolysis). Figure 7 shows the proposed degradation pathways of fipronil in soil and water.

Figure 7. Degradation pathways of fipronil in the environment.



METHODS OF RESIDUE ANALYSIS

Analytical methods

Plant material

A multi-residue enforcement analytical method, DFG S19 modified, was validated for the determination of residues of fipronil, MB 45950, MB 4613, fipronil-desulfinyl, and RPA 200766 in maize, peaches and potatoes (Haussman, 1998). The modified method is outlined below:

- extraction with water/acetone
- subsequent partition into an organic phase after addition of ethyl acetate/cyclohexane
- gel permeation chromatography
- silica gel column fractionation
- GLC with electron capture detection

The method was validated at 0.002 mg/kg (the LOQ) and 0.02 mg/kg per analyte and gave acceptable average recoveries of 70-110% and relative standard deviations of ≤20% at both fortification levels in the three commodities, except of MB 45950 from maize at 0.02 mg/kg and of MB 46136 from maize and potatoes at 0.02 mg/kg, where mean recoveries were slightly below 70%. The average recovery of RPA 200766 from potato at 0.002 mg/kg was slightly above 110% (relative standard deviation of 25%). Residues in control samples were all ≤30% of the LOQ.

In an early general GLC method (Manley, 1993) fipronil and its metabolites are extracted with acetonitrile from plant material, water is added and the solution partitioned with hexane for clean-up. Residues are extracted with dichloromethane, and purified using solid-phase extraction before quantification by GLC with electron capture detection. The method was validated at 0.01 mg/kg for apples, rice bran, cabbage, grapes, maize grain, maize plant, maize silage, rice, sugar beet and sunflower. The limit of detection was estimated at 0.003 mg/kg for fipronil and its metabolites.

In an analytical method (Communal, 1994) to determine residues in vegetables, cereals and fruits fipronil, MB 45950, MB 46136, fipronil-desulfinyl, and RPA 200766 are extracted with acetonitrile, and the crude extract purified on a C-18 cartridge followed by activated charcoal. Analysis is by gas liquid chromatography with electrochemical detection (ELCD). The method has been validated for cabbage, watermelon, mango, maize, sunflower (including oil and oilcake), winter wheat, citrus fruits, cane sugar, potato, mushroom and banana. The LOQ for each compound in various substrates is shown in Table 47.

Table 47. LOQ for fipronil and degradation products in various substrates (Communal, 1994).

Substrate	Validated LOQs (mg/kg)				
	Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA200766
Cabbage	0.005	0.005	0.005	0.005	0.005
Watermelon	0.005	0.005	0.005	0.005	0.005
Mango	0.005	0.005	0.005	0.005	0.005
Maize silage	0.005	0.005	0.005	0.005	0.005
Maize grain	0.002	0.002	0.002	0.002	0.002
Wheat straw	0.01	0.01	0.01	0.01	0.01
Wheat grain	0.002	0.002	0.002	0.002	0.002
Wheat forage	0.01	0.01	0.01	0.01	0.01
Sunflower seed	0.002	0.002	0.002	0.002	0.002
Sunflower oil	0.002	0.002	0.002	0.002	0.002
Sunflower cake	0.002	0.002	0.002	0.002	0.002
Citrus	0.002	0.002	0.002	0.002	0.002
Sugar cane	0.01	0.01	0.01	0.01	0.01
Potato	0.002	0.002	0.002	0.002	0.002
Mushroom	0.002	0.002	0.002	0.002	0.002
Banana	0.002	0.002	0.002	0.002	0.002

In a method to determine residues of fipronil, MB 45950, MB 46136 and fipronil-desulfinyl in maize, cotton, potato and rice substrates (Baillargeon and Plaisance, 1998), residues are extracted from seed, meal, hulls, grain, forage, fodder and straw with acetonitrile/water and partitioned with hexane. After removal of the acetonitrile, the residues are partitioned with dichloromethane. Crude and refined oils are diluted with hexane before extraction. Residues are extracted from potato tubers, flakes, wet and dry peel and chips with acetonitrile/acetone. Column chromatography (various combinations of charcoal, silica gel, alumina, Florisil, amino) is used for clean-up of extracts. Quantification of fipronil and it metabolites is accomplished by gas chromatography using an electron capture or mass selective detector. The LOQs are shown in Table 48.

Table 48. LOQs in various substrates of maize, cotton, potato and rice (Baillargeon and Plaisance, 1998).

LOQ (mg/kg)	GLC/ECD
0.003	Potato - tuber, flakes, wet and dry peel, chips
0.005	Cotton - seed, meal, hulls, crude and refined oil
0.01	Rice - grain
0.01	Maize - grain, crude oil
0.02	Maize - forage, fodder, starch
	GLC/MSD
0.005	Maize - grain, crude oil
0.005	Cotton - crude and refined oil
0.01	Maize - forage, fodder, starch
0.01	Cotton - seed, meal, hulls
0.01	Rice - grain, straw
0.1	Cotton by-product (gin trash)

In a method developed by Le Galliot and Communal (1994) to determine residues of fipronil, MB 45950, MB 46136, fipronil-desulfinyl and RPA 200766 in cabbage, residues are extracted with acetonitrile and the crude extract is purified on a C-18 cartridge followed by activated charcoal. Analysis is by gas liquid chromatography with electrochemical detection (ELCD, Hall detector). The LOQ is 0.005 mg/kg for each compound.

In another method (Maycey *et al.*, 1995) to determine residues of fipronil and its possible metabolites MB 45897, MB 45950, MB 46136, fipronil-desulfinyl and RPA 200766 in rice (green plant, straw, bran, brown and white rice) residues in green plant and straw are extracted with methanol and in bran, brown and white rice with acetonitrile. After liquid/liquid partition, solid-phase extraction and gel-permeation chromatography, residues are quantified by GLC with an electron capture detector. The LOQs were 0.005-0.01 mg/kg in green plant, 0.005 mg/kg in straw and 0.001 mg/kg in brown and white rice and bran.

In a method to determine residues of fipronil and its metabolites in agricultural maize and its processed commodities (Upalawanna, 1993; Baillargeon, 1995a,b, 1996), residues are extracted from grain and fodder with 75% actonitrile/25% water and from forage, crude oil and starch with acetonitrile. After clean-up by partition with hexane and removal of acetonitrile, residues are extracted into dichloromethane, cleaned up by column chromatography on silica gel and charcoal and determined by gas chromatography with a ^{63}Ni electron capture detector. LOQs for fipronil and MB 45950, MB 46136, fipronil-desulfinyl and RPA 200766 were estimated by rounding up to the highest LOQ of any analyte in a substrate and applying that value to all analytes. They were 0.01 mg/kg in grain and crude oil and 0.02 mg/kg in forage, fodder and starch. No major interferences were found from the 45 compounds tested.

In the method of Maycey and Savage (1992) to determine residues of fipronil, MB 45950, MB 46136, fipronil-desulfinyl, and RPA 200766 in bananas residues are extracted with methanol, cleaned up using solid-phase extraction followed by silica column chromatography, and quantified by electron capture gas chromatography with a megabore capillary column. The LOQ was 0.01 mg/kg.

In addition to the method of Baillargeon and Plaisance (1998) which determined residues of fipronil and its metabolites in potato tubers and their processed commodities, an earlier method was validated (Baillargeon, 1995c; Shaffer, 1995). The residues are extracted with acetonitrile, cleaned up by liquid-liquid partition and alumina and silica gel column chromatography, and quantified by GLC with an electron-capture detector. The LOQ was 0.05 mg/kg, and recoveries were 85% (mean of five levels).

Animal material

The multi-residue enforcement analytical method DFG S19 was modified and validated for the determination of residues of fipronil, MB 45950, MB 46136 and fipronil-desulfinyl in bovine muscle, milk and fat, and chicken eggs (Haussman, 1999). The modified method in outline is as follows:

- extraction with water/acetone
- partition into an organic phase after addition of ethyl acetate/cyclohexane (for bovine muscle and milk)
- extraction (2): add acetonitrile/acetone (for bovine fat and eggs)
- gel permeation chromatography
- silica gel column fractionation
- GLC with electron capture detection.

Samples were fortified at 0.002 mg/kg (LOQ) and 0.02 mg/kg with the four analytes, and the method successfully validated in bovine muscle, milk and fat and chicken eggs at these levels. Recoveries ranged from 70% to 108%.

A method of analysis was developed and validated by Wargo (1997) to determine residues of fipronil and its sulfide and sulfone animal metabolites MB 45950 and MB 46136 as the sulfone MB 46136, and the photolysis product fipronil-desulfinyl as a separate entity in bovine milk, muscle, liver, fat and kidney and poultry eggs, muscle, liver, and skin and fat. Residues are extracted with 30:70% acetone/acetonitrile. After column chromatography, the extract is treated with sodium periodate and rhuthenium trichloride to oxidize the sulfide and sulfoxide to sulfone MB 46136. MB 46136 and fipronil-desulfinyl are processed through the method intact, and quantified by gas chromatography using a ^{63}Ni electron capture detector. The performance criteria for validation included LOQ and MLD determination, accuracy, precision, extraction efficiency, linearity, specificity and ruggedness. A confirmatory GC-MSD method was also validated. The method was assessed from average recoveries from all nine substrates; fortification levels ranged from 0.005 to 0.055 mg/kg each of fipronil, MB 45950, MB 46136 and fipronil-desulfinyl. Fipronil-desulfinyl and MB 46136 were both quantified at 0.005 mg/kg. In addition, recoveries of MB 46136 from fortifications with 0.002 mg/kg each of MB 45950, fipronil and MB 46136 were determined. The method is capable of quantifying fipronil-desulfinyl and MB 46136 (intact or from oxidation of MB 45950 and/or fipronil) at less than 0.005 mg/kg each in animal tissues. The MLD was estimated to be approximately 0.0004 mg/kg.

In an individual analyte method to determine residues of fipronil, MB 45950 and MB 46136 in muscle, fat, liver, kidney, milk and eggs the residues are extracted from substrates with 30% acetone in acetonitrile. After column clean-up, residues are quantified by GLC using a ^{63}Ni electron capture detector. Chicken eggs, cattle kidney and fat were used as representative substrates for validation. No interferences were found from the 115 compounds with tolerances in animal substrates that were tested. The LOQ was 0.01 mg/kg for each compound in all the substrates. The method was independently validated (Yarko and Davis, 1994).

An independent laboratory validation of "Method of analysis for the determination of fipronil, (MB 45950 and MB46136) in milk, eggs, liver, kidney, muscle and fat tissues" (Hudson, 1994, Robinson, 1995) was carried out according to US EPA requirements (Yarko and Davis, 1994) by measuring recoveries from beef fat fortified at two levels, using the original version of the method (18 October, 1993): 0.01 and 0.05 mg/kg of fipronil and MB 45950, and 0.06 and 0.3 mg/kg of MB46136. In general the results demonstrated no background interference and recoveries were all within the acceptable limits of 70%-120%.

Soil

Methods have been developed for the determination of fipronil, MB 46136, MB 45950, fipronil-desulfinyl and RPA 200766 in soil (Ibrahim, 1992). The basic method can be adapted to any soil substrate by modifying clean-up procedures to remove chromatographic interference. Residues are extracted from the soil with acetonitrile/acetone (70/30), the sample is centrifuged and the extract dried with sodium sulfate, the analytes are adsorbed onto activated charcoal and eluted with acetonitrile. Quantification is by GLC with electron capture detection. The LOQ is 0.005 mg/kg.

Stability of residues in stored analytical samples

Animal substrates

The results of the studies of the storage stability of fipronil, MB 45950 and MB 46136 in animal commodities (Byrd, 1994a,b) are shown in Table 49. Samples of each of the five substrates were fortified at 0.1 mg/kg with a standard solution containing fipronil and its metabolites.

Table 49. Storage stability in animal substrates, unadjusted for procedural recoveries (Byrd, 1994a,b).

Animal	Sample	Storage temp. (°C)	Storage, months	% remaining, average of 3 samples		
				Fipronil	MB 45950	MB 46136
Cow	Milk	fresh spike	0	90	86	104
		≤-10	1	92	94	99
		≤-10	3	85	88	97
	Liver	fresh spike	0	96	88	106
		≤-10	1	90	92	97
		≤-10	3	83	79	93
	Kidney	fresh spike	0	87	91	92
		≤-10	1	88	90	89
		≤-10	3	82	78	107
	Muscle	fresh spike	0	94	91	104
		≤-10	1	86	90	93
		≤-10	3	83	84	92
	Fat	fresh spike	0	86	84	92
		≤-10	1	84	88	91
		≤-10	3	81	87	86
Hen	Egg	fresh spike	0	79	83	84
		≤-10	1	89	84	100
		≤-10	3	78	82	82
	Liver	fresh spike	0	82	85	88
		≤-10	1	92	87	100
		≤-10	3	77	84	86
	Muscle	fresh spike	0	88	88	96
		≤-10	1	82	83	92
		≤-10	3	78	79	83
	Skin	fresh spike	0	85	87	90
	with fat	≤-10	1	89	87	100
		≤-10	3	81	78	81

In addition as a part of the study on cows (Byrd, 1994a) the storage stability of refrigerated analytical standards was investigated. The results indicate that all the compounds are stable for at least 4 months.

Plant substrates and processed fractions

The stabilities of fipronil, MB 45950, MB 46136 and fipronil-desulfinyl in plant commodities are shown in Tables 50-54.

Table 50. Storage stability in lettuce spiked with 1 mg/kg of each compound (Plaisance, 1998).

Sample	Storage temp. (°C)	Months of storage	% remaining, 2 samples			
			Fipronil	MB 45950	MB 46136	fipronil-desulfinyl
Leaves	fresh spike	0	89 96	93 96	103 108	94 98
	- 20	6	95 93	98 93	99 94	97 96
	- 20	12	82 88	93 92	76 80	84 93

The stability of analytical standard solutions was also reported in the study. Acetonitrile solutions were stable at –20°C over 13 months.

Table 51. Storage stability in potato tubers spiked at 0.1 mg/kg (Eng, 1996b).

Storage temp. (°C)	Storage, months	% remaining, 2 samples			
		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl
fresh spike	0	92 87	86 91	78 72	92 89
- 20	6	92 94	85 90	88 90	88 90
- 20	12	72 85	71 82	67 80	76 89
- 20	24	94 116	93 110	91 106	78 106

Table 52. Stability in whole brassica vegetables stored at -20°C and spiked with 0.1 mg/kg of each compound (Keats, 1997h).

Crop	Storage, months	% remaining, 2 samples			
		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl
Broccoli	11	83 83	90 93	80 78	96 93
Cabbage	11	85 97	91 89	84 82	92 89
Cauliflower	12	86 88	85 85	80 76	88 84

Table 53. Storage stability in maize grain, forage, fodder and processed products, spiked with 0.1 mg/kg of each compound (Upalawanna, 1994).

Sample	Storage temp. (°C)	Storage, months	Corrected % remaining[1], 2 samples			
			Fipronil	MB 45950	MB 46136	fipronil-desulfinyl
Grain	fresh spike	0	103 120	95 108	102 118	NA
	<u><</u>-10	5.8	98 118	90 100	98 109	NA

Sample	Storage temp. (°C)	Storage, months	Corrected % remaining[1], 2 samples			
			Fipronil	MB 45950	MB 46136	fipronil-desulfinyl
	≤-10	11.5	88 84	88 86	105 105	NA
Forage	fresh spike	0	106 104	101 99	104 98	NA
	≤-10	5.9	104 102	96 85	110 95	NA
	≤-10	11.6	91 89	81 85	80 83	NA
Fodder	fresh spike	0	93 97	101 107	100 112	NA
	≤-10	6.4	107 99	103 102	103 101	NA
	≤-10	12	91 88	100 93	108 105	NA
Silage	fresh spike	0	109 111	105 107	103 105	NA
	≤-10	5.6	110 101	119 107	122 110	NA
	≤-10	11.6	92 98	91 96	98 92	NA
Crude oil	fresh spike	0	86 92	88 93	84 92	NA
	≤-10	6.1	82 95	78 92	87 98	NA
	≤-10	11.8	116 102	113 105	124 129	NA
Refined oil	fresh spike	0	114 121	105 108	118 128	NA
	≤-10	6.1	110 110	107 116	122 129	NA
	≤-10	11.9	95 109	100 107	109 116	NA
Grain dust	fresh spike	0	116 107	114 110	108 101	NA
	≤-10	6.1	107 110	107 106	122 113	NA
	≤-10	11.7	91 92	92 94	107 108	NA
Meal	fresh spike	0	94 102	91 97	90 100	NA
	≤-10	6.1	84 80	92 86	88 91	NA
	≤-10	11.7	73 79	87 87	92 84	NA
Starch	fresh spike	0	112 116	108 107	103 103	NA
	≤-10	5.9	107 95	103 101	108 104	NA
	≤-10	11.7	90 84	92 97	92 102	NA

[1] All results were corrected for analytical recoveries

Table 54. Storage stability in cotton spiked with 0.1 mg/kg of each compound (Eng, 1996a, 1997).

Sample	Storage temp. (°C)	Storage, months	Corrected % remaining[1], 2 or 3 samples			
			Fipronil	MB 45950	MB 46136	fipronil-desulfinyl
Eng (1996a)						
Ginned seed	fresh spike	0	106 95 92	93 79 79	103 99 105	98 88 76

Sample	Storage temp. (°C)	Storage, months	Corrected % remaining[1], 2 or 3 samples			
			Fipronil	MB 45950	MB 46136	fipronil-desulfinyl
	- 20	5	99 103	99 100	98 107	100 103
	- 20	12	103 102	94 94	103 94	105 106
Hulls	fresh spike	0	108 104 107	93 94 98	110 104 100	95 99 107
	- 20	5	88 111	97 110	86 121	88 95
	- 20	12	103 106	87 91	92 102	99 102
Meal	fresh spike	0	99 93 83	92 89 82	108 99 83	93 95 85
	- 20	5	93 91	93 95	92 89	91 91
	- 20	12	102 95	99 94	102 96	110 104
Crude oil	fresh spike	0	124 102 102	110 94 97	118 99 104	113 99 103
	- 20	5	104 104	106 106	102 104	101 103
	- 20	12	109 105	101 102	110 102	104 107
Refined oil	fresh spike	0	117 115 110	116 111 100	115 112 111	110 111 97
	- 20	5	104 102	98 97	100 94	103 100
	- 20	12	101 98	99 95	99 95	99 93
Eng (1997)						
Gin trash	Fresh spike	0	96 93 86	86 88 83	98 99 100	92 04 89
	- 20	5	101 97	101 100	93 94	98 95
	- 20	14	104 99	107 109	104 104	105 100

[1] All results were corrected for analytical recoveries

The stability of analytical standard solutions was also reported in the studies (Eng, 1996a, 1997). It was determined that the acetonitrile solutions were stable at -20°C over 15 months.

In a study to examine the stability of residues in maize and cotton samples after a freeze/thaw cycle (Reed, 1998), residues were shown to be stable for at least 9 to 10 days when stored under ambient conditions.

Definition of the residue

Toxicological background. Fipronil was evaluated for toxicology by the 1997 and the 2000 JMPR. The 1997 Meeting concluded that the mammalian metabolites have toxicities comparable to or substantially less than that of fipronil. Because the photodegradation product fipronil-desulfinyl (fipronil-desulfinyl) is of toxicological concern but not a mammalian metabolite of fipronil, it was reviewed separately.

The 1997 JMPR established an ADI of 0-0.0002 mg/kg bw for fipronil, and considered that a separate ADI should be established for fipronil-desulfinyl because it could be a significant residue and its toxicity is greater than that of fipronil. A temporary ADI of 0.00003 mg/kg bw was established for fipronil-desulfinyl, and an acute RfD of 0.003 mg/kg bw for both fipronil and fipronil-desulfinyl.

The 2000 JMPR revised the above and established a group ADI of 0-0.0002 mg/kg bw for fipronil and fipronil-desulfinyl alone or in combination. The acute RfD established by the 1997 JMPR for fipronil and fipronil-desulfinyl alone or in combination was confirmed. Other toxicologically significant compounds are fipronil sulfone (MB 46136) and fipronil sulfide (MB 45950). The Meeting concluded that the metabolite RPA 200766 is significantly less toxic than fipronil and the acknowledged relevant metabolites MB 45950 and MB 46136 as well as the degradation product fipronil-desulfinyl. For this reason, RPA 200766 should not be relevant for dietary risk assessment.

Plant products. Studies of plant metabolism have shown that for soil-incorporated uses, residues of the parent and MB 46136 account for most of the residue, with MB 45950 levels being generally very low.

After foliar application the majority of the residues in human food items (cabbage, potato tubers) consisted of the parent compound and the photodegradation product fipronil-desulfinyl, whereas in animal feed items (rice straw, husk, bran), the parent compound, MB 46136, fipronil-desulfinyl and MB 45950 are potential residues for consideration.

The results of supervised residue trials indicated that the parent compound is the main component of the residue. The Meeting concluded that fipronil should be a good indicator compound for enforcement purposes in plant commodities.

The Meeting concluded that for chronic and acute dietary risk assessment purposes, the residue in plant commodities should be defined as the sum of fipronil, MB 46136, fipronil-desulfinyl and MB 45950, expressed as fipronil.

Animal products. In a goat metabolism study fipronil and the metabolites MB 46136 and MB 45950 were identified as the principal compounds. In a laying hen metabolism study, MB 46136 was identified as the main component of the total radioactive residue in eggs and tissues. The results of feeding studies with fipronil on cows and hens show that most of the residue in milk, eggs and tissues consisted of MB 46136.

The Meeting concluded that the definition of the residue in animal commodities for enforcement purposes should be the sum of fipronil and MB 46136, expressed as fipronil. For long- and short-term dietary risk assessment purposes, the residue should be defined as the sum of fipronil, MB 46136, fipronil-desulfinyl and MB 45950, expressed as fipronil.

Summary - definition of the residue

- For compliance with MRLs for plant commodities: fipronil.
- For compliance with MRLs for animal commodities: sum of fipronil and MB 46136, expressed as fipronil.
- For estimation of chronic and acute dietary intake for plant and animal commodities: sum of fipronil, fipronil-desulfinyl, MB 46136 and MB 45950, expressed as fipronil.

The residue is fat-soluble.

USE PATTERN

Fipronil belongs to a new class of insecticides known as phenylpyrazoles. The main pests controlled are shown in Table 55.

Table 55. Main pests controlled by fipronil.

Crop	Main pests controlled	Application timing
Bananas	banana weevil borer, rust and flower thrips	mat application at any time relative to fruit stage depending upon pest pressure; bud injection after emergence of flower bud
Brassica vegetables	Diamondback moth, white butterfly, cabbage looper, leaf miner, flea beetle, thrips	1 to multiple foliar applications a year, typically depending upon pest pressure and official recommendations
Cereals (rye, wheat, barley)	wireworm, wheat bulbfly, cereal leaf beetle	seed treatment; 1 to 2 foliar applications per year
Cotton	bollworm, boll weevil, plant bugs, thrips, false wireworm, leafworm, green mirid	seed treatment; 1 to multiple foliar applications a year, typically depending upon pest pressure and official recommendations
Maize	maize rootworm, wireworm, maize borers, agricultural termites, click beetle, leaf weevil	seed treatment; at plant in furrow application; ppi[1] soil broadcast application; 1 to 2 foliar applications per year
Pasture grasses	grasshoppers and locusts, beetle, click beetle, stalk borer	1 to multiple broadcast or barrier foliar applications a year, typically depending upon pest pressure and official recommendations
Potato	wireworm, rootworm, thrips, weevils, Colorado potato beetle	at plant in furrow application; ppi banded or broadcast soil application; 1 to multiple foliar applications a year, typically depending upon pest pressure and official recommendations
Rice	rice weevils, stem borers, plant hoppers, leaf folders, gall midges, thrips, bloodworm, maggots	seed treatment; ppi broadcast soil application; nursery box treatment; post transplant broadcast granules in flooded paddy; 1 to 3 foliar applications a year, typically depending upon pest pressure and official recommendations
Sorghum	wireworm, chinch bug, black earwig	ppi soil broadcast application; seed treatment
Sugar beet	click beetle, grubs, wireworm, leaf weevil, root weevil	seed treatment; ppi broadcast soil application; at plant in furrow application; 1 to 2 foliar applications per year
Sugar cane	termites, borers, weevils, beetles, hoppers	at plant in furrow application; inter-row spray application post planting
Sunflower	wireworm, leaf weevil	ppi soil broadcast application; seed treatment

[1] ppi: pre-plant soil incorporation

The following Tables give registered use patterns for the use of fipronil on bananas (Table 56), brassica vegetables (Table 57), root and tuber vegetables (Table 58), cereals (Table 59), oilseeds (Table 60), and pasture grass and sugar cane (Table 61). Because fipronil is a highly effective insecticide on many insect pests, application rates have been expressed as grams of active ingredient rather than kilograms. For spray applications expressed as g ai/ha where application volumes have been recommended on the label, g ai/100 l values have been calculated. Unless otherwise specified, application is outdoor/field only. Copies of the associated national labels were submitted.

Table 56. Uses of fipronil on bananas.

Country	Formulation	Application				PHI
		Method	g ai/ha	g ai/100 l	No.	days
Australia	WG 800 g/kg; SC 200 g/l	soil/lower stem spray	~300 (0.12-0.2 g ai/ stool)	24-40	not specified; CP 2	0
Cameroon	GR 5 g/kg	soil broad-cast at base of plant	300-400 (0.15-0.2 g ai/plant)		not specified	not specified
Ivory Coast	GR 5 g/kg	soil broad-cast at base of plant	300-400 (0.15-0.2 g ai/plant)		not specified	not specified
France (Guadeloupe)	GR 5 g/kg	apply at base of plant, in-corporate	~400 (0.2 g ai/plant)		not specified	not specified
Myanmar	GR 3 g/kg	soil broad-cast at base of plant	90-300 (0.045-0.15 g ai/plant)		not specified; CP 1	7
Philippines	SC 50 g/l	bud injection	0.02 g ai/bud (70 ml diluted suspension)	30	not specified; CP 1	not specified

CP: common practice

Table 57. Uses of fipronil on brassica vegetables. All foliar applications.

Crop	Country	Formulation	Application			PHI
			g ai/ha	g ai/100 l	No.	days
Brassicas, general	Ivory Coast	SC 50 g/l	50 -75	10-37.5	not specified	7
	Kenya	SC 50 g/l	50	6.25-12.5	not specified	3
	Myanmar	SC 50 g/l	25-50	2.8-5.6	not specified; WCP 4-5	7
Broccoli	Australia	WG 800 g/kg; SC 200 g/l	24-48 50	5-12.5	4	7
	Ecuador	WG 800 g/kg	31.5-62.5		not specified	45
Brussels sprouts	Australia	WG 800 g/kg; SC 200 g/l	24-48 50	5-12.5	4	7
Cabbage	Australia	WG 800 g/kg; SC 200 g/l	24-48 50	5-12.5	4	7
	Colombia	WG 200 g/kg	48		not specified	not specified
	Ecuador	WG 200 g/kg	31.5-62.5		not specified	45
	India	SC 50 g/l	40-50	8-10	2-3	7
	Indonesia	SC 50 g/l	12.5-25	2.5-5	not specified; WCP 6-8	14
	Malaysia	SC 50 g/l	36	4.5	not specified; WCP 4-6	5
	New Zealand	SC 200 g/l	24	4.8	4	7
	Panama	WG 200 g/kg; SC 200 g/l	50	16.7-25 by ground	not specified (but requires 15 day spray interval)	not specified
	Peru	SC 200 g/l	40-50	11-12.5	not specified (but requires 15 day spray interval)	14
	Philippines	SC 50 g/l	25-50	5-16	not specified; WCP 6-8	7
	Taiwan	SC 50 g/l	12.5-25	2.5	not specified; WCP 3-4	9
	Venezuela	SC 200 g/l	15-25		not specified	14
Cauliflower	Australia	WG 800 g/kg; SC 200 g/l	24-48 50	5-12.5	4	7
	Ecuador	WG 800 g/kg	31.5-62.5		not specified	45
	Peru	SC 200 g/l	40-50	11-12.5	not specified	14
Crucifers, general	China	SC 50 g/l	12.8-24.8	1.3-4.1	not specified; WCP 4	not specified
	Thailand	SC 50 g/l	50-200	5	not specified; WCP 3-4	7
	Vietnam	WG 800 g/kg	25.6	8.5	not specified; WCP 5	15
Crucifers, leafy	Malaysia	SC 50 g/l	36	4.5	not specified; WCP 4-6	5
Kohlrabi	Australia	WG 800 g/kg; SC 200 g/l	24-48 50	5-12.5	4	7

WCP: worst case practice; estimated from field development expertise and observation

Table 58. Uses of fipronil on root and tuber vegetables.

Crop	Country	Formulation	Application				PHI
			Method	g ai/ha	g ai/100 l	No.	days
Potato	Australia	SC 200 g/l	soil broadcast ppi spray	50		1	NA
	Belarus/Russia	EC 25 g/l	foliar	12.5-15	2.5	2	30
	Belarus/Russia	WG 800 g/kg	foliar	16-20	4-10	2	30
	Brazil	GR 20 g/kg	soil, in furrow at planting	100		1	NA
	Colombia	SC 200 g/l	soil in furrow spray or hill spray	120		2	not specified
	Czech/Slovak	WG 800 g/kg	foliar	20	6.67-10	not specified, CP 2	14
	Ecuador	WG 800 g/kg	soil spray	50-100		2, 2nd application at hilling	30
	Hungary	WG 800 g/kg	foliar	20	4-6.7 by ground	not specified, CP 1-2 (requires 2-3 wk spray interval)	14
	Indonesia	SC 50 g/l	foliar	12.5-50	2.5-5	not specified, CP 2-3	14

Crop	Country	Formulation	Application				PHI
			Method	g ai/ha	g ai/100 l	No.	days
	Italy	GR 20 g/kg	soil, in furrow at planting	150		1	NA
	Myanmar	GR 3 g/kg	soil broadcast or incorporated	75-110		not specified, CP 1	14
	Myanmar	SC 50 g/l	foliar	unclear	2.5-5	4-5	14
	Panama	SC 200 g/l WG 800 g/kg	at plant soil spray or foliar	50-150	16.7-50 by ground	not specified, CP 1-2 (requires 15d spray interval)	not specified
	Peru	SC 200 g/l	soil spray	50-100	12.5-25	not specified	14
	Poland	SC 200 g/l	foliar	20	5-13.3	not specified	14
	Romania	SC 200 g/l	foliar	18-20	2.25-5	3	30
	Spain	WG 800 g/kg	foliar	20-24	2.25-6	3	14
	Turkey	WG 800 g/kg	foliar	16		2	14
	Ukraine	EC 25 g/l	foliar	16		1	28
	Ukraine	WG 800 g/kg	foliar	16-20		2	20
Sugar beet	Belgium	GR 14 g/kg with aldicarb	soil, in furrow at planting	77-154		1	NA
	Belgium GAP pending	WG 800 g/kg	broadcast soil ppi spray	200		1	NA
	Chile	FS 250 g/l	seed treatment	200-400 g ai/ 100 kg seed		1	NA
	France	GR 14 g/kg with aldicarb	soil, in furrow at planting	160		1	NA
	France	WG 800 g/kg	broadcast soil ppi spray	200		1	NA
	Hungary	WG 800 g/kg	foliar	20-24	4-8 by ground	not specified, CP 1-2 (requires 2-3 wk spray interval)	30
	Italy	GR 20 g/kg	soil, in furrow at planting	150		1	NA
	Romania	FS 250 g/l	seed treatment	375 g ai/100 kg seed		1	NA
	Romania	SC 200 g/l	foliar	20	2.25-5	3	30
	Ukraine	FS 500 g/l	seed treatment	25 g ai/100,000 seeds		1	NA

NA: not applicable
CP: common practice

Table 59. Uses of fipronil on cereals.

Crop	Country	Formulation	Application				PHI
			Method	g ai/ha	g ai/100 l	No.	days
Barley	Belarus/ Russia	WG 800 g/kg	Foliar	16	4-8	1	30
Cereals, general	Czech/Slovak	WG 800 g/kg	foliar	20	6.66-10	not specified; CP 1	44
	France	FS 250 g/l plus combination products w/fungicides	seed treatment	100 (50 g ai/ 100 kg seed)		1	NA
	Hungary	WG 800 g/kg	foliar	12	2.4-4	not specified, CP 1-2, (requires 2-3 wk spray interval)	30
	Romania	SC 200 g/l	foliar	18	2.25-4.5	3	30
	Switzerland	FS 500 g/l	seed treatment	Only bag label warning put on treated seed imported into Switzerland from other countries			

Crop	Country	Formulation	Application				PHI
			Method	g ai/ha	g ai/100 l	No.	days
	Turkey	WG 800 g/kg	foliar	20		1	not specified; pest appears during or after tillering
Maize	Belgium	FS 500 g/l GAP pending	seed treatment	40 g ai/50,000 seeds		1	NA
	Belgium	WG 800 g/kg GAP pending	soil broad-cast ppi spray	200		1	NA
	Chile	FS 250 g/l	seed treatment	100-125 g ai/ 100 kg seed		1	NA
	Ivory Coast	SC 50 g/l	soil spray at planting, incorporated	100-200	20-100	1	NA
	France	FS 500 g/l	seed treatment	50-70 (250 g ai/100 kg seed; 27.5 g ai / 50,000 seeds)		1	NA
	France	GR 20 g/kg (w/aldicarb)	soil, in furrow at planting	160		1	NA
	France	WG 800 g/kg	soil broad-cast ppi spray	200		1	NA
	Hungary	WG 800 g/kg	foliar	20	4-6.7 by ground	not specified, CP 1-2 (requires 2-3 wk spray interval)	30
	Italy	FS 500 g/l	seed treatment	250 g ai/100 kg seed (15 g ai / 25,000 seeds)		1	NA
	Italy	GR 20 g/kg	soil, in furrow at planting	100-150		1	NA
	Kenya	GR 3 g/kg	soil, in furrow at planting	90		1	90
	Mexico	GR 20 g/kg	soil, in furrow at planting	200		1	NA
	Mozambique	FS 250 g/l	seed treatment	100 (400 g ai/100 kg seed)		1	NA
	Mozambique	GR 3 g/kg	soil, in furrow at planting	99		1	NA
	Mozambique	SC 200 g/l	soil, in furrow spray at planting	100		1	30
	Myanmar	GR 3 g/kg	soil broad-cast or incorporated	75-110		1	7
	Myanmar	SC 50 g/l	soil spray	100-200	0.2-0.4	1	7
	Panama	SC 200 g/l	soil ppi spray or seed treatment	100	33.3-50 by ground	1	NA
	Panama	WG 800 g/kg	soil ppi spray or seed treatment	50-100	16.7-50 by ground	1	NA
	Romania	FS 250 g/l	seed treatment	125 g ai/100 kg seed		1	NA
	Switzerland	FS 500 g/l	seed treatment	Only bag label warning put on treated seed imported into Switzerland from other countries			
	Turkey	FS 500 g/l	seed treatment	25 g ai/100 kg seed		1	NA
	Ukraine	FS 500 g/l	seed treatment	100 g ai/100 kg seed		1	NA
	USA	GR 15 g/kg	soil, in furrow at planting	112-146		1	NA
	USA	SC 400 g/l WG 800 g/kg	soil, in furrow spray at planting	112-146		1	90
	Zimbabwe	FS 250 g/l FS 500 g/l	seed treatment	400 g ai/100 kg seed		1	NA
	Zimbabwe	SC 200 g/l	soil, in furrow at planting or broad-cast ppi spray	50-150		1	NA
Rice	Argentina	FS 250 g/l	seed treatment	30 g ai/100 kg seed		1	NA
	Australia	FS 500 g/l	seed treatment	12.5 (10 g ai/ 100 kg seed)		1	NA

Crop	Country	Formulation	Application				PHI
			Method	g ai/ha	g ai/100 l	No.	days
	Bangladesh	GR 3 g/kg	broadcast into paddy	30		1 within 25 d post transplant	not specified
	Bangladesh	SC 50 g/l	foliar	25	5	not specified, CP 1-3	7
	Brazil	FS 250 g/l	seed treatment	62.5 g ai/100 kg seed		1	NA
	China	FS 50 g/l	seed treatment	16-125 (0.8-4.5 g ai/kg seed, seeding rate 20-30 kg/ha)		1	NA
	China	GR 3 g/kg	nursery or broad-cast into paddy	49.5-81		1 just before or after sowing or transplant	not specified
	China	SC 50 g/l	foliar	22.5-45	3-10	not specified, CP 1-2	not specified
	Colombia	SC 200 g/l	foliar	60-70		not specified	not specified
	Colombia	WG 800 g/kg	foliar	50-60		not specified	not specified
	France	FS 500 g/l GAP pending	seed treatment	25 (12.5 g ai/ 100 kg seed)		1	NA
	India	GR 3 g/kg	broadcast into paddy	50-75		1	32
	India	SC 50 g/l	foliar	50-75	10-12.5	1 from 25-30d post transplant	32
	Indonesia	FS 50 g/l	seed treatment	12.5-25 (0.6-1.25 g ai/kg seed)		1	NA
	Indonesia	GR 3 g/kg	broadcast into paddy	30		2	25
	Indonesia	SC 50 g/l	foliar	12.5-25	2.5-8.33	not specified, CP 1-2	14
	Ivory Coast	GR 3 g/kg	broadcast into paddy	45		2, 2nd appl. 42 days after sowing or 30 days after transplant	not specified
	Ivory Coast	SC 50 g/l	foliar	25-75	5-37.5	not specified	7
	Japan	GR 10 g/kg	broadcast nursery box	100 (0.5 g ai/box)		1, 3 d before to day of transplant	NA
	Madagascar	SC 50 g/l	foliar	50	25	1	50
	Malaysia	GR 3 g/kg	broadcast into paddy	25-50		1, 15-30 d post sowing	90 (14d proposed)
	Myanmar	GR 3 g/kg	broadcast into paddy	37-75		1, 5-30 d post sowing	7
	Myanmar	GR 3 g/kg	nursery box	0.15 g ai/box		1	NA
	Myanmar	SC 50 g/l	foliar	25-50		not specified, CP 1	7
	Panama	SC 200 g/l	foliar	50-150	16.7-75 by ground; 50-150 by air	not specified, CP 1-2 (requires 15 d spray interval)	not specified
	Panama	WG 800 g/kg	foliar	50-100	16.7-50 by ground; 50-100 by air	not specified, CP 1-2 (requires 15 d spray interval)	not specified
	Philippines	GR 3 g/kg	broadcast into paddy	30-51		1 no more than 45 d post transplant or 55 d post seeding	not specified
	Philippines	SC 50 g/l	foliar	20-30	3.9-12	not specified, CP 1	7
	Sri Lanka	GR 3 g/kg	broadcast into paddy	36		not specified, CP 1	not specified
	Sri Lanka	SC 50 g/l	foliar	9	2-2.5	not specified	5
	Taiwan	GR 3 g/kg	broadcast into paddy	60		2, last one at tillering	not specified
	Taiwan	GR 3 g/kg	nursery box	0.15 g ai/ box		1	NA

Crop	Country	Formulation	Application				PHI
			Method	g ai/ha	g ai/100 l	No.	days
	Thailand	GR 2 or 3 g/kg	broadcast into paddy	25-50		1-2	7[1]
	Thailand	SC 50 g/l	foliar	25-62.5	5-12.5	not specified, CP 1-2	7[1]
	USA	FS 750 g/l	seed treatment	28-56 (60-120 g ai/100 kg seed)		1	NA
	USA	SC 750 g/l WG 800 g/kg	soil ppi spray	28-56	0.1-0.6 by ground; 1.2-2.4 by air	1	NA
	Venezuela	FS 250 g/l	seed treatment	56 g ai/100 kg seed		1	NA
	Venezuela	SC 200 g/l	foliar	62		not specified	7
	Vietnam	GR 3 g/kg	broadcast into paddy	30		not specified, CP 1	not specified
	Vietnam	WG 800 g/kg	foliar	26	8	not specified, CP 1	15
Sorghum	Australia	FS 500 g/l GAP pending	seed treatment	75 g ai/100 kg seed		1	NA
	Australia	SC 200 g/l WG 800 g/kg	foliar	1.25	3-7.5	not specified	14
	Australia	UL 8.5 g/l	foliar (aerial)	1.3		not specified	14
	France	WG 800 g/kg	soil broadcast ppi spray	200		1	NA
	Myanmar	GR 3 g/kg	soil broadcast or incorporated	75-111		1	7
	Panama	SC 200 g/l WG 800 g/kg	foliar or seed treatment	25-30	8.3-15 by ground	not specified, CP 1-2 (requires 15d spray interval)	not specified
Wheat	Belarus/ Russia	WG 800 g/kg	foliar	18-24	4.5-12	1	30
	Belgium	FS 125 g/l (in combination w/ fungicides)	seed treatment	50 g ai/100 kg seed		1	NA
	Chile	FS 250 g/l	seed treatment	50-100 g ai/100 kg seed		1	NA
	Romania	SC 200 g/l	foliar	15-18 (depending on pest)	1.88-4.5	3	30

NA: not applicable
CP: common practice
[1] The label PHI of 7 days represents the standard PHI for fipronil in Thailand. However, the pest spectrum dictates the timing of applications. Fipronil is used in Thailand on rice to control thrips, leaffolder, stemborer and brown plant hopper. Common practice is 1-2 foliar applications at 12.5-37.5 g ai/ha made from 7 to a maximum 60 days after planting (rice is harvested 110-120 days after planting).

Table 60. Uses of fipronil on oilseeds.

Crop	Country	Formulation	Application				PHI
			Method	g ai/ha	g ai/100 l	No.	days
Cotton	Australia	FS 500 g/l	seed treatment	50 g ai/100 kg seed		1	NA
	Australia	SC 200 g/l WG 800 g/kg	foliar	12.5-24	1.3-70	not specified, CP 1-2	28
	Bolivia	WG 800 g/kg	foliar	12-80 ground 24-80 air	3-80 ground; 75-400 air	4-7 at 7 day intervals WCP 2-3	15
	Brazil	WG 800 g/kg	foliar	12-80 ground 24-80 air	3-80 ground; 75-400 air	4-7 at 7 day intervals; WCP 2-3	15
	Colombia	SC 200 g/l	foliar	65-70	172-308 air	not specified; WCP 2-3	not specified
	Colombia	WG 800 g/kg	foliar	64-68		not specified; WCP 2-3	not specified

Crop	Country	Formulation	Application				PHI days
			Method	g ai/ha	g ai/100 l	No.	
	Ivory Coast	SC 50 g/l	foliar	12.5-80	2.5-40	not specified	7
	Mexico	SC 200 g/l	foliar	50		2 at 7 day intervals	45
	Mexico	WG 800 g/kg	foliar	32-48		2 at 5-7 day intervals	45
	Mozambique	FS 250 g/l	seed treatment	100 (400 g ai/100 kg seed)		1	NA
	Mozambique	GR 3 g/kg	soil, in furrow at planting	99		1	NA
	Myanmar	SC 50 g/l	foliar	12.5-370		not specified, CP 1	7
	Paraguay	WG 800 g/kg	foliar	12-80	3-80 ground; 30-400 air	4-7 at 7 day intervals; WCP 2-3	15
	Peru	SC 200 g/l	foliar	60-70	15-18 ground; 120-150 air	not specified; WCP 2-3	14
	USA (GAP pending)	EC 300 g/l WG 800 g/kg	foliar	28-56	12-188	maximum 224 g ai/ ha/ season	60
	Venezuela	SC 200 g/l	foliar	70		not specified; WCP 2-3	15
	Zimbabwe	FS 250 g/l FS 500 g/l	seed treatment	400 g ai/100 kg seed		1	NA
	Zimbabwe	SC 200 g/l	soil, in furrow spray at planting	100		1	NA
Sunflower	Australia	FS 500 g/l GAP pending	seed treatment	75 g ai/100 kg seed		1	NA
	France	FS 500 g/l	seed treatment	50-70 (500 g ai/100 kg seed)		1	NA
	France	GR 20 g/kg w/aldicarb	soil in furrow	160		1	NA
	France	WG 800 g/kg	broadcast soil ppi spray	200		1	NA
	Italy	GR 20 g/kg	soil, in furrow at planting	100-150		1	NA
	Myanmar	GR 3 g/kg	soil broadcast or incorporated	111-148		not specified, CP 1 at planting	7
	Romania	FS 250 g/l	seed treatment	125 g ai/100 kg seed		1	NA
	Ukraine	FS 500 g/l	seed treatment	100 g ai/100 kg seed		1	NA

NA: not applicable
CP: common practice
WCP: worst-case practice

Table 61. Uses of fipronil on pasture grasses and sugar cane.

Crop	Country	Formulation	Application				PHI days
			Method	g ai/ha	g ai/100 l	No.	
Grassland	China	UL 4 g/l	foliar	4-8		not specified	not specified
Uncultivated land	Ethiopia*	UL 12.5 g/l	foliar	1-6.25		not specified	7
	Mozambique	UL 7.5 g/l	foliar	7.5		1	21
	Sudan	UL 12.5 g/l	foliar	1-6.25		not specified	7
	Madagascar	UL 4 g/l	foliar	2-4		not specified	3
Pasture	Australia	UL 8.5 g/lSC 200 g/l WG 800 g/kg	foliar	1.3		not specified	14
	South Africa	UL 2, 3, 5 & 7.5 g/l	foliar	7.5		not specified	21

Crop	Country	Formulation	Application				PHI days
			Method	g ai/ha	g ai/100 l	No.	
Sugar cane	Australia	SC 200 g/l WG 800 g/kg	inter row directed spray to soil and bottom 40 cm stalk	75	37.5	1	84
	Bangladesh	GR 3 g/kg	soil, in furrow at planting	100		1	NA
	Bangladesh	SC 50 g/l	soil, in furrow drench at planting	100	20	1	NA
	Bolivia	WG 800 g/kg	soil, in furrow spray at planting	200	50-200	1	NA
	Brazil	GR 20 g/kg	soil, in furrow at planting	200		1	NA
	Brazil	WG 800 g/kg	soil, in furrow spray at planting	160-200	80-200	1	NA
	China	GR 3 g/kg	soil, in furrow at planting	100-150		1	NA
	Ivory Coast	SC 50 g/l	soil incorporated spray	50-200	10-100	not specified	7
	Indonesia	FS 50 g/l	cane set dipping	125-250 ppm ai in solution		1	NA
	Indonesia	GR 3 g/kg	soil incorporated	75-150		2	25
	Malaysia	SC 50 g/l	soil, in furrow spray at planting	20	2.5	1	NA
	Mozambique	SC 200 g/l	soil, in furrow spray	100		1	60
	Myanmar	GR 3 g/kg	soil, in furrow at planting	37-111		1	NA
	Myanmar	SC 50 g/l	soil, in furrow spray at planting	100	11	1	NA
	Panama	SC 200 g/l	soil spray or foliar spray	50-60	16.7-30 by ground; 50-60 by air	not specified	not specified
	Panama	WG 800 g/kg	soil spray or foliar spray	50-200	16.7-100 by ground; 50-200 by air	not specified	not specified
	Paraguay	WG 800 g/kg	soil, in furrow spray at planting	160 (2.8 g ai/100 m of furrow)	40-160	1	NA
	Sudan	SC 50 g/l	soil, in furrow spray at planting	120	5	1	NA
	Thailand	SC 50 g/l	soil, in furrow at planting	100	20	1	7
	Zimbabwe	SC 200 g/l	soil, in furrow spray at planting	100	50	1	NA

* the same label is used in Kenya, Tanzania and Uganda

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received supervised trials residue data on plants as well as animal feeding studies. Residue data on bananas, potatoes, sugar beet, vegetables, cereals, grasses and oilseeds are summarized in Tables 62-84.

The concentrations of fipronil and the metabolites MB 45950, MB 46136, fipronil-desulfinyl and RPA 200766 are expressed in the Tables below as individual residues, but in the appraisal were calculated as fipronil according to the appropriate definition of the residue: MB 45950 (421.1 g/mol) factor 1.04, MB 46136 (453.1 g/mol) factor 0.965, fipronil-desulfinyl (389.02 g/mol) factor 1.1 and RPA 200766 (455.08 g/mol) factor 0.96.

Where residues were not detected they are recorded as below the LOQ, e.g. <0.002 mg/kg, except in the US data where "less than the minimum limit of detection (MLD)" is shown; for residues between the MLD and LOQ, <LOQ is shown. The explanation is noted in the Tables containing US data.

Residues, application rates and spray concentrations have generally been rounded to 2 significant figures or, for residues near the LOQ, to 1 significant figure. Residues from trials according to GAP are underlined, those used for the estimation of STMRs and HRs are double underlined.

Bananas (Table 62). The common practice is to apply granules or spray the soil around the base of the plant stem to control banana weevil as pest pressure dictates, generally without incorporation but with incorporation in Guadeloupe. The trials listed in Table 62 were according to common practice.

Table 62. Results of supervised trials on bananas.

Country, Year, reference	Application				PHI, days	Residues, mg/kg				
	Form	g ai/ha	No.	Comments; time between appl.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Australia, 1992-1993, PR93725AT1 Richard and Muller, 1994a	SC	800	1	no decline study, separate treatments, spraying to stem and soil	63	<0.002	<0.002	<0.002	<0.002	<0.002
			1		91	<0.002	<0.002	<0.002	<0.002	<0.002
			1		141	<0.002	<0.002	<0.002	<0.002	<0.002
			1		166	<0.002	<0.002	<0.002	<0.002	<0.002
Australia, 1994, 94663AU1, Richard and Muller, 1995a	SC	300	1	no decline study, separate treatments, spraying to stem and soil	0	<0.002	<0.002	<0.002	<0.002	<0.002
			1		1	<0.002	<0.002	<0.002	<0.002	<0.002
			1		7	<0.002	<0.002	<0.002	<0.002	<0.002
			1		14	<0.002	<0.002	<0.002	<0.002	<0.002
			1		28	<0.002	<0.002	<0.002	NR	<0.002
			1		60	<0.002	<0.002	<0.002	NR	<0.002
Australia, 1994, 94663AU1 Richard and Muller, 1995a	SC	600	1	no decline study, separate treatments, spraying to stem and soil	0	0.003[1] <0.002	<0.002	<0.002	<0.002	<0.002
			1		1	<0.002	<0.002	<0.002	<0.002	<0.002
			1		7	<0.002	<0.002	<0.002	<0.002	<0.002
			1		14	<0.002	<0.002	<0.002	<0.002	<0.002
			1		28	<0.002	<0.002	<0.002	<0.002	<0.002
			1		60	<0.002	<0.002	<0.002	<0.002	<0.002
Australia, 1994, 94663AU2 Richard and Muller, 1995a	SC	300	1	no decline study, separate treatments, spraying to stem and soil	1	<0.002	<0.002	<0.002	<0.002	<0.002
			1		7	<0.002	<0.002	<0.002	<0.002	<0.002
			1		17	<0.002	<0.002	<0.002	<0.002	<0.002
			1		29	<0.002	<0.002	<0.002	<0.002	<0.002
Australia, 1994, 94663AU2 Richard and Muller, 1995a	SC	600	1	no decline study, separate treatments, spraying to stem and soil	1	<0.002	<0.002	<0.002	<0.002	<0.002
			1		7	<0.002	<0.002	<0.002	<0.002	<0.002
			1		17	<0.002	<0.002	<0.002	<0.002	<0.002
			1		29	<0.002	<0.002	<0.002	<0.002	<0.002
Cameroon, 1992-1993, 90CAM04I Richard and Muller, 1993c	GR	200	1		295	<0.002	<0.002	<0.002	<0.002	<0.002
		400	1	soil treatment	295	<0.002	<0.002	<0.002	<0.002	<0.002
Guadeloupe, 1992-1993, GU-NEU 196, 92-319 Meteo III, Richard and Muller, 1993a	GR	400	1	no decline study, separate soil treatments	0	<0.002	<0.002	<0.002	<0.002	<0.002
			1		14	<0.002	<0.002	<0.002	<0.002	<0.002
			1		21	<0.002	<0.002	<0.002	<0.002	<0.002
			1		58	<0.002	<0.002	<0.002	<0.002	<0.002
			1		92	<0.002	<0.002	<0.002	<0.002	<0.002
Guadeloupe, 1992-1993 92-319 GU-NEU 196 Garage IV, Richard and Muller, 1993a	GR	400	1	no decline study, separate soil treatments	0	<0.002	<0.002	<0.002	<0.002	<0.002
			1		14	<0.002	<0.002	<0.002	<0.002	<0.002
			1		21	<0.002	<0.002	<0.002	<0.002	<0.002
			1		58	0.003[2]	<0.002	<0.002	<0.002	<0.002
			1		92	0.025[3]	<0.002	<0.002	<0.002	<0.002

Country, Year, reference	Application				PHI, days	Residues, mg/kg				
	Form	g ai/ha	No.	Comments; time between appl.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Guadeloupe, 1992-1993[2] GU-NEU 196, 92-319, Meteo II, Richard and Muller, 1993a	GR	400	2	interval 6 months, soil treatment	149	≤0.002	≤0.002	≤0.002	<0.002	<0.002
Guadeloupe, 1992-1993[2] GU-NEU 196 Meteo III, 92-319 Richard and Muller, 1993a	GR	400	2	interval 4 months, soil treatment	43	≤0.002	≤0.002	≤0.002	<0.002	<0.002
Guadeloupe, 1992-1993[2] GU-NEU 196, Garage IV, 92-319 Richard and Muller, 1993a	GR	400	2	interval 4 months, soil treatment	43	≤0.002	≤0.002	≤0.002	<0.002	<0.002
Guadeloupe, 1992-1993[2] GU-NEU 196 Meteo II, 92-319 Richard and Muller, 1993a	GR	200	3	interval 3 months, soil treatment	86	<0.002	<0.002	<0.002	<0.002	0.002
Ivory Coast, 1993[2] BACJEX 420 Richard and Muller, 1993b	GR	800	1 1 1 1 1	no decline study, separate soil treatments	3 19 33 63 94	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002
Ivory Coast, 1993[2] BACJEX 420 Richard and Muller, 1993b	GR	1600	1 1 1 1 1	no decline study, separate soil treatments	3 19 33 63 94	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002
Ivory Coast, 1993-1994[2] CAD93I05 Richard and Muller, 1994e	GR	250 500 1000	2 2 2	interval 3 months, soil treatment	78 78 78	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	NR NR NR	<0.002 <0.002 <0.002

NR: not reported owing to analytical problems
[1]Fipronil was reported at 0.003 mg/kg, but the untreated control contained a quantifiable quantity of parent (0.006 mg/kg). Value at day 0 is considered to be contamination.
[2] Three individual field samples of fruit from the lower, middle and upper portions of the bunch were analysed at each interval (in trial 92-319: 0.003 mg/kg is mean of 0.005, 0.002, 0.002 mg/kg).
[3] Contamination, not included in evaluation.

Broccoli and cauliflower (Table 63). Fipronil was applied as a foliar spray, so residues of parent, MB 45950, MB 46136 and the photoproduct fipronil-desulfinyl were reported.

Table 63. Residues from supervised trials in Australia on flowering brassicas. All WG formulation.

CROP Year Reference	Application				PHI, days	Residues, mg/kg				
	g ai/ha	g ai/hl	No.	Interval, days		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Keats, 1996a										
BROCCOLI 1996 AK96052 4/2	12	2	4	7,7,7	0 1 2 3 5 7 14 21	0.026 0.023 0.015 0.007 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	0.003 0.004 0.003 <0.002 <0.002 <0.002 <0.002 <0.002	0.005 0.006 0.003 <0.002 <0.002 <0.002 <0.002 <0.002	NR
BROCCOLI 1996 AK96052 4/3	24	4	4	7,7,7	0 1 2 3 5 7 14 21	0.027 0.025 0.015 0.003 <0.002 <0.002 <0.002 <0.002	0.003 0.003 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	0.004 0.005 0.002 <0.002 <0.002 <0.002 <0.002 <0.002	0.008 0.007 0.003 <0.002 <0.002 <0.002 <0.002 <0.002	NR

CROP Year Reference	Application				PHI, days	Residues, mg/kg				
	g ai/ha	g ai/hl	No.	Interval, days		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
BROCCOLI	48	8	4	7,7,7	0	0.049	0.003	0.005	0.009	NR
1996					1	0.036	<0.002	0.007	0.005	
AK96052 4/4					2	0.013	<0.002	0.003	0.006	
					3	0.008	<0.002	<0.002	0.004	
					5	0.004	<0.002	<0.002	<0.002	
					7	0.006	≤0.002	≤0.002	≤0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
BROCCOLI	96	16	4	7,7,7	0	0.043	0.004	0.006	0.011	NR
1996					1	0.031	0.003	0.005	0.01	
AK96052 4/5					2	0.020	0.002	0.007	0.009	
					3	0.012	<0.002	0.003	0.007	
					5	0.009	<0.002	0.003	0.01	
					7	0.004	<0.002	<0.002	0.005	
					14	0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
BROCCOLI	12	2	6	7,7,7,8,7	0	0.033	0.003	0.003	0.008	NR
1996					1	0.023	<0.002	0.003	0.005	
AK96052 6/2					2	0.011	<0.002	0.002	0.004	
					3	0.008	<0.002	<0.002	0.002	
					5	<0.002	<0.002	<0.002	<0.002	
					7	<0.002	<0.002	<0.002	<0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
BROCCOLI	24	4	6	7,7,7,8,7	0	0.046	0.003	0.004	0.01	NR
1996					1	0.015	<0.002	0.004	0.004	
AK96052 6/3					2	0.014	<0.002	0.003	0.006	
					3	0.009	<0.002	<0.002	0.004	
					5	0.009	<0.002	<0.002	<0.002	
					7	<0.002	<0.002	<0.002	<0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
BROCCOLI	48	8	6	7,7,7,8,7	0	0.049	0.004	0.005	0.014	NR
1996					1	0.036	0.004	0.007	0.02	
AK96052 6/4					2	0.037	0.005	0.008	0.022	
					3	0.008	<0.002	<0.002	0.005	
					5	0.008	<0.002	0.002	0.005	
					7	≤0.002	≤0.002	≤0.002	≤0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
BROCCOLI	96	16	6	7,7,7,8,7	0	0.060	0.006	0.007	0.022	NR
1996					1	0.041	0.005	0.011	0.022	
AK96052 6/5					2	0.041	0.004	0.009	0.021	
					3	0.031	0.004	0.006	0.016	
					5	0.009	<0.002	0.003	0.005	
					7	0.007	<0.002	<0.002	0.004	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
BROCCOLI	12	2	10	7,7,7,8,7, 6,6,8,7	0	0.034	0.003	<0.002	0.011	NR
1996					1	0.014	<0.002	<0.002	<0.002	
AK96052 10/2					2	0.013	<0.002	<0.002	<0.002	
					3	0.005	<0.002	<0.002	<0.002	
					5	0.004	<0.002	<0.002	<0.002	
					7	0.002	<0.002	<0.002	<0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
BROCCOLI	24	4	10	7,7,7,8,7, 6,6,8,7	0	0.029	0.003	0.005	0.01	NR
1996					1	0.016	<0.002	0.002	0.005	
AK96052 10/3					2	0.007	<0.002	<0.002	<0.002	
					3	0.007	<0.002	0.002	0.002	
					5	0.005	<0.002	<0.002	<0.002	
					7	0.003	<0.002	<0.002	<0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	

CROP Year Reference	Application				PHI, days	Residues, mg/kg				
	g ai/ha	g ai/hl	No.	Interval, days		Fipronil	MB 45950	MB 46136	fipronil- desulfinyl	RPA 200766
BROCCOLI 1996 AK96052 10/4	48	8	10	7,7,7,8,7, 6,6,8,7	0 1 2 3 5 7 14 21	0.068 0.055 0.025 0.007 0.007 0.002 <0.002 <0.002	0.005 0.006 0.003 <0.002 <0.002 ≤0.002 <0.002 <0.002	0.008 0.008 0.004 0.004 0.003 ≤0.002 <0.002 <0.002	0.019 0.016 0.009 0.004 0.003 ≤0.002 <0.002 <0.002	NR
BROCCOLI 1996 AK96052 10/5	96	16	10	7,7,7,8,7, 6,6,8,7	0 1 2 3 5 7 14 21	0.079 0.058 0.030 0.021 0.009 0.006 0.004 0.003	0.017 0.006 0.004 0.002 <0.002 <0.002 <0.002 <0.002	0.008 0.01 0.006 0.004 0.004 0.004 0.003 <0.002	0.022 0.019 0.011 0.009 0.004 0.003 0.002 <0.002	NR
Keats, 1996d										
CAULIFLOWER 1995 AUS 94i47aR	12	2.4	2	7	0 1 3 5 7 14 21	0.014 0.009 0.004 0.003 0.003 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	NR
CAULIFLOWER 1995 AUS 94i47aR	24	4.8	2	7	0 1 3 5 7 14 21	0.018 0.014 0.007 0.003 0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	NR
CAULIFLOWER 1995 AUS 94i47aR	48	9.6	2	7	0 1 3 5 7 14 21	0.019 0.016 0.006 0.002 ≤0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 ≤0.002 <0.002 <0.002	0.002 <0.002 <0.002 <0.002 ≤0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 ≤0.002 <0.002 <0.002	NR
CAULIFLOWER 1995 AUS 94i47aR	96	19.2	2	7	0 1 3 5 7 14 21	0.024 0.018 0.009 0.004 0.003 0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	0.004 0.004 <0.002 <0.002 0.002 <0.002 <0.002	<0.002 <0.002 0.002 <0.002 <0.002 <0.002 <0.002	NR
CAULIFLOWER 1995 AUS 94i47aR	12	2.4	4	7,7,7	0 1 3 5 7 14 21	0.013 0.01 0.006 0.003 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	NR
CAULIFLOWER 1995 AUS 94i47aR	24	4.8	4	7,7,7	0 1 3 5 7 14 21	0.016 0.01 0.005 0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	NR
CAULIFLOWER 1995 AUS 94i47aR	48	9.6	4	7,7,7	0 1 3 5 7 14 21	0.017 0.008 0.006 0.005 0.003 0.002 <0.002	<0.002 <0.002 <0.002 <0.002 ≤0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 ≤0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 ≤0.002 <0.002 <0.002	NR

CROP	Application				PHI, days	Residues, mg/kg				
Year Reference	g ai/ha	g ai/hl	No.	Interval, days		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
CAULIFLOWER	96	19.2	4	7,7,7	0	0.024	<0.002	<0.002	<0.002	NR
1995					1	0.015	<0.002	<0.002	<0.002	
AUS 94i47aR					3	0.006	<0.002	<0.002	<0.002	
					5	0.004	<0.002	<0.002	<0.002	
					7	0.003	<0.002	<0.002	<0.002	
					14	0.003	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
CAULIFLOWER	12	2.4	8	7,7,7,8,7,6,8,7	0	0.018	<0.002	<0.002	<0.002	NR
1995					1	0.012	<0.002	<0.002	<0.002	
AUS 94i47aR					3	0.012	<0.002	0.003	<0.002	
					5	0.009	<0.002	0.002	<0.002	
					7	0.009	<0.002	<0.002	<0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
CAULIFLOWER	24	4.8	8	7,7,7,8,7,6,8,7	0	0.020	<0.002	<0.002	<0.002	NR
1995					1	0.014	<0.002	<0.002	<0.002	
AUS 94i47aR					3	0.006	<0.002	<0.002	<0.002	
					5	0.004	<0.002	<0.002	<0.002	
					7	0.004	<0.002	<0.002	<0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
CAULIFLOWER	48	9.6	8	7,7,7,8,7,6,8,7	0	0.026	<0.002	<0.002	<0.002	NR
1995					1	0.016	<0.002	<0.002	<0.002	
AUS 94i47aR					3	0.005	<0.002	<0.002	<0.002	
					5	0.005	<0.002	<0.002	<0.002	
					7	0.003	<0.002	<0.002	<0.002	
					14	0.003	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
CAULIFLOWER	96	19.2	8	7,7,7,8,7,6,8,7	0	0.061	<0.002	0.009	<0.002	NR
1995					1	0.028	<0.002	0.003	<0.002	
AUS 94i47aR					3	0.013	<0.002	0.004	0.003	
					5	0.008	<0.002	<0.002	<0.002	
					7	0.002	<0.002	<0.002	<0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
Keats, 1996c										
CAULIFLOWER	12	2	2	7	0	0.011	<0.002	<0.002	<0.002	NR
1995					1	0.008	<0.002	<0.002	<0.002	
AUS 94i47R					3	0.002	<0.002	<0.002	<0.002	
					5	0.003	<0.002	<0.002	<0.002	
					7	0.002	<0.002	<0.002	<0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
CAULIFLOWER	24	4	2	7	0	0.012	<0.002	<0.002	0.003	NR
1995					1	0.008	<0.002	<0.002	0.003	
AUS 94i47R					3	0.006	<0.002	<0.002	<0.002	
					5	0.004	<0.002	<0.002	<0.002	
					7	<0.002	<0.002	<0.002	<0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
CAULIFLOWER	48	8	2	7	0	0.015	<0.002	<0.002	<0.002	NR
1995					1	0.016	<0.002	0.002	<0.002	
AUS 94i47R					3	0.011	<0.002	<0.002	<0.002	
					5	0.007	<0.002	<0.002	<0.002	
					7	0.003	<0.002	<0.002	<0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
CAULIFLOWER	96	16	2	7	0	0.037	<0.002	0.003	<0.002	NR
1995					1	0.029	<0.002	0.005	0.002	
AUS 94i47R					3	0.014	<0.002	<0.002	0.002	
					5	0.008	<0.002	<0.002	0.011	
					7	0.008	<0.002	<0.002	0.003	
					14	0.003	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
CAULIFLOWER	12	2	5	7,7,8	0	0.014	<0.002	<0.002	<0.002	NR
1995					1	0.012	<0.002	<0.002	<0.002	
AUS 94i47R					3	0.011	<0.002	<0.002	<0.002	
					5	0.006	<0.002	<0.002	<0.002	
					7	<0.002	<0.002	<0.002	<0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	

CROP Year Reference	Application				PHI, days	Residues, mg/kg				
	g ai/ha	g ai/hl	No.	Interval, days		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
CAULIFLOWER	24	4	5	7,7,8	0	0.044	0.003	0.01	0.011	NR
1995					1	0.040	0.002	0.009	0.009	
AUS 94i47R					3	0.036	<0.002	0.008	0.008	
					5	0.012	<0.002	<0.002	0.004	
					7	0.004	<0.002	<0.002	<0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
CAULIFLOWER	48	8	5	7,7,8	0	0.048	0.003	0.009	0.011	NR
1995					1	0.040	0.002	0.006	0.008	
AUS 94i47R					3	0.032	<0.002	0.005	0.008	
					5	0.012	<0.002	<0.002	0.003	
					7	0.008	<0.002	<0.002	<0.002	
					14	0.003	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
CAULIFLOWER	96	16	5	7,7,8	0	0.065	0.004	0.014	0.016	NR
1995					1	0.045	0.003	0.009	0.012	
AUS 94i47R					3	0.042	<0.002	0.009	0.011	
					5	0.009	<0.002	<0.002	<0.002	
					7	0.003	<0.002	<0.002	<0.002	
					14	0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
CAULIFLOWER	12	2	9	7,7,8,7,8 ,6,8,6	0	0.022	<0.002	0.002	0.004	NR
1995					1	0.015	<0.002	0.002	0.003	
AUS 94i47R					3	0.008	<0.002	<0.002	<0.002	
					5	0.003	<0.002	<0.002	<0.002	
					7	0.002	<0.002	<0.002	<0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
CAULIFLOWER	24	4	9	7,7,8,7,8 ,6,8,6	0	0.037	<0.002	0.01	0.005	NR
1995					1	0.039	<0.002	0.009	0.005	
AUS 94i47R					3	0.007	<0.002	0.008	<0.002	
					5	0.008	<0.002	<0.002	<0.002	
					7	0.004	<0.002	<0.002	<0.002	
					14	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
CAULIFLOWER	48	8	9	7,7,8,7,8 ,6,8,6	0	0.081	0.004	0.016	0.015	NR
1995					1	0.062	0.003	0.011	0.013	
AUS 94i47R					3	0.012	<0.002	<0.002	<0.002	
					5	0.009	<0.002	<0.002	<0.002	
					7	0.005	<0.002	<0.002	<0.002	
					14	0.006	<0.002	<0.002	<0.002	
					21	0.003	<0.002	<0.002	<0.002	
CAULIFLOWER	96	16	9	7,7,8,7,8 ,6,8,6	0	0.168	0.013	0.033	0.046	NR
1995					1	0.100	0.006	0.015	0.024	
AUS 94i47R					3	0.012	<0.002	<0.002	0.002	
					5	0.011	<0.002	<0.002	0.002	
					7	0.005	<0.002	<0.002	<0.002	
					14	0.003	<0.002	<0.002	<0.002	
					21	0.003	<0.002	<0.002	<0.002	

NR: not reported owing to analytical problems

Head and leafy cabbages (Table 64). Fipronil was applied as a foliar spray so residues of the degradation product fipronil-desulfinyl were reported.

Table 64. Supervised trials on head and leafy cabbages.

CROP, Country, Year, Reference, Remarks	Application					PHI, days	Residues, mg/kg				
	Form	g ai/ha	g ai/hl	No.	Interval, days		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
BRUSSELS	SC	50	12.5	5	8,6,8,7	0	0.223	0.004	0.022	0.021	NR
SPROUTS							0.232	0.005	0.024	0.023	
Australia, 1997,						3	0.104	0.004	0.011	0.024	
AK97023 R1 & R2							0.113	0.004	0.013	0.025	
Keats, 1997 j,						5	0.097	0.004	0.013	0.024	
2 replicates							0.099	0.003	0.015	0.025	
						7	0.017	0.003	0.006	0.008	
							0.016	0.002	0.004	0.007	
CABBAGE	SC	50	5	4	9,10,8	0	0.016	<0.002	<0.002	0.004	<0.002

CROP, Country, Year, Reference, Remarks	Application					PHI, days	Residues, mg/kg				
	Form	g ai/ha	g ai/hl	No.	Interval, days		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Australia, 1994,							0.014	<0.002	<0.002	0.003	<0.002
93AUS09i South						4	0.005	<0.002	<0.002	0.004	<0.002
Australia R1 & R2							0.005	<0.002	<0.002	0.004	<0.002
Keats, 1996 b,						8	0.004	<0.002	<0.002	<0.002	<0.002
2 replicates							0.004	<0.002	<0.002	<0.002	<0.002
						14	<0.002	<0.002	<0.002	<0.002	<0.002
							<0.002	<0.002	<0.002	<0.002	<0.002
CABBAGE	SC	100	10	4	9,10,8	0	0.027	<0.002	<0.002	0.005	<0.002
Australia, 1994,							0.026	<0.002	<0.002	0.005	<0.002
93AUS09i South						4	0.011	<0.002	<0.002	0.006	<0.002
Australia R1 & R2,							0.011	<0.002	<0.002	0.007	<0.002
Keats, 1996 b,						8	0.005	<0.002	<0.002	<0.002	<0.002
2 replicates							0.006	<0.002	<0.002	<0.002	<0.002
						14	<0.002	<0.002	<0.002	<0.002	<0.002
							0.002	<0.002	<0.002	<0.002	<0.002
Keats, 1994											
CABBAGE	WG	12.5	3.1	8	4,4,6,13,1	0	<0.002	<0.002	<0.002	<0.002	<0.002
Australia, 1994,					1,		0.002				
93AUS09i DMG					8,7	1	<0.002	<0.002	<0.002	0.004	<0.002
94007 Trial 1 R1 &										<0.002	
R2						3	<0.002	<0.002	<0.002	<0.002	<0.002
2 replicates						5	<0.002	<0.002	<0.002	<0.002	<0.002
						7	<0.002	<0.002	<0.002	<0.002	<0.002
						14	<0.002	<0.002	<0.002	<0.002	<0.002
						21	<0.002	<0.002	<0.002	<0.002	<0.002
CABBAGE	WG	25	6.1	8	4,4,6,13,1	0	0.008	<0.002	<0.002	0.004	<0.002
Australia, 1994,					1,		0.008			0.005	<0.002
93AUS09i DMG					8,7	1	0.004	<0.002	<0.002	0.003	<0.002
94007 Trial 1 R1 &							0.005			0.003	<0.002
R2,						3	<0.002	<0.002	<0.002	0.004	<0.002
2 replicates							0.002			0.004	<0.002
						5	<0.002	<0.002	<0.002	0.003	<0.002
evaluated acc. to										0.003	<0.002
New Zealand's						7	<0.002	<0.002	<0.002	<0.002	<0.002
GAP						14	<0.002	<0.002	<0.002	<0.002	<0.002
						21	<0.002	<0.002	<0.002	<0.002	<0.002
CABBAGE	WG	50	12.5	8	4,4,6,13,1	0	0.011	<0.002	<0.002	0.006	<0.002
Australia, 1994,					1,		0.01	<0.002	<0.002	0.006	<0.002
93AUS09i DMG					8,7	1	0.006	<0.002	<0.002	0.003	<0.002
94007 Trial 1 R1 &							0.006	<0.002	<0.002	0.003	<0.002
R2,						3	0.002	<0.002	<0.002	0.007	<0.002
2 replicates							0.002	0.002	<0.002	0.007	<0.002
						5	0.002	<0.002	<0.002	0.005	<0.002
							0.002	<0.002	<0.002	0.005	<0.002
						7	0.002	<0.002	<0.002	0.003	<0.002
							0.002	<0.002	<0.002	0.003	<0.002
						14	<0.002	<0.002	<0.002	0.002	<0.002
							<0.002	<0.002	<0.002	0.002	<0.002
						21	<0.002	<0.002	<0.002	<0.002	<0.002
CABBAGE	WG	100	25	8	4,4,6,13,1	0	0.018	<0.002	<0.002	0.006	<0.002
Australia, 1994					1,		0.017	<0.002	<0.002	0.006	<0.002
93AUS09i DMG					8,7	1	0.01	<0.002	<0.002	0.005	<0.002
94007 Trial 1 R1 &							0.01	<0.002	<0.002	0.005	<0.002
R2,						3	0.004	<0.002	<0.002	0.011	<0.002
2 replicates							0.004	<0.002	<0.002	0.011	<0.002
						5	0.004	<0.002	<0.002	0.008	<0.002
							0.004	<0.002	<0.002	0.008	<0.002
						7	0.003	<0.002	<0.002	0.005	<0.002
							0.003	<0.002	<0.002	0.005	<0.002
						14	<0.002	<0.002	<0.002	0.003	<0.002
							<0.002	<0.002	<0.002	0.003	<0.002
						21	<0.002	<0.002	<0.002	<0.002	<0.002
							<0.002	<0.002	<0.002	<0.002	<0.002
CABBAGE	WG	25	6.1	2	7	0	<0.002	<0.002	<0.002	<0.002	<0.002
Australia, 1994						1	0.003	<0.002	<0.002	<0.002	<0.002
93AUS09i DMG						3	<0.002	<0.002	<0.002	0.003	<0.002
94007 Trial 2						5	<0.002	<0.002	<0.002	<0.002	<0.002
evaluated acc. to						7	<0.002	<0.002	<0.002	<0.002	<0.002
New Zealand's						14	<0.002	<0.002	<0.002	<0.002	<0.002
GAP						21	<0.002	<0.002	<0.002	0.002	<0.002
CABBAGE	WG	50	12.5	2	7	0	0.003	<0.002	<0.002	<0.002	<0.002

CROP, Country, Year, Reference, Remarks	Application					PHI, days	Residues, mg/kg				
	Form	g ai/ha	g ai/hl	No.	Interval, days		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Australia, 1994 93AUS09i DMG 94007 Trial 2						1 3 5 7 14 21	0.005 <0.002 0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002	0.002 <0.002 0.002 <0.002 0.003 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002
CABBAGE Australia, 1994 93AUS09i DMG 94007 Trial 2	WG	100	25	2	7	0 1 3 5 7 14 21	0.004 0.007 0.002 0.002 0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	0.003 0.004 0.003 0.003 0.003 0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002
CABBAGE Australia, 1994 93AUS09i DMG 94007 Trial 2, no sticker, *evaluated acc. to New Zealand's GAP*	WG	25	6.1	2	7	0 1 3 5 7	0.003 0.003 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002
CABBAGE Australia, 1994 93AUS09i DMG 94007 Trial 2, no sticker	WG	50	12.5	2	7	0 1 3 5 7	0.003 0.004 0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	0.003 <0.002 0.004 0.002 0.002	<0.002 <0.002 <0.002 <0.002 <0.002
CABBAGE Australia, 1994 93AUS09i DMG 94007 Trial 2, no sticker	WG	100	25	2	7	0 1 3 5 7	0.005 0.006 0.002 0.002 0.002	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	0.003 0.004 0.003 0.002 0.002	<0.002 <0.002 <0.002 <0.002 <0.002
CABBAGE Australia, 1994 93AUS09i DMG 94007 Trial 2 *evaluated acc. to New Zealand's GAP*	WG	25	6.1	4	11,8,7	0 1 3 5 7 14	0.003 0.003 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 0.004 0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002
CABBAGE Australia, 1994 93AUS09i DMG 94007 Trial 2	WG	50	12.5	4	11,8,7	0 1 3 5 7 14	0.008 0.005 0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 0.003 0.005 0.003 0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002
CABBAGE Australia, 1994 93AUS09i DMG 94007 Trial 2	WG	100	25	4	11,8,7	0 1 3 5 7 14	0.012 0.007 0.002 0.002 0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002	0.003 0.004 0.004 0.003 0.004 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002
CABBAGE Australia, 1994 93AUS09i DMG 94007 Trial 2	WG	25 50 100	6.1 12.5 24	7	4,6,13,11, 8,7	0	0.004 0.012 0.022	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.002 0.002 0.003	<0.002 <0.002 <0.002
Muller, 1994a											
Brassica Juncea (Leafy brassica) Malaysia, 1993 93-712 Trial 1	SC	25	5, 3.1	2	7	1 3 5 7 10	0.091 0.020 <0.005 0.008 <0.005	<0.005 <0.005 <0.005 <0.005 <0.005	0.022 0.011 <0.005 0.005 <0.005	0.092 0.033 0.016 0.033 0.005	<0.005 <0.005 <0.005 <0.005 <0.005
Brassica Juncea (Leafy brassica) Malaysia, 1993 93-712 Trial 1	SC	50	10, 6.2	2	7	1 3 5 7 10	0.19 0.037 0.009 <0.005 <0.005	<0.005 <0.005 <0.005 <0.005 <0.005	0.048 0.018 0.007 <0.005 <0.005	0.23 0.090 0.035 0.036 0.019	<0.005 <0.005 <0.005 <0.005 <0.005
Brassica Juncea (Leafy brassica) Malaysia, 1993 93-712 Trial 2	SC	25	4.5, 3,6	2	7	1 2 4	0.025 0.012 <0.005	<0.005 <0.005 <0.005	0.005 <0.005 <0.005	0.016 0.019 0.009	<0.005 <0.005 <0.005
Brassica Juncea (Leafy brassica) Malaysia, 1993	SC	50	9.1, 7.1	2	7	1 2 4	0.042 0.033 0.007	<0.005 <0.005 <0.005	0.008 0.01 0.006	0.033 0.041 0.020	<0.005 <0.005 <0.005

CROP, Country, Year, Reference, Remarks	Application					PHI, days	Residues, mg/kg				
	Form	g ai/ha	g ai/hl	No.	Interval, days		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
93-712 Trial 2											
Keats, 1997l											
CABBAGE New Zealand, 1997 97NZL03	SC	24		4	10,10,11	0 1 3 7 14	0.082 0.041 0.018 ≤0.002 <0.002	<0.002 <0.002 <0.002 ≤0.002 <0.002	0.003 0.002 0.002 ≤0.002 <0.002	0.003 0.003 0.003 ≤0.002 <0.002	NR
CABBAGE New Zealand, 1997 97NZL03 *evaluated acc. to Australian GAP*	SC	48		4	10,10,11	0 1 3 7 14	0.192 0.083 0.045 0.014 0.004	0.002 <0.002 <0.002 ≤0.002 <0.002	0.01 0.005 0.004 0.002 <0.002	0.007 0.008 0.012 0.005 0.002	NR

NR: not reported owing to analytical problems
NA: not analysed

Potatoes (Table 65). Both soil and foliar pests can be controlled with fipronil. In some trials where fipronil was applied as a seed treatment, a broadcast incorporated soil spray or as granules applied in furrow, only the residues of fipronil, RPA 200766, MB 45950 and MB 46136 are reported here. Trials with foliar applications, except in Spain, also include fipronil-desulfinyl.

Table 65. Residues from supervised trials on potatoes.

Country, Year, Reference, Remarks	Application			PHI, days	Residues, mg/kg				
	Form.	g ai/ha	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Foliar (Orosz, 1995)									
Hungary, 1995 95-RHOP-AB-14-001	WG	20	1	1 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 <0.01 ≤0.01	0.01 <0.01 0.01 <0.01 <0.01
Hungary, 1995 95-RHOP-AB-14-001	WG	20	2	1 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 <0.01 ≤0.01	0.01 <0.01 <0.01 <0.01 <0.01
Foliar (Maestracci, 1998g)									
Germany, 1997 97746de1 Lower Saxony *evaluated acc. to Romanian GAP*	WG	20*	3	27	≤0.002	≤0.002	≤0.002	≤0.002	<0.002
Germany, 1997 97746de1 Lower Saxony *evaluated acc. to Polish GAP*	WG	20*	4	14 21	≤0.002 <0.002	≤0.002 <0.002	≤0.002 <0.002	≤0.002 <0.002	<0.002 <0.002
Germany, 1997 97746DE2 Rheinland-Pfalz *evaluated acc. to Romanian GAP*	WG	20*	3	31	≤0.002	≤0.002	≤0.002	≤0.002	<0.002
Germany, 1997 97746DE2 Rheinland-Pfalz *evaluated acc. to Polish GAP*	WG	20*	4	14 21	≤0.002 <0.002	≤0.002 <0.002	≤0.002 <0.002	≤0.002 <0.002	<0.002 <0.002
Germany, 1997 97746DE3, North Rhine Westphalia *evaluated acc. to Romanian GAP*	WG	20*	3	20	≤0.002	≤0.002	≤0.002	≤0.002	<0.002
Germany, 1997 97746DE3, North Rhine Westphalia *evaluated acc. to Polish GAP*	WG	20*	4	14 21	≤0.002 <0.002	≤0.002 <0.002	≤0.002 <0.002	≤0.002 <0.002	<0.002 <0.002
Germany, 1997 97746DE4 Bavaria *evaluated acc. to Romanian GAP*	WG	20*	3	27	≤0.002	≤0.002	≤0.002	≤0.002	<0.002

Country, Year, Reference, Remarks	Application			PHI, days	Residues, mg/kg				
	Form.	g ai/ha	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Germany, 1997 97746DE4 Bavaria *evaluated acc. to Polish GAP*	WG	20*	4	14 21	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002
Foliar (Yslan and Baudet, 1999d)									
Germany, 1998 98670DE1 Lower Saxony *evaluated acc. to Romanian GAP*	WG	20*	3	21	<0.002	<0.002	<0.002	<0.002	NR
Germany, 1998 98670DE1 Lower Saxony *evaluated acc. to Polish GAP*	WG	20*	4	14 21	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
Germany, 1998 98670DE2 North Rhine Westphalia *evaluated acc. to Polish GAP*	WG	20*	3	15	<0.002	<0.002	<0.002	<0.002	NR
Germany, 1998 98670DE2 North Rhine Westphalia *evaluated acc. to Polish GAP*	WG	20*	4	14 21	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
Germany, 1998 98670DE3 Rhine Palinate *evaluated acc. to Polish GAP*	WG	20*	3	20	<0.002	<0.002	<0.002	<0.002	NR
Germany, 1998 98670DE3 Rhine Palinate	WG	20*	4	14 21	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
Germany, 1998 98670DE4 Hesse *evaluated acc. to Polish GAP*	WG	20*	3	21	<0.002	<0.002	<0.002	<0.002	NR
Germany, 1998 98670DE4 Hesse *evaluated acc. to Polish GAP*	WG	20*	4	14 21	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
Foliar									
Poland, 1994/95 95794PL1 Bonin (after flowering) Muller, 1996d	WG	20	1	14	<0.002	<0.002	<0.002	<0.002	<0.002
Poland, 1994/95 95794PL2 Bonin (after planting) Muller, 1996d	WG	20	1	14	<0.002	<0.002	<0.002	<0.002	<0.002
Poland, 1994/95 95794PL3 Poznan (after flowering) Muller, 1996d *evaluated acc. to Romanian GAP*	WG	20	1	28	<0.002	<0.002	<0.002	<0.002	<0.002
Spain, 1994 94666SE1 Madrid Muller, 1996b, 2 replicates	WG	24	3	0 3 7 14 21	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	NR	<0.002 <0.002 <0.002 <0.002 <0.002
Spain, 1994 94666SE2 Seville Muller, 1996 b, 2 replicates	WG	24	2	0 3 7 14 21	0.002 0.004 0.002 0.002 0.002 0.003 <0.002 0.003 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 < 0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 0.004 <0.002 <0.002 <0.002 0.002 <0.002 <0.002	NR	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002
Spain, 1994 94666SE3 Valencia Muller, 1996 b, 2 replicates	WG	24	2	15	<0.002	<0.002	<0.002	NR	<0.002
Spain, 1994 94666SE4 Avila Muller, 1996b, 2 replicates	WG	24	2	15	<0.002	<0.002	<0.002	NR	<0.002
Spain, 1995 95715SE1 Madrid Richard and Muller, 1996a, 2 replicates	WG	24	3	0 3 7 14 21	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002

Country, Year, Reference, Remarks	Application			PHI, days	Residues, mg/kg				
	Form.	g ai/ha	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Spain, 1995 95715SE2 Valencia Richard and Muller, 1996a, 2 replicates	WG	24	3	0 3 7 14	<0.002 <0.002 <0.002 ≤0.002	<0.002 <0.002 <0.002 ≤0.002	<0.002 <0.002 <0.002 ≤0.002	<0.002 <0.002 <0.002 ≤0.002	<0.002 <0.002 <0.002 <0.002
Spain, 1995 95762SE1 Avila Richard and Muller, 1996b, 2 replicates	WG	24	2	16	≤0.002	≤0.002	≤0.002	≤0.002	<0.002
Spain, 1995 95762SE2 Seville Richard and Muller, 1996b, 2 replicates	WG	24	2	14	≤0.002	≤0.002	≤0.002	≤0.002	<0.002
Soil, granules applied in furrow at planting									
Greece, 1993 R93702XX1 Richard and Muller, 1994d, 2 replicates	GR	200 400	1	96	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR	<0.002 <0.002
Greece, 1994 94673GR1 Richard and Muller, 1995b, 2 replicates	GR	200 400	1	113	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR	<0.002 <0.002
Italy, 1993 R93636BO1 Bologna Richard and Muller, 1994j, 2 replicates	GR	150 300	1	118	0.017 0.005 0.008 0.009	0.002 <0.002 0.002 <0.002	0.009 0.003 0.004 0.004	NR	0.007 0.002 0.003 0.003
Italy, 1993 Corticelle, Bologna R93641BO1 Richard and Muller, 1994i, 2 replicates	GR	105 210	1	118	0.003 0.003 0.007 0.008	<0.002 <0.002 <0.002 <0.002	<0.002 <0.002 0.002 0.002	NR	<0.002 <0.002 <0.002 <0.002
Italy, 1995 95739BO1 Corticella Muller, 1996h, 2 replicates	GR	143	1	123	≤0.002	≤0.002	≤0.002	<0.002	<0.002
Italy, 1995 95739BO2 Minerbio Muller, 1996h, 2 replicates	GR	176	1	129	≤0.002	≤0.002	≤0.002	<0.002	<0.002
Italy, 1995 95746BO1 Corticella Muller,1996f, 2 replicates	GR	102 163	1	123	<0.002 ≤0.002	<0.002 ≤0.002	<0.002 ≤0.002	<0.002 <0.002	<0.002 <0.002
Italy, 1995 95746BO2 Minerbio Muller, 1996f, 2 replicates	GR	162	1	129	0.003 0.005	<0.002 ≤0.002	<0.002 ≤0.002	<0.002 <0.002	<0.002 0.002
Italy, 1996 96705BO1 Bologna Maestracci, 1997d, 2 replicates	GR	111 141	1	90	<0.002 ≤0.002	<0.002 ≤0.002	<0.002 ≤0.002	<0.002 <0.002	<0.002 <0.002

*Target rate; achieved rate per application in some cases varied by ±5% (1 g ai/ha).
NR: not reported

Table 66. Supervised trials on sugar beet roots (most trials include duplicate plots).

Country, Year, Reference	Application			PHI days	Residues, mg/kg				
	Form	g ai/ha	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Soil treatment at sowing (Claviere and Muller, 1990, 1991)									
France, 1990 Rachecourt, France (52) XE190I11	GR	100 200 300	1	148	<0.01 <0.01 0.16 <0.01 0.011	<0.01 <0.01 ≤0.01 <0.01 <0.01	<0.01 <0.01 0.015 0.011 0.018	NR	<0.01 <0.01 <0.01 <0.01 <0.01
France, 1990 Allogny, France (45) XB290I11	GR	100 200 300	1	179	<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	NR	<0.01 <0.01 <0.01 <0.01 <0.01

Country, Year, Reference	Application			PHI days	Residues, mg/kg				
	Form	g ai/ha	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
					<0.01	<0.01	<0.01		<0.01
France, 1990	GR	100	1	181	<0.01	<0.01	<0.01	NR	<0.01
Beaulieu, France					<0.01	<0.01	<0.01		<0.01
(45) XB190I11		200			<0.01	<0.01	<0.01		<0.01
					<0.01	<0.01	<0.01		<0.01
		300			<0.01	<0.01	<0.01		<0.01
					<0.01	<0.01	<0.01		<0.01
France, 1990	GR	100	1	168	0.015	<0.01	<0.01	NR	<0.01
Le Meillard, France					0.016	<0.01	<0.01		<0.01
(80) XD290I11		200			<0.01	<0.01	<0.01		<0.01
					0.014	<0.01	<0.01		<0.01
		300			0.012	<0.01	<0.01		<0.01
					0.032	<0.01	0.011		<0.01
France, 1990	GR	100	1	167	<0.01	<0.01	<0.01	NR	<0.01
Allas, France (17)					<0.01	<0.01	<0.01		<0.01
FRX90I11		200			<0.01	<0.01	<0.01		<0.01
					<0.01	<0.01	<0.01		<0.01
		300			<0.01	<0.01	<0.01		<0.01
					<0.01	<0.01	<0.01		<0.01
France, 1990	GR	100	1	176	<0.01	<0.01	NR	NR	NR
Mericourt, France					0.017	<0.01	NR		NR
(80) XD190I11		200[1]			<0.05	<0.01	NR		NR
					<0.05	<0.01	NR		NR
		300			0.043	<0.01	NR		NR
					0.028	<0.01	NR		NR
France, 1990 Mezerolles, France (80) XD190I12 Claviere and Muller, 1991	GR	300	1	144	0.016	<0.01	<0.01	NR	<0.01
Soil treatment at sowing (Dupont and Muller, 1992)									
France, 1991	GR	150	1	178	<0.01	<0.01	<0.01	NR	<0.01
Faverolles, France					NR	<0.01	<0.01		<0.01
(51) LE291I15		200			<0.01	<0.01	<0.01		<0.01
					<0.01	<0.01	<0.01		<0.01
France, 1991	GR	150	1	181	<0.01	<0.01	<0.01	NR	<0.01
Guignonville,					0.018	<0.01	<0.01		<0.01
France (45) LB391I15		200			0.018	<0.01	<0.01		<0.01
					<0.01	<0.01	<0.01		<0.01
France, 1991	GR	150	1	161	<0.01	<0.01	<0.01	NR	<0.01
Autruche, France					<0.01	<0.01	<0.01		<0.01
(51) LE191I15		200			<0.01	<0.01	<0.01		<0.01
					<0.01	<0.01	<0.01		<0.01
France, 1991	GR	150	1	198	<0.01	<0.01	<0.01	NR	<0.01
Bellegarde, France					0.011	<0.01	<0.01		<0.01
(45) LB191I15		200			<0.01	<0.01	<0.01		<0.01
					<0.01	<0.01	<0.01		<0.01
France, 1991	GR	150	1	213	<0.01	<0.01	<0.01	NR	<0.01
Meranville, France					<0.01	<0.01	<0.01		<0.01
(45) LB491I15		200			<0.01	<0.01	<0.01		<0.01
					0.072	<0.01	<0.01		<0.01
Soil treatment at sowing (Muller, 1994j)									
France, 1993 R93568B1	GR	305	1	193	<0.002	<0.002	<0.002	NR	<0.002
Gidy (45)					<0.002	<0.002	<0.002		<0.002
France, 1993	GR	243	1	196	0.006	<0.002	0.007	NR	<0.002

Country, Year, Reference	Application			PHI days	Residues, mg/kg				
	Form	g ai/ha	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
R93568B2 Patay (45)		 334			0.013 0.005 0.006	<0.002 <0.002 <0.002	0.011 0.006 0.005		0.002 <0.002 <0.002
France, 1993 R93568D1 Frohen le Grand (80)	GR	179 251	1	181	0.003 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002	0.002 <0.002 0.002 0.002	NR	<0.002 <0.002 <0.002 <0.002
France, 1993 R93568D2 Frohen le Grand (80)	GR	188[2] 281	1	190	0.009 0.002 <0.002	<0.002 <0.002 <0.002	0.005 <0.002 <0.002	NR	<0.002 <0.002 <0.002
France, 1993 R93568D3 Frohen le Grand (80)	GR	174[2] 211	1	175	0.013 0.002 0.002 <0.002 0.003	<0.002 <0.002 <0.002 <0.002 <0.002	0.005 0.004 0.004 <0.002 0.005	NR	<0.002 <0.002 <0.002 <0.002 <0.002
France, 1993 R93568E1 Bazancourt (51)	GR	159 253	1	166	0.007 0.005 0.017 0.024	<0.002 <0.002 <0.002 <0.002	0.004 0.003 0.007 0.008	NR	<0.002 <0.002 <0.002 <0.002
Soil treatment at sowing (Muller, 1995e)									
France, 1994 945520R1 Patay (45)	GR	161 245	1	189	0.002 0.003 0.004 <0.002	<0.002 <0.002 <0.002 <0.002	0.004 0.004 0.008 0.003	NR	<0.002 <0.002 0.002 <0.002
France, 1994 94552AM1 Barly (80)	GR	161 240	1	153	0.002 0.003 0.004 <0.002	0.002 <0.002 <0.002 <0.002	0.003 0.006 0.003 <0.002	NR	<0.002 <0.002 <0.002 <0.002
France, 1994 94552RS1 Muizon(51)	GR	161 244	1	174	0.003 0.002 0.003 0.003	<0.002 <0.002 <0.002 <0.002	0.005 0.004 0.005 0.003	NR	<0.002 <0.002 <0.002 <0.002
Soil treatment before sowing (Maestracci, 1998b)									
France, 1997 97521AM1 Barly (80)	WG	200	1	168	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002
France, 1997 97521DJ1 Neuilly les Dijon (21)	WG	210	1	188	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002
France, 1997 97521OR1 Merevile (91)	WG	200	1	181	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002
France, 1997 97521RS1 Reims (51)	WG	200	1	186	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002
Foliar treatment (Lantos, 1997a)									
Hungary, 1996 RP AB 15003	WG	24	1	30	<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
Soil treatment at sowing (Richard and Muller, 1994b)									
Italy, 1993 93640BO1 CNS Bologna	GR	105 210	1	169	0.003 <0.002 0.005 <0.002	<0.002 <0.002 <0.002 <0.002	0.002 0.003 0.005 <0.002	NR	<0.002 <0.002 <0.002 <0.002
Italy, 1993 93630BO1 CNS Bologna	GR	150 300	1	169	0.003 <0.002 0.002	<0.002 <0.002 <0.002	0.004 <0.002 0.003	NR	<0.002 <0.002 <0.002

Country, Year, Reference	Application			PHI days	Residues, mg/kg				
	Form	g ai/ha	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
					0.003	<0.002	0.006		<0.002
Soil treatment in furrow at sowing (Muller, 1996e)									
Italy, 1995 95742BO1 CNS Corticella (BO)	GR	124	1	140	0.002	<0.002	0.003	<0.002	<0.002
					0.004	<0.002	0.004	<0.002	<0.002
		166			<0.002	<0.002	<0.002	<0.002	<0.002
					<0.002	<0.002	<0.02	<0.002	<0.002
Italy, 1995 95742BO2 Minerbio (BO)	GR	103	1	136	0.002	<0.002	0.002	<0.002	<0.002
					0.002	<0.002	0.002	<0.002	<0.002
		146			<0.002	<0.002	<0.002	<0.002	<0.002
					0.002	<0.002	0.003	<0.002	<0.002
Soil treatment (Muller, 1996l)									
Italy, 1995 95741BO1 CNS Corticella (BO)	GR	90	1	140	<0.002	<0.002	<0.002	<0.002	<0.002
					<0.002	<0.002	<0.002	<0.002	<0.002
		141			<0.002	<0.002	<0.002	<0.002	<0.002
					<0.002	<0.002	<0.002	<0.002	<0.002
Italy, 1995 95741BO2 Minerbio (BO)	GR	106	1	136	0.002	<0.002	0.002	<0.002	<0.002
					0.003	<0.002	0.004	<0.002	<0.002
		140			0.004	<0.002	<0.002	<0.002	<0.002
					0.005	<0.002	<0.002	<0.002	<0.002
Soil treatment (Yslan and Baudet, 1999c)									
Spain, 1998 98589M1 Sta. Olalla Toledo	WG	197	1	198	<0.002	<0.002	<0.002	<0.002	NR
					<0.002	<0.002	<0.002	<0.002	

NR: not reported
[1]LOQ increased to 0.05 mg/kg owing to interference; not included in evaluation
[2]only one plot

Cereal grains (barley, maize, rice, sorghum, wheat, Tables 67-71). Soil and foliar pests can be controlled with fipronil. In terrestrial trials where fipronil has been applied as a seed treatment, as granules in furrow or as an incorporated soil spray, only the residues of fipronil, RPA 200766, MB 45950 and MB 46136 have generally been reported. Trials with foliar and aquatic applications (flooded rice paddy) also include fipronil-desulfinyl.

Barley (Table 67). Residue trials in France were 2-3 times overdosed but show a 'nil residue situation' in the grain.

Table 67. Supervised trials on barley grain (seed treatment) in France, 1993-94.

Reference	Application			PHI, days	Residues, mg/kg				
	Form	g ai/t	No.		Fipronil	MB 45950	MB 46136	Fipronil-desulfinyl	RPA 200766
Muller, 1995a									
94501OR1	FS	1500	1	260	<0.002	<0.002	<0.002	NR	<0.002
94501AM1	FS	1500	1	271	<0.002	<0.002	<0.002	NR	<0.002
94501RS1	FS	1500	1	260	<0.002	<0.002	<0.002	NR	<0.002
94501DJ1	FS	1500	1	250	<0.002	<0.002	<0.002	NR	<0.002
94501LY1	FS	1500	1	249	<0.002	<0.002	<0.002	NR	<0.002
Muller, 1995c									
94502OR1	FS	1000	1	260	<0.002	<0.002	<0.002	NR	<0.002

NR: not reported

Maize (Table 68). The supervised trials on maize included applications by seed treatment, incorporated granules in furrow, incorporated soil spray in furrow at sowing, pre-sowing incorporated broadcast soil spray, and foliar spray. Each trial was with duplicate plots, the US trials with two or three plots.

Table 68. Supervised trials on maize. Residues in grain.

Country, Year, Reference	Application				PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Seed treatment (Muller, 1994c)										
France, 1993, R93562H1	FS	2500			161	≤0.002	≤0.002	≤0.002	NR	<0.002
Seed treatment (Muller, 1994f)										
France, 1993, R93565A1	FS	3750			151	≤0.002	≤0.002	≤0.002	NR	<0.002
France, 1993, R93565B1	FS	3750			165	≤0.002	≤0.002	≤0.002	NR	<0.002
France, 1993, R93565G1	FS	3750			182	≤0.002	≤0.002	≤0.002	NR	<0.002
France, 1993, R93565H1	FS	3750			152	≤0.002	≤0.002	≤0.002	NR	<0.002
France, 1993, R93565H2	FS	3750			154	≤0.002	≤0.002	≤0.002	NR	<0.002
France, 1993, R93565K1	FS	3750			154	≤0.002	≤0.002	≤0.002	NR	<0.002
Soil treatment incorporated granules in furrow (Muller, 1996i)										
France, 1995, 95535BX1	GR		298		150	<0.002	<0.002	<0.002	<0.002	<0.002
France, 1995, 95535AM1	GR		177		147	≤0.002	≤0.002	≤0.002	<0.002	<0.002
Soil treatment pre-sowing, 2 replicates (Maestracci, 1998d)										
France, 1997, 97540AM1	WG		200	66.7	180*	≤0.002	≤0.002	≤0.002	<0.002	<0.002
France, 1997, 97540OR1	WG		200	60.6	174*	≤0.002	≤0.002	≤0.002	<0.002	<0.002
France, 1997, 97540RS1	WG		200	84.7	169*	≤0.002	≤0.002	≤0.002	<0.002	<0.002
France, 1997, 97540RS2 **replicate 1**	WG		200	100	156*	<0.002[3]	<0.002[3]	<0.002[3]	<0.002[3]	<0.002[3]
France, 1997, 97540RS2 **replicate 2**	WG		200	100	156*	0.004[3]	≤0.002[3]	≤0.003[3]	<0.002[3]	0.008[3]
France, 1997, 97540BX1 **replicate 1**	WG		200	60.6	159*	<0.002[3]	<0.002[3]	<0.002[3]	<0.002[3]	<0.002[3]
France, 1997, 97540BX1 **replicate 2**	WG		200	60.6	159*	≤0.002	≤0.002	≤0.002	<0.002	<0.002
France, 1997, 97540BX2	WG		200	60.6	159*	≤0.002	≤0.002	≤0.002	<0.002	<0.002
France, 1997, 97540TL1 **replicate 1**	WG		200	60.6	154*	≤0.002[3]	≤0.002[3]	≤0.002[3]	<0.002[3]	<0.002[3]
France, 1997, 97540TL1 **replicate 2**	WG		200	60.6	154*	<0.002[3]	<0.002[3]	<0.002[3]	<0.002[3]	<0.002[3]
France, 1997, 97540TL2	WG		200	60.6	159*	≤0.002	≤0.002	≤0.002	<0.002	<0.002
Seed treatment (Muller, 1994d)										
France, 1993, R933563A1 (maize, sweet)	FS	2500			109	≤0.002 (ear)	≤0.002 (ear)	≤0.002 (ear)	NR	<0.002 (ear)
Soil treatment at sowing (Richard and Muller, 1994k)										
France, 1993, 93735 Sevignacq (maize, sweet)	GR		200		99	≤0.002 (ear)	≤0.002 (ear)	≤0.002 (ear)	NR	<0.002 (ear)
Soil treatment at sowing (Muller, 1995d)										
Greece, 1994, 94672GR2	GR		200		99	≤0.002	≤0.002	≤0.002	NR	<0.002
Greece, 1994, 94672GR2	GR		400		99	<0.002	<0.002	<0.002	NR	<0.002
Foliar spray (Lantos, 1997b)										
Hungary, 1997, RP AB 15 004	WG		24	6	30	≤0.01	≤0.006	≤0.01	≤0.005	<0.014
Soil treatment at sowing (Richard and Muller, 1994g)										
Italy, 1993, R93633BO1	GR		240		143	<0.002	<0.002	<0.002	NR	<0.002
Soil treatment at sowing, 2 replicates (Muller, 1996c)										
Italy, 1995, 95744BO1	GR		112		174	≤0.002	≤0.002	≤0.002	<0.002	<0.002
Italy, 1995, 95744BO2	GR		153		161	0.002 <0.002	≤0.002 <0.002	≤0.002 <0.002	<0.002 <0.002	<0.002 <0.002
Seed treatment (Richard and Muller, 1995d)										
Spain, 1994, 94665SE1	FS	2500			174	≤0.002	≤0.002	≤0.002	NR	<0.002
Seed treatment (Maestracci, 1996b)										
Spain, 1995, 95712SE1	FS	2500			155	≤0.002	≤0.002	≤0.002	<0.002	<0.002
Spain, 1995, 95712SE2	FS	2500			153	≤0.002	≤0.002	≤0.002	<0.002	<0.002
Soil treatment pre-sowing (Yslan and Baudet, 1999b)										
Spain, 1998, 98588SE1	WG		195.2	42.4	163*	≤0.002	≤0.002	≤0.002	<0.002	NR
Spain, 1998, 98588SE2	WG		176.6	40.8	147*	≤0.002	≤0.002	≤0.002	<0.002	NR
Soil treatment at sowing, 3 replicates (Kowite, 1993a)										
USA, 1992[1], 92-015 INC	GR		146		146	≤0.002	≤0.002	≤0.003	<0.002	<0.003

Country, Year, Reference	Application				PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
USA, 1992[1], 92-015 T-band	GR		146		146	<0.002	<0.002	<0.003	<0.002	<0.003
USA, 1992[1], 92-016	GR		146		157	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.003 <0.003 <0.003	<0.002 <0.002 <0.01	<0.003 <0.003 <0.003
USA, 1992[1], 92-019 INC	GR		146		191	<0.002	<0.002	<0.003	<0.002	<0.003
USA, 1992[1], 92-019 T-band	GR		146		191	<0.002	<0.002	<0.003	<0.002	<0.003
USA, 1992[1], 92-029	GR		157		153	<0.002	<0.002	<0.003	<0.002	<0.003
USA, 1992[1], 92-039	GR		135		156	<0.002	<0.002	<0.003	<0.002	<0.003
USA, 1992[1], 92-058 INC	GR		157		159	<0.002	<0.002	<0.003	<0.002	<0.003
USA, 1992[1], 92-058 T-band	GR		157		159	<0.002	<0.002	<0.003	<0.002	<0.003
USA, 1992[1], 92-076	GR		135		151	<0.002 <0.01 <0.002	<0.002 <0.002 <0.002	<0.003 <0.003 <0.003	<0.002 <0.002 <0.002	<0.003 <0.003 <0.003
USA, 1992[1], 92-094	GR		146		173	<0.002	<0.002	<0.003	<0.002	<0.003
USA, 1992[1], 92-097	GR		146		162	<0.002	<0.002	<0.003	<0.002	<0.003
USA, 1992[1], 92-097 T-band	GR		146		169	<0.002	<0.002	<0.003	<0.002	<0.003
Soil treatment at sowing, 3 replicates (Kowite, 1994)										
USA, 1993[1], 93-206 T-band	GR		146		148	<0.002	<0.002	<0.003	NR	<0.003
USA, 1993[1], 93-206 INC	GR		146		148	<0.002	<0.002	<0.003	NR	<0.003
USA, 1993[1], 93-207	GR		146		140	<0.002	<0.002	<0.003	NR	<0.003
USA, 1993[1], 93-208	GR		146		125	<0.002 <0.002 <0.01	<0.01 <0.002 <0.01	<0.003 <0.003 <0.003	NR	<0.003 <0.003 <0.003
USA, 1993[1], 93-209	GR		146		168	<0.002	<0.002	<0.003	NR	<0.003
USA, 1993[1], 93-210	GR		146		128	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.003 <0.003 <0.01	NR	<0.003 <0.003 <0.003
USA, 1993[1], 93-211	GR		146		124	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.003 <0.003 <0.003	NR	<0.003 <0.01 <0.01
USA, 1993[1], 93-212 T-band	GR		146		171	<0.002	<0.002	<0.003	NR	<0.003
USA, 1993[1], 93-212 INC	GR		146		171	<0.002	<0.002	<0.003	NR	<0.003
USA, 1993[1], 93-213 T-band	GR		146		171	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.003 <0.003 <0.003	NR	<0.003 <0.01 <0.01
USA, 1993[1], 93-213 INC	GR		146		171	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.003 <0.003 <0.003	NR	<0.01 <0.003 <0.003
USA, 1993[1], 93-214 T-band	GR		146		131	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.003 <0.003 <0.003	NR	<0.003 <0.003 <0.01
USA, 1993[1], 93-214 INC	GR		146		131	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.003 <0.003 <0.01	NR	<0.01 <0.01 <0.01
USA, 1993[1], 93-215 T-band	GR		146		140	<0.002	<0.002	<0.003	NR	<0.003
USA, 1993[1], 93-215 INC	GR		146		140	<0.002	<0.002	<0.003	NR	<0.003
Soil treatment at sowing, 2 replicates (Kowite, 1996)										
USA, 1995[1], 95-0212 GR INC	GR		146		126	<0.01 <0.004	<0.01 <0.01	<0.003 <0.003	NR	<0.002 <0.002
USA, 1995[1], 95-0212 SS INC	WG		146		126	<0.004 <0.004	<0.01 <0.004	<0.003 <0.003	NR	<0.002 <0.002
USA, 1995[1], 95-0213 GR INC	GR		146		148	<0.004	<0.01[2]	<0.003	NR	<0.002
USA, 1995[1], 95-0213 SS INC	WG		146		148	<0.004	<0.01[2]	<0.003	NR	<0.002
USA, 1995[1], 95-0214 GR INC	GR		146		153	<0.004 <0.004	<0.01 <0.004	<0.003 <0.003	NR	<0.002 <0.002
USA, 1995[1], 95-0214 SS INC	WG		146		153	<0.004	<0.004	<0.003	NR	<0.002

Country, Year, Reference	Application				PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
USA, 1995[1], 95-0215 GR INC	GR		146		159	<0.01 <0.004	<0.01 <0.004	<0.01 0.003	NR	<0.002 <0.002
USA, 1995[1], 95-0215 SS INC	WG		146		159	<0.004	<0.004	<0.003	NR	<0.002
USA, 1995[1], 95-0216 GR INC	GR		146		136	<0.004	<0.004	<0.003	NR	<0.002
USA, 1995[1], 95-0216 SS INC	WG		146		136	<0.004	<0.004	<0.003	NR	<0.002
USA, 1995[1], 95-0217 GR INC	GR		146		122	<0.004	<0.01	<0.003	NR	<0.002
USA, 1995[1], 95-0217 SS INC	WG		146		122	<0.004	<0.01	<0.003	NR	<0.002
USA, 1995[1], 95-0218 GR INC	GR		146		122	<0.004	<0.01[2]	<0.003	NR	<0.002
USA, 1995[1], 95-0218 SS INC	WG		146		122	<0.004	<0.01[2]	<0.003	NR	<0.002
USA, 1995[1], 95-0219 GR INC	GR		146		116	<0.004	<0.01[2]	<0.003	NR	<0.002
USA, 1995[1], 95-0219 SS INC	WG		146		116	<0.004	<0.01[2]	<0.003	NR	<0.002
USA, 1995[1], 95-0220 GR INC	GR		146		143	<0.004 <0.004	<0.01 <0.004	<0.003 <0.003	NR	<0.002 <0.002
USA, 1995[1], 95-0220 SS INC	WG		146		143	<0.004 <0.004	<0.01 <0.004	<0.003 <0.003	NR	<0.002 <0.002
USA, 1995[1], 95-0221 GR INC	GR		146		160	<0.004	<0.004	<0.003	NR	<0.002
USA, 1995[1], 95-0221 SS INC	WG		146		160	<0.004	<0.004	<0.003	NR	<0.002
USA, 1995[1], 95-0222 GR INC	GR		146		150	<0.004 <0.004	<0.01 <0.004	<0.003 <0.003	NR	<0.002 <0.002
USA, 1995[1], 95-0222 SS INC	WG		146		150	<0.004	<0.004	<0.003	NR	<0.002
USA, 1995[1], 95-0223 GR INC	GR		146		125	<0.004 <0.004	<0.01 <0.004	<0.003 <0.003	NR	<0.002 <0.002
USA, 1995[1], 95-0223 SS INC	WG		146		125	<0.004	<0.004	<0.003	NR	<0.002

*days between sowing and harvest, not between soil spray and harvest
NR: not reported
AP: analytical problem
[1] LOQ for each compound 0.01 mg/kg (lowest fortification level). Residues reported as ND (not detected) in the original report are shown as <0.00x (i.e. <MLD)
[2] Contamination of control reported
[3] Result confirmed by GC-MS-MS

Rice (Table 69). The results of supervised trials after seed treatment, granules broadcast into the flooded paddy, granules in nursery boxes, pre-plant incorporated broadcast soil spray, and foliar applications were reported. Although many of the registered labels for liquid formulations either show no PHI restriction or indicate PHIs of a week or so (generally applicable to all crops and not specific to rice), fipronil's pest spectrum and insect pest timing needs to be taken into account to understand what a normal PHI would be for rice. Except in Indonesia where the stink bug, a late-season pest, is present, PHIs of 28 days or more are common practice. In many countries, one application of fipronil per crop is made early, in which case PHIs are longer than 40 days.

Table 69. Residues in rice grain.

Commodity, Country, Year, Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Seed box treatment, 2 replicates (Anon., 1994)											
Unpolished rice, Japan, 1993, I.E.T. Official residue trial, Ushuku	GR		0.5 g ai/box		1	132	<0.001	<0.001	<0.001	<0.001	0.003

Commodity, Country, Year, Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Unpolished rice, Japan, 1993, I.E.T. Official residue trial, Shiga	GR		0.5 g ai/box		1	141	<0.001	<0.001	<0.001	<0.001	<0.001
Seed box treatment, 2 replicates (Anon., 1995a)											
Rice, brown, Japan, 1995, Nihon Noyaku	GR		0.5 g ai/box		1	123	<0.001	<0.001	<0.001	<0.001	0.003
Seed box treatment, 2 replicates (Anon., 1995b)											
Rice, brown, Japan, 1995, Nissan Fukui	GR		0.5 g ai/box		1	140	<0.001	<0.001	<0.001	<0.001	<0.001
Rice, brown, Japan, 1995, Nissan Mie	GR		0.5 g ai/box		1	118	<0.001	<0.001	<0.001	<0.001	<0.001
Seed treatment, 2 replicates (Keats, 1996e)											
Australia, 1996, AK96059	FS		25 50 100		1	167	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	NR
Seed treatment, 2 replicates (Keats, 1996h)											
Australia, 1996, AK96060	FS		25 50 100		1	215	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	NR
Seed treatment, 2 replicates (Keats, 1996i)											
Australia, 1996, AK96061	FS		12.5 25 50		1	144	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	NR
Seed treatment (Yslan and Baudet, 1999a)											
Spain, 1998, 98639TR1	FS	263	52.6		1	133	<0.002	<0.002	<0.002	<0.002	NR
Seed treatment (Maestracci, 1997c)											
France, 1996, 96561AV1	FS	130			1	119 143	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002
Seed treatment (Maestracci, 1998e)											
France, 1997, 97545AV1 variety Thaibonnet	FS	130			1	127[2] 149	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002
France, 1997, 97545AV2 variety Ariete	FS	130			1	127[2] 149	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002
Seed treatment (Maestracci, 1998f)											
France, 1997, 97546AV1 variety Ariete	FS	130			1	133	<0.002	<0.002	<0.002	<0.002	NR
France, 1997, 97546AV2 variety Thaibonnet	FS	130			1	133	<0.002	<0.002	<0.002	<0.002	NR
Seed treatment, 2 replicates (Keats, 1996m)											
Thailand, 1996, 95THA01i	FS		50		1	116	0.003 0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
Thailand, 1996, 95THA01i	FS		100		1	116	<0.002 0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
Seed treatment, 2 replicates (Mede, 1996b)											
USA, 1995[1], 95-0248AR	FS		56		1	119	<0.003	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0249AR	FS		56		1	134	<0.003	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0250CA	FS		56		1	139	<0.003	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0251CA	FS		56		1	139	<0.003	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0252LA	FS		56		1	107	<0.003	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0253LA	FS		56		1	112	<0.003 <0.01	<0.003 <0.003	<0.003 <0.003	<0.003 <0.003	<0.003 <0.003
USA, 1995[1], 95-0254MS	FS		56		1	128	<0.003	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0255TX	FS		56		1	119	<0.003	<0.003	<0.003	<0.003	<0.003

Commodity, Country, Year, Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
USA, 1995[1], 95-0256MS	FS		56		1	130	<0.003	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0257LA	FS		56		1	109	<0.003	<0.003	<0.003	<0.003	<0.003
Seed treatment, 2 replicates (Mede, 1997)											
USA, 1996[1], 10392-01	FS		54		1	126	<0.003	<0.003	<0.003	<0.003	NR
USA, 1996[1], 10392-02	FS		58		1	125	<0.003	<0.003	<0.003	<0.003	NR
USA, 1996[1], 10392-03	FS		57		1	129	<0.01 <0.003	<0.003 <0.003	<0.003 <0.003	<0.003 <0.003	NR
USA, 1996[1], 10392-04	FS		56		1	110	<0.003	<0.003	<0.003	<0.003	NR
USA, 1996[1], 10392-05	FS		56		1	117	<0.003	<0.003	<0.003	<0.003	NR
USA, 1996[1], 10392-07	FS		56		1	128	<0.003	<0.003	<0.003	<0.003	NR
USA, 1996[1], 10392-08	FS		57		1	138	<0.003	<0.003	<0.003	<0.003	NR
Soil pre-plant incorporated broadcast treatment, 2 replicates (Mede, 1996b)											
USA, 1995[1], 95-0248AR	WG		56		1	119	<0.003	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0249AR	WG		56		1	134	<0.003	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0250CA	WG		56		1	141	<0.003	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0251CA	WG		56		1	140	<0.003	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0252LA	WG		56		1	112	<0.003	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0253LA	WG		56		1	114	<0.003	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0254MS	WG		56		1	128	<0.003	<0.003	<0.003	<0.003	<0.01
USA, 1995[1], 95-0255TX	WG		56		1	119	<0.003	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0256MS	WG		56		1	130	<0.003	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0257LA	WG		56		1	110	<0.003	<0.003	<0.003	<0.003	<0.003
Soil pre-plant incorporated broadcast treatment, 2 replicates (Mede, 1997)											
USA, 1996[1], 10392-01	SC		57		1	133	<0.003	<0.003	<0.003	<0.003	NR
USA, 1996[1], 10392-02	SC		57		1	125	<0.003	<0.003	<0.003	<0.003	NR
USA, 1996[1], 10392-03	SC		57		1	136	<0.003	<0.003	<0.003	<0.003	NR
USA, 1996[1], 10392-04	SC		56		1	113	<0.003	<0.003	<0.003	<0.003	NR
USA, 1996[1], 10392-05	SC		57		1	120	<0.003	<0.003	<0.003	<0.003	NR
USA, 1996[1], 10392-07	SC		59		1	128	<0.003	<0.003	<0.003	<0.003	NR
USA, 1996[1], 10392-08	SC		56		1	143	<0.01 <0.01	<0.01 <0.003	<0.003 <0.003	<0.003 <0.003	NR
Broadcast into flooded paddy, 2 replicates (Garcia and Oiveira, 1994c)											
Brazil, 1994, 032/94PC	GR		100 200		1	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
Broadcast into flooded paddy, 2 replicates (Garcia and Oiveira 1994e)											
Brazil, 1994, 062/94PC	GR		100 200		1	30	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
Broadcast into flooded paddy, 3 replicates (Maycey *et al.*, 1994d)											
Rice, brown, Indonesia, 1992/3, P92/278 Pusaharuta	GR		50		1	98	<0.001	NR	NR	<0.001	NR
Rice, brown, Indonesia, 1992/3, P92/278 Kutasari	GR		50		1	97	<0.001	NR	NR	<0.001	NR

Commodity, Country, Year, Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Rice, brown, Indonesia, 1992/3, P92/278 Mundu	GR		50		1	97	0.003 <0.001 <0.001	NR	NR	<0.001 <0.001 <0.001	NR
Broadcast treatment into flooded paddy (Maycey *et al.*, 1994a)											
Rice, brown, Philippines, 1992, 92/277 Luzon	GR		50		1	81	<0.001	NR	NR	<0.001	NR
Rice, white Philippines, 1992, 92/277 Luzon	GR		50		1	81	<0.001	NR	NR	<0.001	NR
Rice, brown, Philippines, 1992, 92/277 Visayas	GR		50		1	76	<0.001	NR	NR	<0.001	NR
Rice, white, Philippines, 1992, 92/277 Visayas	GR		50		1	76	<0.001	NR	NR	<0.001	NR
Rice, brown, Philippines, 1992, 92/277 Mindanao	GR		50		1	76	<0.001	NR	NR	<0.001	NR
Rice, white, Philippines, 1992, 92/277 Mindanao	GR		50		1	76	<0.001	NR	NR	<0.001	NR
Broadcast treatment into flooded paddy, (Maycey *et al.*, 1994b)											
Rice, brown Taiwan, 1993, 92/275 Hsinung Li **Plot 1**	GR		50		1	89	<0.001	NR	NR	<0.001	NR
Rice, brown Taiwan, 1993, 92/275 Hsinung Li **Plot 2**	GR		50		1	89	<0.001	NR	NR	<0.001	NR
Broadcast treatment into flooded paddy (Maycey *et al.*, 1994c)											
Rice, brown Thailand, 1992/1993, 92/276 Supanuri Rice Research Station **replicate 1**	GR		50		1	79	<0.001	NR	NR	<0.001	NR
Rice, white Thailand, 1992/1993, 92/276 Supanuri Rice Research Station **replicate 1**	GR		50		1	79	<0.001	NR	NR	<0.001	NR
Rice, brown Thailand, 1992/1993, 92/276 Supanuri Rice Research Station **replicate 2**	GR		50		1	75	<0.001	NR	NR	<0.001	NR
Rice, white Thailand, 1992/1993, 92/276 Supanuri Rice Research Station **replicate 2**	GR		50		1	75	<0.001	NR	NR	<0.001	NR
Rice, brown Thailand, 1992/1993, 92/276 Supanuri	GR		50		1	78	<0.001	NR	NR	<0.001	NR
Rice, white Thailand, 1992/1993, 92/276 Supanuri	GR		50		1	78	<0.001	NR	NR	<0.001	NR
Rice, brown Thailand, 1992/1993, 92/276 Nontaburi	GR		50		1	75	<0.001	NR	NR	<0.001	NR

Commodity, Country, Year, Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Rice, white Thailand, 1992/1993, 92/276 Nontaburi	GR		50		1	75	<0.001	NR	NR	<0.001	NR
Foliar treatment, 3 replicates (Maycey *et al.*, 1994d)											
Indonesia, 1992/3, P92/278 Kutasari	SC		50		1	67	<0.001	NR	NR	<0.001	NR
Indonesia, 1992/3, P92/278 Mundu	SC		50		1	67	<0.001	NR	NR	<0.001	NR
Indonesia, 1992/3, P92/278 Pusaharuta	SC		50		1	67	<0.001	NR	NR	<0.001	NR
Foliar treatment, residues for rice with husks/without husks (Keats, 1997 a,d)											
Indonesia, 1996, AK97004/AK97012	SC		12.5	3.1	1	7	0.028/ 0.002	0.002/ <0.002	0.005/ <0.002	0.022/ <0.002	NR
Indonesia, 1996, AK97004/AK97012	SC		12.5	3.1	1	14	0.029/ 0.002	<0.002/ <0.002	0.007/ <0.002	0.02/ 0.002	NR
Indonesia, 1996, AK97004/AK97012	SC		12.5	3.1	2	7	0.069/ 0.006	0.005/ <0.002	0.013/ <0.002	0.047/ 0.003	NR
Indonesia, 1996, AK97004/AK97012	SC		25	6.2	1	14	0.037/ 0.008	<0.002/ <0.002	0.009/ 0.002	0.029/ 0.005	NR
Indonesia, 1996, AK97004/AK97012	SC		25	6.2	1	7	0.086/ 0.013	0.011/ <0.002	0.159/ 0.004	0.076/ 0.011	NR
Indonesia, 1996, AK97004/AK97012	SC		25	6.2	2	7	0.099/ 0.03	0.016/ 0.002	0.042/ 0.009	0.103/ 0.019	NR
Indonesia, 1996, AK97004/AK97012	SC		50	12.5	1	7	0.099/ 0.017	0.017/ <0.002	0.028/ 0.004	0.093/ 0.009	NR
Indonesia, 1996, AK97004/AK97012	SC		50	12.5	1	14	0.097/ 0.013	0.005/ <0.002	0.024/ 0.003	0.082/ <0.002	NR
Indonesia, 1996, AK97004/AK97012	SC		50	12.5	2	7	0.101/ 0.029	0.026/ 0.002	0.065/ 0.008	0.12/ 0.019	NR
Foliar treatment (Maycey *et al.*, 1994a)											
Rice, brown Philippines, 1992, 92/277 Luzon	SC		50		1	51	<0.001	NR	NR	<0.001	NR
Rice, white Philippines, 92/277 Luzon	SC		50		1	51	<0.001	NR	NR	<0.001	NR
Rice, brown Philippines, 1992, 92/277 Visayas	SC		50		1	46	<0.001	NR	NR	<0.001	NR
Rice, white Philippines, 1992, 92/277 Visayas	SC		50		1	46	<0.001	NR	NR	<0.001	NR
Rice, brown Philippines, 1992, 92/277 Mindanao	SC		50		1	46	<0.001	NR	NR	<0.001	NR
Rice, white Philippines, 1992, 92/277 Mindanao	SC		50		1	46	<0.001	NR	NR	<0.001	NR
Broadcast treatment after transplanting followed by foliar treatment (Maycey *et al.*, 1994a)											
Rice, brown Philippines, 1992, 92/277 Luzon	GR SC		50		2	51	<0.001	NR	NR	<0.001	NR
Rice, white Philippines, 1992, 92/277 Luzon	GR SC		50		2	51	<0.001	NR	NR	<0.001	NR
Rice, brown Philippines, 1992, 92/277 Visayas	GR SC		50		2	46	<0.001	NR	NR	<0.001	NR
Rice, white Philippines, 1992, 92/277 Visayas	GR SC		50		2	46	<0.001	NR	NR	<0.001	NR
Rice, brown Philippines, 1992, 92/277 Mindanao	GR SC		50		2	46	<0.001	NR	NR	<0.001	NR
Rice, white, Philippines, 1992, 92/277 Mindanao	GR SC		50		2	46	<0.001	NR	NR	<0.001	NR

Commodity, Country, Year, Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Foliar treatment, (Maycey *et al.*, 1994b)											
Rice, brown Taiwan, 1993, 92/275 Chitong Li **Plot 1**	SC		50		1	79	<0.001	NR	NR	<0.001	NR
Rice, brown Taiwan, 1993, 92/275 Chitong Li **Plot 2**	SC		50		1	79	<0.001	NR	NR	<0.001	NR
Foliar treatment, 3 replicates (Maycey *et al.*, 1994c)											
Rice, brown Thailand, 1992/1993, 92/276 Supanuri Rice Research Station	SC		50		1	49	<0.001 <0.001 <0.001	NR	NR	<0.001 0.001 0.001	NR
Rice, white Thailand, 1992/1993, 92/276 Supanuri Rice Research Station	SC		50		1	49	<0.001	NR	NR	<0.001	NR
Rice, brown Thailand, 1992/1993, 92/276 Supanuri Rice Research Station	SC		50		1	45	<0.001 <0.001 <0.001	NR	NR	0.001 0.001 0.001	NR
Rice, white Thailand, 1992/1993, 92/276 Supanuri Rice Research Station	SC		50		1	45	<0.001	NR	NR	<0.001	NR
Rice, brown Thailand, 1992/1993, 92/276 Supanuri	SC		50		1	48	<0.001 0.002 0.001	NR	NR	<0.001 <0.001 <0.001	NR
Rice, white Thailand, 1992/1993, 92/276 Supanuri	SC		50		1	48	<0.001	NR	NR	<0.001	NR
Rice, brown Thailand, 1992/1993, 92/276 Nontaburi	SC		50		1	45	<0.001 0.001 <0.001	NR	NR	<0.001 <0.001 <0.001	NR
Rice, white Thailand, 1992/1993, 92/276 Nontaburi	SC		50		1	45	<0.001 <0.001 <0.001	NR	NR	<0.001 <0.001 0.001	NR

[1] residues reported as ND (not detectable) in the original report are shown as <0.00x (i.e. MLD)
[2] analysed sample: ear
NR: not reported

Table 70. Residues in sorghum grain from seed treatment or foliar applications.

Country, Year Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Foliar treatment at stage BBCH 85**, 2 replicates (Keats, 1996n)**											
Australia, 1995, AK96074	UL		10		1	0 1 3 7 21	0.24 0.18 0.11 0.099 0.049 0.05 0.052 0.049 0.011 0.011	0.004 0.003 0.002 0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	0.002 0.003 0.04 0.04 0.011 0.01 0.009 0.01 0.011 0.008	0.004 0.003 0.019 0.017 0.008 0.009 0.008 0.008 0.004 0.003	NR
Australia, 1995, AK96074	UL		20		1	0 1	0.33 0.29 0.18	0.008 0.006 0.004	0.007 0.005 0.017	0.002 0.002 0.005	NR

Country, Year Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
							0.17	0.004	0.018	0.006	
						3	0.065	0.002	0.012	0.007	
							0.061	<0.002	0.01	0.007	
						7	0.059	<0.002	0.013	0.009	
							0.057	<0.002	0.013	0.009	
						21	0.017	<0.002	0.009	0.005	
							0.015	<0.002	0.01	0.005	
Foliar treatment at stage BBCH 75 (Keats, 1996o)											
Australia, 1996, AK96075	UL		10		1	0	0.093	0.004	0.008	0.002	NR
						1	0.066	0.003	0.01	0.003	
						3	0.025	0.002	0.014	0.005	
						7	0.024	<0.002	0.013	0.005	
						17	0.027	<0.002	0.013	0.004	
Australia, 1996, AK96075	UL		20		1	0	0.12	0.005	0.004	<0.002	NR
						1	0.092	0.003	0.005	0.002	
						3	0.071	0.002	0.006	0.003	
						7	0.058	0.002	0.006	0.004	
						17	0.031	<0.002	0.006	0.006	
Foliar treatment at maturity, BBCH 87, 2 replicates (Keats, 1996p)											
Australia, 1996, AK96076	UL		10		1	0	0.27	0.058	0.01	<0.002	NR
							0.29	0.055	0.011	<0.002	
						1	0.14	0.023	0.099	0.007	
							0.13	0.021	0.074	0.006	
						3	0.10	0.017	0.039	0.003	
							0.11	0.016	0.041	0.004	
						7	0.087	0.013	0.034	0.004	
							0.077	0.013	0.032	0.004	
						21	0.019	0.002	0.022	<0.002	
							0.013	0.002	0.015	<0.002	
Australia, 1996, AK96076	UL		20		1	0	0.42	0.078	0.025	<0.002	NR
							0.43	0.079	0.022	<0.002	
						1	0.35	0.059	0.059	0.003	
							0.34	0.053	0.062	0.003	
						3	0.12	0.019	0.044	0.003	
							0.12	0.018	0.04	0.003	
						7	0.11	0.016	0.052	0.006	
							0.11	0.016	0.052	0.006	
						21	0.032	0.005	0.032	0.003	
							0.032	0.004	0.03	0.003	
Foliar treatment at maturity, BBCH 87**, 2 replicates (Keats, 1998a)**											
Australia, 1998, AK98024	SC		1.25		1	0	0.011	0.002	<0.002	<0.002	NR
							0.01	0.002	<0.002	<0.002	
						4	0.002	<0.002	<0.002	<0.002	
						7	0.008	<0.002	<0.002	<0.002	
							0.008	<0.002	0.002	<0.002	
						15	0.002	<0.002	<0.002	<0.002	
						21	<0.002	<0.002	<0.002	<0.002	
						28	<0.002	<0.002	<0.002	<0.002	
Australia, 1998, AK98024	SC		2.5		1	0	0.036	0.008	0.002	<0.002	NR
							0.038	0.008	0.002	<0.002	
						4	0.002	<0.002	<0.002	<0.002	
						7	0.008	<0.002	0.003	<0.002	
						15	0.003	<0.002	<0.002	0.002	
						21	<0.002	<0.002	<0.002	<0.002	
						29	<0.002	<0.002	<0.002	<0.002	
Australia, 1998, AK98024	SC		5.0		1	0	0.086	0.02	0.007	0.009	NR
							0.082	0.021	0.006	0.009	
						4	0.003	<0.002	<0.002	<0.002	
						7	0.009	<0.002	0.004	<0.002	
							0.008	<0.002	0.003	0.003	
						15	0.01	<0.002	<0.002	0.003	
							0.009	<0.002	<0.002	<0.002	
						21	0.002	<0.002	<0.002	<0.002	
						29	<0.002	<0.002	<0.002	<0.002	
Foliar treatment at maturity, BBCH 87, 2 replicates (Keats, 1998b)											
Australia, 1998, AK98025	UL		1.25		1	0	0.008	0.002	<0.002	<0.002	NR
							0.01	0.002	<0.002	<0.002	
						2	0.005	0.002	<0.002	<0.002	
							0.003	<0.002	<0.002	0.003	
						4	0.002	<0.002	0.002	<0.002	
							0.002	<0.002	<0.002	<0.002	

Country, Year Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
						7	0.002	<0.002	0.002	<0.002	
						15	<0.002	<0.002	<0.002	<0.002	
						21	0.002	≤0.002	≤0.002	≤0.002	
							<0.002	<0.002	<0.002	<0.002	
						28	<0.002	<0.002	<0.002	<0.002	
Australia, 1998, AK98025	UL		2.5		1	0	0.009	0.003	<0.002	0.002	NR
							0.011	0.005	<0.002	<0.002	
						2	0.014	<0.002	<0.002	0.005	
							0.01	<0.002	0.003	0.007	
						4	0.004	<0.002	0.007	<0.002	
							0.006	<0.002	0.005	<0.002	
						7	0.005	<0.002	0.004	<0.002	
							0.005	<0.002	0.004	<0.002	
						15	0.002	<0.002	0.002	<0.002	
							0.002	<0.002	0.002	<0.002	
						21	<0.002	<0.002	0.002	<0.002	
							<0.002	<0.002	<0.002	<0.002	
						28	<0.002	<0.002	<0.002	<0.002	
Australia, 1998, AK98025	UL		5.0		1	0	0.024	0.006	<0.002	0.008	NR
							0.02	0.008	<0.002	0.004	
						2	0.011	0.002	0.003	0.004	
							0.017	0.003	<0.002	0.004	
						4	0.005	<0.002	0.006	0.004	
							0.003	0.003	0.005	0.003	
						7	0.01	<0.002	0.006	<0.002	
							0.009	<0.002	0.006	<0.002	
						15	0.004	<0.002	0.004	<0.002	
							0.002	<0.002	0.004	<0.002	
						21	0.002	<0.002	0.002	<0.002	
						28	<0.002	<0.002	<0.002	<0.002	
Australia, 1998, AK98025	UL		7.5		1	0	0.035	0.006	<0.002	0.012	NR
							0.041	0.006	<0.002	0.008	
						2	0.018	0.002	0.002	0.006	
							0.014	0.002	<0.002	0.008	
						4	0.012	<0.002	0.008	0.003	
							0.009	0.003	0.009	0.005	
						7	0.016	<0.002	0.013	<0.002	
							0.017	<0.002	0.013	<0.002	
						15	0.002	<0.002	0.006	<0.002	
							0.006	<0.002	0.005	<0.002	
						21	0.003	<0.002	0.004	<0.002	
							0.004	<0.002	0.004	<0.002	
						28	<0.002	<0.002	<0.002	<0.002	
Foliar treatment at stage BBCH 73, 2 replicates (Keats, 1998c)											
Australia, 1998, AK98027	UL		1.25		1	0	0.008	<0.002	<0.002	<0.002	NR
							0.002	<0.002	<0.002	<0.002	
						2	0.006	<0.002	<0.002	0.002	
							0.002	<0.002	<0.002	0.002	
						4	<0.002	<0.002	0.002	<0.002	
							<0.002	<0.002	<0.002	<0.002	
						7	<0.002	<0.002	0.002	<0.002	
						15	≤0.002	≤0.002	≤0.002	≤0.002	
						21	<0.002	<0.002	<0.002	<0.002	
						28	<0.002	<0.002	<0.002	<0.002	
Australia, 1998, AK98027	UL		2.5		1	0	0.007	0.003	<0.002	<0.002	NR
							0.013	<0.002	<0.002	<0.002	
						2	0.01	<0.002	<0.002	0.004	
							0.014	<0.002	0.003	0.008	
						4	0.003	<0.002	0.005	<0.002	
							0.002	<0.002	0.003	0.003	
						7	0.002	<0.002	0.002	<0.002	
							0.002	<0.002	0.002	<0.002	
						15	<0.002	<0.002	<0.002	<0.002	
						21	<0.002	<0.002	<0.002	<0.002	
						28	<0.002	<0.002	<0.002	<0.002	
Australia, 1998, AK98027	UL		5.0		1	0	0.017	0.006	<0.002	0.002	NR
							0.011	0.002	<0.002	0.003	
						2	0.012	0.002	0.003	0.002	
							0.016	0.002	<0.002	0.004	
						4	0.003	<0.002	0.003	0.004	
							0.005	<0.002	0.005	0.003	
						7	0.002	<0.002	0.002	<0.002	

Country, Year Reference	Application					PHI,	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.	days	Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
						 15 21 28	0.003 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002	0.004 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002	
Australia, 1998, AK98027	UL		7.5		1	0 2 4 7 15 21 28	0.025 0.017 0.016 0.015 0.009 0.005 0.004 0.002 <0.002 <0.002 <0.002	0.003 0.005 0.002 0.002 <0.002 0.003 <0.002 <0.002 <0.002 <0.002 <0.002	<0.002 0.003 <0.002 0.003 0.006 0.004 0.007 0.003 <0.002 <0.002 <0.002	0.006 0.002 0.006 0.008 0.006 0.002 <0.002 <0.002 <0.002 <0.002 <0.002	NR
Seed treatment, GAP pending, 2 replicates (Keats, 1998g)											
Australia, 1998, AK98030	FS	750 1500			1	138	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
Seed treatment, GAP pending, 2 replicates (Keats, 1998h)											
Australia, 1998, AK98031	FS	750 1500			1	104	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR

Table 71. Residues in wheat grain from seed treatment or foliar applications (most trials include 2 replicates).

Crop, Country, Year, Reference	Application					PHI,	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.	days	Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Seed treatment (Muller, 1994k)											
Wheat, winter, France, 1992/3, 93-507E1	FS	1000			1	251	<0.002	<0.002	<0.002	NR	<0.002
Seed treatment (Richard and Muller, 1994h)											
Wheat, winter, France, 1992/3, 93-508B1	FS	1500			1	279	<0.002	<0.002	<0.002	NR	<0.002
Wheat, winter, France, 1992/3, 93-508C1	FS	1000 1500			1	286	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR	<0.002 <0.002
Wheat, winter, France, 1992/3, 93-508D1	FS	1500			1	269	<0.002	<0.002	<0.002	NR	<0.002
Wheat, winter, France, 1992/3, 93-508E1	FS	1500			1	264	<0.002	<0.002	<0.002	NR	<0.002
Wheat, winter, France, 1992/3, 93-508F1	FS	1500			1	268	<0.002	<0.002	<0.002	NR	<0.002
Wheat, winter, France, 1992/3, 93-508K1	FS	1000 1500			1	226	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR	<0.002 <0.002
Seed treatment (Muller, 1995b)											
Wheat, winter, France, 1993/4, 94-500BX1	FS	1500			1	244	<0.002 0.003	<0.002 <0.002	<0.002 <0.002	NR	<0.002 0.002
Wheat, winter, France, 1993/4, 94-500RN1	FS	1500			1	260	<0.002	<0.002	<0.002	NR	<0.002
Wheat, winter, France, 1993/4, 94-500AM1	FS	1500			1	286	<0.002	<0.002	<0.002	NR	<0.002
Wheat, winter, France, 1993/4, 94-500DJ1	FS	1500			1	262	<0.002	<0.002	<0.002	NR	<0.002

Crop, Country, Year, Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Wheat, winter, France, 1993/4, 94-500LY1	FS	1500			1	245	<0.002	<0.002	<0.002	NR	<0.002
Wheat, winter, France, 1993/4, 94-500AV1	FS	1500			1	245	<0.002	<0.002	<0.002	NR	<0.002
Seed treatment (Muller, 1996k)											
Wheat, spring, France, 1995, 95-507BX1	FS	500			1	140	<0.002	<0.002	<0.002	NR	<0.002
Wheat, spring, France, 1995, 95-507AM1	FS	500			1	128	<0.002	<0.002	<0.002	NR	<0.002
Wheat, spring, France, 1995, 95-507RS1	FS	500			1	131	<0.002	<0.002	<0.002	NR	<0.002
Wheat, spring, France, 1995, 95-507LY1	FS	500			1	145	<0.002	<0.002	<0.002	NR	<0.002
Seed treatment (Muller, 1996j)											
Wheat, spring, France, 1995 95-518RS1	FS	500			1	131	<0.002	<0.002	<0.002	NR	<0.002
Seed treatment (Maestracci, 1998a)											
Wheat, winter, Greece, 1997/8, 96600GR1	FS	750			1	152	0.004 0.03	<0.002 <0.002	<0.002 0.003	<0.002 <0.002	<0.002 <0.002
Seed treatment (Souvinet, 1999)											
Wheat, winter, Greece, 1997/8, 97741GR1	FS	750			1	181	0.003 0.003	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
Foliar treatment at stage BBCH 51 (Muller, 1996a)											
Wheat, winter, Poland, 1995, 95795PL1	WG		20	6.67	1	44	<0.002	<0.002	<0.002	<0.002	<0.002
Foliar treatment no reference (summary table, no GLP report available)											
Wheat, winter, Russia, 1997, Krasnodar	EC		24	12	1	27	<0.005	NR	NR	NR	NR
Wheat, winter, Russia, 1997, Rostov	EC		20	10	1	25	<0.005	NR	NR	NR	NR
Wheat, winter, Russia, 1997, Voronesh	EC		20	10	1	38	<0.005	NR	NR	NR	NR
Wheat, spring, Russia, 1998, Volgograd	EC		21	10.5	1	49	<0.005	NR	<0.004	<0.005	NR
Wheat, spring, Russia, 1998, Saratov	EC		21	10.5	1	48	<0.005	NR	<0.004	<0.005	NR

NR: not reported

Table 72. Residues in sugar cane from soil and foliar applications.

Country, Year, Reference	Application			PHI, days	Residues, mg/kg				
	Form	g ai/ha	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Soil treatment, in furrow spray at planting, 2 replicates (Keats, 1997n)									
Australia, 1995, AUS94i48r Tully Queensland	WG	100 200 400	1	340	<0.002 0.002 0.002	<0.002 <0.002 <0.002	<0.002 <0.002 0.003	<0.002 <0.002 <0.002	NR

Country, Year, Reference	Application			PHI, days	Residues, mg/kg				
	Form	g ai/ha	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Australia, 1995, AUS94i48r Tully Queensland	WG	100 200 400	2	245	0.002 <0.002 0.002 0.003	<0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 0.003	<0.002 <0.002 <0.002 <0.002	NR
Spray on the bottom of the stalk, 2 replicates (Keats, 1997m)									
Australia, 1995, AUS94i74r Mowilyan Queensland	WG	100	1	181	0.002	<0.002	0.002	<0.002	NR
Australia, 1995, AUS94i74r Mowilyan Queensland	WG	200	1	181	0.002	<0.002	0.002	0.002	NR
Australia, 1995, AUS94i74r Mowilyan Queensland	WG	50	2	134	<0.002 0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
Australia, 1995, AUS94i74r Mowilyan Queensland	WG	100	2	53	0.025 0.02	<0.002 <0.002	0.008 0.007	0.003 0.003	NR
Spray on the bottom of the stalk, 2 replicates (Keats, 1997k)									
Australia, 1996, 97NST14 Kurrimine Beach Queensland	SC	75	1	101	<0.002	<0.002	<0.002	<0.002	NR
Soil treatment, in furrow spray followed by stool spray, 2 replicates (Keats, 1997k)									
Australia, 1996, AUS94i74cr Kurrimine Beach Queensland	WG + SC	100 soil + 50 foliar	1 + 2	95	0.002	<0.002	0.002	<0.002	NR
Australia, 1996, AUS94i74cr Kurrimine Beach Queensland	WG + SC	200 soil + 100 foliar	1 + 2	95	0.002 0.003	<0.002 <0.002	0.003 0.003	0.002 0.002	NR
AUS94i74cr Kurrimine Beach Queensland	WG + SC	400 soil + 200 foliar	1 + 2	95	0.005	<0.002	0.006	0.008	NR
Soil treatment, in furrow spray at planting, 2 replicates (Garcia and Oliveira, 1994a)									
Brazil, 1994, 154/94 Sao Paulo	WG	400 800	1	87	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
Soil treatment, in furrow spray at planting, 2 replicates (Garcia and Oliveira, 1994b)									
Brazil, 1994 155/94 Sao Paulo	WG	400 800	1	87	<0.01 0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 0.01

NR: not reported

Oilseeds - cotton and sunflower (Tables 73, 74). Both soil and foliar pests can be controlled with fipronil. For trials where fipronil has been applied as a seed treatment, as in-furrow granules or as a soil incorporated pre-plant spray, only the residues of fipronil and its soil degradation products MB 45950, MB 46136 and RPA 200766 have been reported. For trials using foliar or combined soil/foliar applications fipronil-desulfinyl residues are included.

Table 73. Residues in cotton seed after varied applications of fipronil.

Country, Year, Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Seed treatment, 2 replicates(Keats, 1997i)											
Australia, 1995, AK97018 Clare, QLD	FS	5000 10 000			1	169	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
Foliar treatment, 2 replicates (Keats, 1997e)											
Australia, 1996, AK97016 Breeza, NSW	EC		200		4	14	<0.002 0.002	<0.002 <0.002	<0.002 <0.002	0.002 <0.002	NR
Seed treatment followed by foliar treatments, 2 replicates (Keats, 1997f)											
Australia, 1996, AK97015 Kincora Queensland	FS + SC +	5000	 25		1 1						

Country, Year, Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
	ULV		25		1	113	<0.002	<0.002	<0.002	<0.002	NR
Australia, 1996, AK97015 Kincora Queensland	FS + SC + ULV	10 000	50 50		1 1 1	113	<0.002 <0.002	<0.002 <0.002	<0.002 0.002	<0.002 <0.002	NR
Australia, 1996, AK97015 Kincora Queensland	FS + SC + ULV	10 000	100 100		1 1 1	113	<0.002	<0.002	<0.002	<0.002	NR
Australia, 1996, AK97015 Kincora Queensland	FS + SC + ULV	5000	25 25		1 1 3	28	0.003 0.002	0.002 <0.002	0.003 0.003	0.002 <0.002	NR
Australia, 1996, AK97015 Kincora Queensland	FS + SC + ULV	10 000	50 50		1 1 3	28	0.002 <0.002	<0.002 <0.002	0.002 0.002	0.002 0.002	NR
Australia, 1996, AK97015 Kincora Queensland	FS + SC + ULV	10 000	100 100		1 1 3	28	0.002	<0.002	0.002	<0.002	NR
Seed treatment followed by foliar treatments, 2 replicates (Keats, 1997g)											
Australia, 1996, AK97014 Breeza NSW	FS + SC + ULV	5000	25 25		1 1 1	109	<0.002	<0.002	<0.002	<0.002	NR
Australia, 1996, AK97014 Breeza NSW	FS + SC + ULV	10 000	50 50		1 1 1	109	<0.002	<0.002	<0.002	<0.002	NR
Australia, 1996, AK97014 Breeza NSW	FS + SC + ULV	10 000	100 100		1 1 1	109	<0.002	<0.002	<0.002	<0.002	NR
Australia, 1996, AK97014 Breeza NSW	FS + SC + ULV	5000	25 25		1 1 3	31	0.004 0.002	<0.002 <0.002	0.004 0.003	<0.002 <0.002	NR
Australia, 1996, AK97014 Breeza NSW	FS + SC + ULV	10 000	50 50		1 1 3	31	0.002	<0.002	0.003	<0.002	NR
Australia, 1996, AK97014 Breeza NSW	FS + SC + ULV	10 000	100 100		1 1 3	31	<0.002	<0.002	<0.002	<0.002	NR
Foliar treatment, 2 replicates (Lynch, 1998)											
Australia, 1998, AK99029 Jambin, QLD **Single applications to separate plots**	SC		35.4	44.8	1 1 1 1 1	0 15 30 43 57	0.014 0.016 0.01 0.008 0.002 0.005 <0.002 <0.002 <0.002	0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	0.004 0.009 0.004 0.006 0.005 0.004 0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002 <0.002	NR

Country, Year, Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
					1	71	<0.002	<0.002	<0.002	<0.002	
					1	85	<0.002	<0.002	<0.002	<0.002	
					1	99	<0.002	<0.002	<0.002	<0.002	
Australia, 1998,	SC		71.4	92.7	1	0	0.027	0.002	<0.002	<0.002	NR
AK99029							0.034	0.004	0.004	<0.002	
Jambin, QLD					1	15	0.02	0.006	0.007	<0.002	
							0.026	0.009	0.007	<0.002	
Single					1	30	0.011	0.004	0.006	<0.002	
applications							0.018	0.003	0.005	<0.002	
to separate plots					1	43	0.005	0.002	0.006	<0.002	
							0.009	0.004	0.01	<0.002	
					1	57	0.002	<0.002	0.002	<0.002	
							<0.002	<0.002	0.004	<0.002	
					1	71	<0.002	<0.002	<0.002	<0.002	
					1	85	<0.002	<0.002	<0.002	<0.002	
					1	99	<0.002	<0.002	<0.002	<0.002	
Foliar treatment, 2 replicates (Lynch, 1999)											
Australia, 1998,	SC		25	28.4	1	0	<0.002	<0.002	<0.002	<0.002	NR
AK99032					1	15	<0.002	<0.002	<0.002	<0.002	
Wee Waa					1	29	<0.002	<0.002	<0.002	<0.002	
NSW					1	43	<0.002	<0.002	<0.002	<0.002	
Single					1	57	<0.002	<0.002	<0.002	<0.002	
applications					1	71	<0.002	<0.002	<0.002	<0.002	
to separate plots					1	85	<0.002	<0.002	<0.002	<0.002	
					1	99	<0.002	<0.002	<0.002	<0.002	
Australia, 1998,	SC		50	56.8	1	0	0.003	<0.002	<0.002	<0.002	NR
AK99032							0.003	<0.002	<0.002	<0.002	
Wee Waa					1	15	<0.002	<0.002	<0.002	<0.002	
NSW					1	29	<0.002	<0.002	<0.002	<0.002	
Single					1	43	<0.002	<0.002	<0.002	<0.002	
applications					1	57	<0.002	<0.002	<0.002	<0.002	
to separate plots					1	71	<0.002	<0.002	<0.002	<0.002	
					1	85	<0.002	<0.002	<0.002	<0.002	
					1	99	<0.002	<0.002	<0.002	<0.002	
Foliar treatment, 2 replicates (Garcia and Oliveira, 1994d)											
Brazil, 1994,	WG		100		2	15	<0.01	<0.01	<0.01	<0.01	<0.01
028/94PC			200		2	15	0.01	<0.01	<0.01	<0.01	<0.01
							<0.01	<0.01	<0.01	<0.01	<0.01
Foliar treatment, 2 replicates (Carringer, 1998a)											
Mexico, 1996,	WG		50		6	16	<0.004	<0.002	<0.005	<0.01	NR
12046-01						31	<0.004	<0.002	<0.005	<0.01	
							<0.01	<0.01	<0.01	<0.01	
						54	<0.004	<0.002	<0.005	<0.01	
						61	<0.004	<0.002	<0.01	<0.01	
Mexico, 1996, 12046-01	WG		250		6	54	<0.01	<0.002	<0.01	<0.01	NR
Mexico, 1996,	WG		50		6	17	<0.01	<0.002	<0.01	<0.01	NR
12046-02						31	<0.004	<0.002	<0.01	<0.01	
						46	<0.01	<0.002	<0.01	<0.01	
						61	<0.01	<0.002	<0.01	<0.01	
Mexico, 1996,	WG		250		6	46	0.037	<0.002	0.037	0.067	NR
12046-02							<0.01	<0.002	<0.01	0.02	
Soil treatment in-furrow at sowing followed by foliar treatments, or foliar only, 2 replicates (Norris, 1995a)											
USA, 1994,	WG		168 inc		1						
94-0343TX			+								
			84 foliar		2	45	<0.004	<0.002	<0.005	<0.003	<0.005
							<0.004	<0.002	<0.005	<0.01	<0.005
USA, 1994,	WG		84		4	45	<0.004	<0.002	<0.005	<0.003	<0.005
94-0343TX			foliar				<0.01	<0.002	<0.005	<0.003	<0.005
USA, 1994,	WG		168 inc		1						
94-0344NC			+								
			84 foliar		2	45	<0.004	<0.002	<0.005	<0.003	<0.005
USA, 1994, 94-0344NC	WG		84 foliar		4	45	<0.004	<0.002	<0.005	<0.003	<0.005
USA, 1994,	WG		168 inc		1						
94-0345AZ			+								
			84 foliar		2	45	<0.004	<0.002	<0.005	<0.003	<0.005
USA, 1994, 94-0345AZ	WG		84 foliar		4	45	<0.004	<0.002	<0.005	<0.01	<0.005
USA, 1994,	WG		168 inc		1						
94-0346CA			+								

Country, Year, Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
			84 foliar		2	46	<0.004	<0.002	<0.005	<0.003	<0.005
USA, 1994, 94-0346CA	WG		84 foliar		4	46	<0.004	<0.002	<0.005	<0.003	<0.005
USA, 1994, 94-0347AR	WG		168 inc + 84 foliar		1 2	45	<0.01 <0.004	<0.002 <0.002	<0.005 <0.005	<0.003 <0.003	<0.005 <0.005
USA, 1994, 94-0347AR	WG		84 foliar		4	45	<0.004	<0.002	<0.005	<0.003	<0.005
USA, 1994, 94-0348MS	WG		168 inc + 84 foliar		1 2	45	<0.004	<0.002	<0.005	<0.003	<0.005
USA, 1994, 94-0348MS	WG		84 foliar		4	45	<0.004	<0.002	<0.005	<0.003	<0.005
USA, 1994, 94-0349AR	WG		168 inc + 84 foliar		1 2	46	<0.004	<0.002	<0.005	<0.003	<0.005
USA, 1994, 94-0349AR	WG		84 foliar		4	46	<0.004	<0.002	<0.005	<0.003	<0.005
USA, 1994, 94-0350LA	WG		168 inc + 84 foliar		1 2	57	<0.004 <0.004	<0.002 <0.002	<0.005 <0.005	<0.01 <0.003	<0.005 <0.005
USA, 1994, 94-0350LA	WG		84 foliar		4	57	<0.004	<0.002	<0.005	<0.01	<0.005
USA, 1994, 94-0351TX	WG		168 inc + 84 foliar		1 2	46	<0.004 <0.004	<0.002 <0.002	<0.005 <0.005	<0.003 <0.01	<0.005 <0.005
USA, 1994, 94-0351TX	WG		84 foliar		4	46	<0.004 <0.004	<0.002 <0.002	<0.01 <0.005	<0.01 <0.01	<0.005 <0.005
USA, 1994, 94-0352GA	WG		168 inc + 84 foliar		1 2	44	<0.004	<0.002	<0.005	<0.003	<0.005
USA, 1994, 94-0352GA	WG		84 foliar		4	44	<0.004	<0.002	<0.005	<0.003	<0.005
USA, 1994, 94-0353TX	WG		1681 inc + 420 foliar		1 4	45	0.056 0.049	0.021 0.017	0.069 0.066	0.173 0.143	<0.004 <0.004
Soil treatment in furrow at sowing followed by foliar treatments, or foliar only, 2 replicates (Norris, 1997a)											
USA, 1995, 95-0023NC	WG		168 inc + 84 foliar		1 2	44	<0.003	<0.003	<0.004	<0.003	<0.004
USA, 1995, 95-0023NC	WG		84 foliar		4	44	<0.003	<0.003	<0.004	<0.003	<0.004
USA, 1995, 95-0026AR	WG		168 inc + 84 foliar		1 2	45	<0.003	<0.003	<0.004	<0.003	<0.004
USA, 1995, 95-0026AR	WG		84 foliar		4	45	<0.003	<0.003	<0.004	<0.003	<0.004
USA, 1995, 95-0027MS	WG		168 inc + 84 foliar		1 2	44	<0.003 <0.003	<0.003 <0.003	<0.004 <0.01	<0.003 <0.003	<0.004 <0.01
USA, 1995, 95-0027MS	WG		84 foliar		4	44	<0.003	<0.003	<0.01	<0.003	<0.01
USA, 1995, 95-0028LA	WG		168 inc + 84 foliar		1 2	45	<0.003 <0.003	<0.003 <0.003	<0.004 <0.004	<0.01 0.012	<0.004 <0.004
USA, 1995, 95-0028LA	WG		84 foliar		4	45	<0.003	<0.003	<0.004	<0.01 <0.01	<0.004
USA, 1995, 95-0029TX	WG		168 inc + 84 foliar		1 2	43	<0.003	<0.003	<0.004	<0.003	<0.004
USA, 1995, 95-0029TX	WG		84 foliar		4	43	<0.003	<0.003	<0.004	<0.003	<0.004
USA, 1995, 95-0030TX	WG		168 inc + 84 foliar		1 2	44	<0.003	<0.003	<0.004	<0.003	<0.004
USA, 1995,	WG		84 foliar		4	44	<0.003	<0.003	<0.004	<0.003	<0.004

Country, Year, Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
95-0030TX											
USA, 1995, 95-0031OK	WG		168 inc + 84 foliar		1 2	44	<0.003	<0.003	<0.004	<0.003	<0.004
USA, 1995, 95-0031OK	WG		84 foliar		4	44	<0.003	<0.003	<0.004	<0.003	<0.004
USA, 1995, 95-0032TX	WG		168 inc + 84 foliar		1 2	46	<0.003	<0.003	<0.004	<0.003	<0.004
USA, 1995, 95-0032TX	WG		84 foliar		4	46	<0.003 <0.003	<0.003 <0.003	<0.01 <0.004	<0.01 <0.01	<0.004 <0.004
USA, 1995, 95-0033TX	WG		168 inc + 84 foliar		1 2	46	<0.003 <0.003	<0.003 <0.003	<0.004 <0.004	<0.003 <0.01	<0.004 <0.004
USA, 1995, 95-0033TX	WG		84 foliar		4	46	<0.003	<0.003	<0.004	<0.01	<0.004
USA, 1995, 95-0034AZ	WG		168 inc + 84 foliar		1 2	45	<0.01 <0.003	<0.003 <0.003	<0.004 <0.004	<0.01 <0.003	<0.004 <0.004
USA, 1995, 95-0034AZ	WG		84 foliar		4	45	<0.003 <0.01	<0.003 <0.01	<0.004 <0. 010	<0.003 <0.01	<0.004 <0.004
USA, 1995, 95-0035CA	WG		168 inc + 84 foliar		1 2	45	<0.01 <0.01	<0.003 <0.003	<0.004 <0.004	0.011 <0.01	<0.004 <0.004
USA, 1995, 95-0035CA	WG		84 foliar		4	45	0.011 <0.01	<0.003 <0.003	<0.01 <0.004	0.025 0.013	<0.004 <0.004
USA, 1995, 95-0036CA	WG		168 inc + 84 foliar		1 2	46	<0.003 <0.01	<0.003 <0.003	<0.004 <0.01	<0.01 0.021	<0.004 <0.004
USA, 1995, 95-0036CA	WG		84 foliar		4	46	<0.01 <0.01	<0.003 <0.003	<0.01 <0.004	0.019 0.018	<0.004 <0.004
Foliar treatment, 2 replicates (Norris, 1997b)											
USA, 1995, 95-0276GA	WG		56		6	43	<0.003 <0.01	<0.003 <0.003	<0.01 <0.01	<0.01 <0.01	<0.004 <0.004
USA, 1995, 95-0277LA	WG		56		6	45	<0.003 0.015	<0.003 <0.003	<0.01 <0.01	<0.01 <0.01	<0.004 <0.004
USA, 1995, 95-0278TX	WG		56		6	46	<0.01 <0.01	<0.003 <0.003	<0.01 <0.01	<0.01 <0.01	<0.004 <0.004
USA, 1995, 95-0279TX	WG		56		6	44	<0.01 <0.01	<0.003 <0.003	<0.01 <0.01	<0.01 0.01	<0.004 <0.004
USA, 1995, 95-0280CA	WG		56		6	46	<0.01 0.015	<0.003 <0.003	<0.01 <0.01	0.015 0.025	<0.004 <0.004
Foliar treatment, 2 replicates (Norris, 1998)											
USA, 1996, 10669-01NC	EC		56		6	43	<0.003	<0.003	<0.004	<0.003	NR
USA, 1996, 10669-02GA	EC		56		6	44	<0.003 <0.003	<0.003 <0.003	<0.004 <0.01	<0.01 <0.01	NR
USA, 1996, 10669-03AR	EC		56		6	45	<0.003	<0.003	<0.004	<0.01	NR
USA, 1996, 10669-04AR	EC		56		6	46	<0.003 <0.003	<0.003 <0.003	<0.004 <0.01	<0.01 <0.01	NR
USA, 1996, 10669-05MS	EC		56		6	45	<0.003	<0.003	<0.004	<0.01	NR
USA, 1996, 10669-06LA	EC		56		6	45	<0.003	<0.003	<0.004	<0.01	NR
USA, 1996, 10669-07TX	EC		56		6	47	<0.01	<0.003	<0.01	<0.01	NR
USA, 1996, 10669-08TX	EC		56		6	45	<0.01	<0.003	<0.01	<0.01	NR
USA, 1996, 10669-09OK	EC		56		6	45	<0.003 <0.01	<0.003 <0.003	<0.01 <0.01	<0.01 <0.01	NR
USA, 1996, 10669-10TX	EC		56		6	47	<0.01	<0.003	<0.01	<0.01	NR
USA, 1996, 10669-11TX	EC		56		6	46	<0.01	<0.003	<0.01	<0.01	NR
USA, 1996, 10669-12AZ	EC		56		6	45	<0.01	<0.003	<0.01	<0.01	NR
USA, 1996, 10669-13CA	EC		56		6	44	<0.01	<0.003	<0.01	<0.01	NR
USA, 1996,	EC		56		6	45	<0.01	<0.003	<0.004	<0.01	NR

Country, Year, Reference	Application					PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
100669-14CA							<0.01	<0.003	0.011	<0.01	
Foliar treatment, GAP pending, 2 replicates (Gough, 1999)											
USA, 1997, 13499-01 Kerman CA	EC		56		3	75	<0.003	<0.003	<0.004	<0.01	NR
USA, 1997, 13499-01 Kerman CA	EC		56		4	75	<0.003 <0.003	<0.003 <0.003	<0.004 <0.004	<0.01 <0.003	NR
USA, 1997, 13499-02 Tulare CA	EC		56		3	75	<0.003	<0.003	<0.004	<0.003	NR
USA, 1997, 13499-02 Tulare CA	EC		56		4	75	<0.003 <0.003	<0.003 <0.003	<0.004 <0.01	<0.003 <0.01	NR
USA, 1997, 13499-03 Brawley CA	EC		56		3	76	<0.003 <0.003	<0.003 <0.003	<0.004 <0.01	<0.003 <0.01	NR
USA, 1997, 13499-03 Brawley CA	EC		56		4	76	<0.003	<0.003	<0.01	<0.01	NR
Foliar treatment, GAP pending, 2 replicates (Macy, 1998)											
USA, 1997, 13501-01AR	EC		56		3	58	<0.003	<0.003	<0.004	<0.003	NR
USA, 1997, 13501-01AR	EC		56		4	58	<0.003	<0.003	<0.004	<0.003	NR
USA, 1997, 13501-02AR	EC		56		3	61	<0.003	<0.003	<0.004	<0.003	NR
USA, 1997, 13501-02AR	EC		56		4	61	<0.003	<0.003	<0.004	<0.003	NR
USA, 1997, 13501-03LA	EC		56		3	60	<0.003	<0.003	<0.004	<0.003	NR
USA, 1997, 13501-03LA	EC		56		4	60	<0.003	<0.003	<0.004	<0.01	NR
USA, 1997, 13501-04LA	EC		56		3	61	<0.003	<0.003	<0.004	<0.003	NR
USA, 1997, 13501-04LA	EC		56		4	61	<0.003 <0.003	<0.003 <0.003	<0.004 <0.004	<0.01 <0.003	NR
USA, 1997, 13501-05MS	EC		56		3	61	<0.003	<0.003	<0.004	<0.003	NR
USA, 1997, 13501-05MS	EC		56		4	61	<0.003	<0.003	<0.004	<0.003	NR
USA, 1997, 13501-06AR	EC		56		3	57	<0.003	<0.003	<0.004	<0.01	NR
USA, 1997, 13501-06AR	EC		56		4	57	<0.003 <0.003	<0.01 <0.003	<0.004 <0.004	<0.01 <0.003	NR

NR: not reported inc: soil incorporation

Table 74. Residues in sunflower seed from seed treatment, in-furrow granular or pre-sowing incorporated spray application (each trial includes 2 replicates). All single applications.

Country, Year, Reference	Application			PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Seed treatment (Keats, 1998l)									
Australia, 1998, AK 98032, 96i38gR	FS	750 1500		113 113	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
Seed treatment (Keats, 1998k)									
Australia, 1998, AK 98035, 96i38bR	FS	750 1500		144 144	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
Seed treatment (Keats, 1998i)									
Australia, 1998, AK 98033, 97i004a	FS	750 1500		138 138	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
Seed treatment (Keats, 1998j)									
Australia, 1998, AK 98034, 97i004b	FS	750 1500		112 112	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
Soil application at sowing (Diot and Muller, 1992)									

Country, Year, Reference	Application			PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
France, 1990, 91-214, LA19I27 Leovillle	GR		200	147	<0.01	<0.01	<0.01	NR	<0.01
France, 1990, 91-214, LA19I27 Chadenac	GR		200	151	<0.01	<0.01	<0.01	NR	<0.01
France, 1990, 91-214, LA19I27 Castlenaudary	GR		200	149	<0.01	<0.01	<0.01	NR	<0.01
France, 1990, 91-214, LA19I27 St. Gilles	GR		200	140	<0.01	<0.01	<0.01	NR	<0.01
Soil application at sowing (Muller, 1993a)									
France, 1992, 92-142 part 1, XA192R84	GR		200 294	146 146	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	NR	<0.01 <0.01
France, 1992, 92-142 part 1, XH192R84	GR		206 295	137 137	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	NR	<0.01 <0.01
France, 1992, 92-142 part 1, XK192R84	GR		200 300	140 140	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	NR	<0.01 <0.01
Soil application at sowing (Muller, 1993b)									
France, 1992, 92-143 XK192R85	GR		206	136	<0.01	<0.01	<0.01	NR	<0.01
Seed treatment (Muller, 1993c)									
France, 1992, 92-178 XH192R112	FS	10 000 15 000		124 124	<0.01 0.014 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	NR	<0.01 <0.01 <0.01
France, 1992 92-178 XK192R112	FS	10 000 15 000		140 140	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	NR	<0.01 <0.01
France, 1992, 92-178 XE192R112	FS	10 000 15 000		148 148	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	NR	<0.01 <0.01
Seed treatment (Muller, 1994g)									
France, 1993, R93566H1	FS	2500		124	<0.002	<0.002	<0.002	NR	<0.002
Seed treatment (Muller, 1994h)									
France, 1993, R93567A1	FS	3750		146	<0.002 <0.002	<0.002 <0.002	0.002 <0.002	NR	<0.002 <0.002
France, 1993, R93567A2	FS	3750		147	<0.002 <0.002	<0.002 0.004	0.003 <0.002	NR	<0.002 <0.002
France, 1993, R93567E1	FS	3750		165	<0.002 <0.002	<0.002 0.002	<0.002 0.003	NR	<0.002 <0.002
France, 1993, R93567F1	FS	3750		148	<0.002	<0.002	<0.002	NR	<0.002
France, 1993, R93567H1	FS	3750		166	<0.002	<0.002	<0.002	NR	<0.002
France, 1993, R93567K1	FS	3750	1	89	<0.002	<0.002	<0.002	NR	<0.002
Soil application at sowing (Muller, 1996g)									
France, 1995, 95536OR1	GR		252	153	<0.002	<0.002	<0.002	<0.002	<0.002
France, 1995, 95536AV1	GR		174	139	<0.002	<0.002	<0.002	<0.002	<0.002
Soil application at sowing (Maestracci, 1996a)									
France, 1995, 95537DJ1	GR		190	154	<0.002	<0.002	<0.002	<0.002	<0.002
France, 1995, 95537TL1	GR		187	164	<0.002	<0.002	<0.002	<0.002	<0.002
Soil application before sowing (Maestracci, 1998c)									
France, 1997, 97542DJ1	WG		200	160	<0.002	<0.002	<0.002	<0.002	<0.002
France, 1997, 97542OR1	WG		200	146	<0.002	<0.002	<0.002	<0.002	<0.002
France, 1997, 97542BX1	WG		200	146	<0.002	<0.002	<0.002	<0.002	<0.002
France, 1997, 97542LY1	WG		200	134	<0.002	<0.002	<0.002	<0.002	<0.002
France, 1997, 97542TL1	WG		200	140	<0.002	<0.002	<0.002	<0.002	<0.002
Soil application before sowing (Richard and Muller, 1994f)									
Italy, 1993, R93635BO1	GR		240	112	<0.002	<0.002	<0.002	NR	<0.002
Seed treatment (Muller, 1995f)									
Spain, 1994, 94667SE1	FS	10 000		133	<0.002	<0.002	<0.002	NR	<0.002
Seed treatment (Maestracci, 1997b)									
Spain, 1996, 96637M1	FS	10 000		179	<0.002	<0.002	<0.002	<0.002	<0.002
Spain, 1996, 96637SE1	FS	10 000		161	<0.002	<0.002	<0.002	<0.002	<0.002

NR: not reported

Table 75. Supervised trials on sugar beet, leaves and tops analysed (each trial 2 replicates). All single applications.

Country, Year, Location, Reference	Application		PHI days	Residues, mg/kg				
	Form	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Soil treatment at sowing (Claviere and Muller, 1990)								
France, 1990	GR	100	148	<0.01	<0.01	0.011	NR	<0.01
Rachecourt,				0.015	<0.01	0.013		<0.01
XE190I11		200	148	0.027	<0.01	<0.01		<0.01
				0.029	<0.01	0.012		<0.01
		300	148	0.028	<0.01	<0.01		<0.01
				0.029	<0.01	0.015		<0.01
France, 1990	GR	100	179	0.021	<0.01	<0.01	NR	<0.01
Allogny,				0.013	<0.01	<0.01		<0.01
XB290I11		200	179	<0.01	<0.01	0.012		<0.01
				<0.01	<0.01	0.013		<0.01
		300	179	<0.01	<0.01	0.011		<0.01
				0.012	<0.01	0.013		<0.01
France, 1990	GR	100	181	<0.01	<0.01	<0.01	NR	<0.01
Beaulieu,				<0.01	<0.01	<0.01		<0.01
XB190I11		200	181	<0.01	<0.01	<0.01		<0.01
				<0.01	<0.01	<0.01		<0.01
		300	181	<0.01	<0.01	0.011		<0.01
				0.013	<0.01	0.012		<0.01
France, 1990	GR	100	168	0.013	<0.01	<0.01	NR	<0.01
Le Meillard,				0.018	<0.01	<0.01		<0.01
XD290I11		200	168	0.017	<0.01	<0.01		<0.01
				0.021	<0.01	0.012		<0.01
		300	168	0.011	<0.01	0.011		<0.01
				0.017	<0.01	0.012		<0.01
France, 1990	GR	100	176	<0.01	<0.01	0.012	NR	<0.01
Mericourt,				<0.01	<0.01	0.016		<0.01
XD190I11		200	176	<0.01	<0.01	0.011		<0.01
				0.012	<0.01	0.018		<0.01
		300	176	0.01	<0.01	0.012		<0.01
				0.014	<0.01	0.017		<0.01
Soil treatment at sowing (Dupont and Muller, 1992)								
France, 1991	GR	150	178	<0.01	<0.01	<0.01	NR	<0.01
Faverolles, LE291I15				<0.01	<0.01	<0.01		<0.01
		200		<0.01	<0.01	<0.01		<0.01
				<0.01	<0.01	<0.01		<0.01
France, 1991	GR	150	181	<0.01	<0.01	<0.01	NR	<0.01
Guignonville,				0.014	<0.01	<0.01		<0.01
LB391I15		200	181	<0.01	<0.01	<0.01		<0.01
				0.011	<0.01	<0.01		<0.01
France, 1991	GR	150	161	<0.01	<0.01	<0.01	NR	<0.01
Autruche, LE191I15				<0.01	<0.01	<0.01		<0.01
		200	161	<0.01	<0.01	<0.01		<0.01
				<0.01	<0.01	<0.01		<0.01
France, 1991	GR	150	198	0.012	<0.01	<0.01	NR	<0.01
Bellegarde, LB191I15				0.015	<0.01	<0.01		<0.01
		200	198	<0.01	<0.01	<0.01		<0.01
				0.017	<0.01	<0.01		<0.01

Country, Year, Location, Reference	Application		PHI	Residues, mg/kg				
	Form	g ai/ha	days	Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
France, 1991 Meranville, LB491I15	GR	150	213	<0.01	<0.01	<0.01	NR	<0.01
				<0.01	<0.01	<0.01		<0.01
		200	213	<0.01	<0.01	<0.01		0.016
				<0.01	<0.01	<0.01		0.012
Soil treatment at sowing (Muller, 1994j)								
France, 1993 R93568B1 Gidy	GR	305	193	<0.01	<0.01	<0.01	NR	<0.01
				<0.01	<0.01	<0.01		<0.01
France, 1993 R93568B2 Patay	GR	334	196	<0.01	<0.01	<0.01	NR	<0.01
				<0.01	<0.01	<0.01		<0.01
France, 1993 R93568D1 Frohen le Grand	GR	251	181	<0.01	<0.01	<0.01	NR	<0.01
				<0.01	<0.01	<0.01		<0.01
France, 1993 R93568D2 Frohen le Grand	GR	281	190	<0.01	<0.01	<0.01	NR	<0.01
				<0.01	<0.01	<0.01		<0.01
France, 1993 R93568D3 Frohen le Grand	GR	211	175	<0.01	<0.01	<0.01	NR	<0.01
				<0.01	<0.01	<0.01		<0.01
France, 1993 R93568E1 Bazancourt	GR	253	166	<0.01	<0.01	<0.01	NR	<0.01
				<0.01	<0.01	<0.01		<0.01
Soil treatment at sowing (Muller, 1995e)								
France, 1994 945520R1 Patay	GR	245	189	<0.01	<0.01	<0.01	NR	<0.01
				<0.01	<0.01	<0.01		<0.01
France, 1994 94552AM1 Barly	GR	240	153	<0.01	<0.01	<0.01	NR	<0.01
				<0.01	<0.01	<0.01		<0.01
France, 1994 94552RS1 Muizon	GR	244	174	<0.01	<0.01	<0.01	NR	<0.01
				<0.01	<0.01	<0.01		<0.01
Soil treatment before sowing (Maestracci, 1998b)								
France, 1997 97521AM1 Barly	WG	200	168	<0.002	<0.002	<0.002	<0.002	<0.002
				<0.002	<0.002	<0.002	<0.002	<0.002
France, 1997 97521DJ1 Neuilly les Dijon	WG	210	188	<0.002	<0.002	<0.002	<0.002	<0.002
				<0.002	<0.002	<0.002	<0.002	<0.002
France, 1997 97521OR1 Merevile	WG	200	181	<0.002	<0.002	0.006	<0.002	<0.002
				<0.002	<0.002	0.005	<0.002	0.004
France, 1997 97521RS1 Reims	WG	200	186	<0.002	<0.002	0.003	<0.002	<0.002
				<0.002	<0.002	0.002	<0.002	<0.002
Soil treatment at sowing (Richard and Muller, 1994b)								
Italy, 1993 93640BO1 CNS Bologna	GR	210	169	<0.01	<0.01	<0.01	NR	<0.01
				<0.01	<0.01	<0.01		<0.01
Italy, 1993 93630BO1 CNS Bologna	GR	150	169	<0.01	<0.01	<0.01	NR	<0.01
				0.018	<0.01	0.01		<0.01
		300		<0.01	<0.01	<0.01		<0.01
				<0.01	<0.01	<0.01		<0.01
Soil treatment in furrow at sowing (Muller, 1996e)								
Italy, 1995 95742BO1 CNS Corticella	GR	166	140	<0.01	<0.01	<0.01	<0.01	<0.01
				<0.01	<0.01	<0.01	<0.01	<0.01
Italy, 1995 95742BO2 Minerbio	GR	146	136	<0.01	<0.01	<0.01	<0.01	<0.01
				<0.01	<0.01	<0.01	<0.01	<0.01
Soil treatment (Muller, 1996l)								
Italy, 1995 95741BO1 CNS Corticella	GR	90	140	<0.01	<0.01	<0.01	<0.01	<0.01
		141	140	<0.01	<0.01	<0.01	<0.01	<0.01
Italy, 1995 95741BO2 Minerbio	GR	106	136	<0.01	<0.01	<0.01	<0.01	<0.01
		140	136	<0.01	<0.01	<0.01	<0.01	<0.01

Country, Year, Location, Reference	Application		PHI days	Residues, mg/kg				
	Form	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Soil treatment (Yslan and Baudet, 1999c)								
Spain, 1998 98589M1 Sta. Olalla Toledo	WG	197 1	197	<0.002 <0.002	<0.002 <0.002	0.002 <0.002	<0.002 <0.002	NR

NR: not reported

Table 76. Residues in maize forage, fodder, and green plant (forage) for use as silage. All single applications.

Country, Year, Reference	Sample	Application				PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha	g ai/hl		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Seed treatment, 2 replicates (Muller, 1994c)											
France, 1993, R9356A1	silage green plant	FS	2500			118 60	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	NR NR	<0.01 <0.01
Seed treatment, 2 replicates (Muller, 1994e)											
France, 1993, R93564A1	silage	FS	3750			118	<0.005	<0.005	<0.005	NR	<0.005
France, 1993, R93564B1	silage	FS	3750			118	<0.005	<0.005	<0.005	NR	<0.005
France, 1993, R93564C1	silage	FS	3750			118	<0.005	<0.005	<0.005	NR	<0.005
France, 1993, R93564E	silage	FS	3750			118	<0.005	<0.005	<0.005	NR	<0.005
France, 1993, R93564F1	silage	FS	3750			118	<0.005	<0.005	<0.005	NR	<0.005
France, 1993, R93564K1	silage	FS	3750			118	<0.005	<0.005	<0.005	NR	<0.005
Soil treatment incorporated granules in furrow, 2 replicates (Muller, 1996i)											
France, 1995, 95535BX1	silage	GR		298		116	<0.005	<0.005	<0.005	<0.005	<0.01
France, 1995, 95535AM1	silage	GR		177		112	<0.005	<0.005	<0.005	<0.005	<0.005
Soil treatment pre-sowing (Maestracci, 1998d)											
France, 1997, 97540AM1	green plant	WG		200	66.7	159*	<0.005	<0.005	<0.005	<0.005	NR
France, 1997, 97540OR1	green plant	WG		200	60.6	141*	<0.005	<0.005	<0.005	<0.005	NR
France, 1997, 97540RS1	green plant	WG		200	84.7	139*	<0.005	<0.005	<0.005	<0.005	NR
France, 1997, 97540RS2	green plant	WG		200	100	141*	<0.005	<0.005	<0.005	<0.005	NR
France, 1997, 97540BX1	green plant cob	WG		200	60.6	132* 118	<0.005 <0.002	<0.005 <0.002	<0.005 <0.002	<0.005 <0.002	NR <0.002
France, 1997, 97540BX2	green plant cob	WG		200	60.6	132* 118	<0.005 <0.002	<0.005 <0.002	<0.005 <0.002	<0.005 <0.002	NR <0.002
France, 1997, 97540TL1	green plant cob	WG		200	60.6	127* 105	<0.005 <0.002 <0.002	<0.005 <0.002 0.003	<0.005 <0.002 <0.002	<0.005 <0.002 <0.002	NR <0.002 <0.002
France, 1997, 97540TL2	green plant cob	WG		200	60.6	132* 110	<0.005 <0.002 0.003	<0.005 0.003 0.005	<0.005 <0.002 <0.002	<0.005 <0.002 <0.002	NR <0.002 <0.002
Soil treatment at sowing (Muller, 1995d)											
Greece, 1994, 94672GR1	silage	GR		200		77	<0.002	<0.002	<0.002	NR	<0.002
Greece, 1994, 94672GR1	silage	GR		400		77	<0.002	<0.002	<0.002	NR	<0.002
Soil treatment at sowing (Richard and Muller, 1994g)											
Italy, 1993, R93633BO1	shoot	GR		240		122	<0.01	<0.01	<0.01	NR	0.01

Country, Year, Reference	Sample	Application				PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha	g ai/hl		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Soil treatment at sowing, 2 replicates (Muller, 1996c)											
Italy, 1995, 95744BO1	shoot	GR		153		115	<0.005	<0.005	<0.005	<0.005	<0.005
Italy, 1995, 95744BO2	shoot	GR		155		100	<0.005	<0.005	<0.005	<0.005	<0.005
Soil treatment at sowing, 3 replicates (Kowite, 1993a)											
USA, 1992, 92-015 INC	forage	GR		146		45	<0.02 <0.001 (2)	<0.003 <0.003 (2)	<0.005 <0.005 (2)	<0.004 <0.004 (2)	<0.02 <0.003 (2)
	silage					110	<0.007 <0.007 <0.007	<0.004 <0.004 <0.004	<0.02 <0.005 <0.005	<0.003 <0.003 <0.003	<0.02 <0.004 <0.02
	fodder					146	<0.02 <0.003	<0.007 <0.007	<0.02 <0.02	<0.004 <0.004	<0.02 <0.006
USA, 1992, 92-015 T-band	forage	GR		146		45	<0.001 (2) <0.02	<0.003 (2) <0.003	<0.005 (2) <0.005	<0.004 (2) <0.004	<0.003 (2) <0.003
	silage					110	<0.007	<0.004	<0.005	<0.003	<0.004
	fodder					146	<0.003 <0.02 <0.02	<0.007 <0.007 <0.007	<0.02 0.022 <0.02	<0.004 <0.004 <0.004	<0.02 <0.02 <0.02
USA, 1992, 92-016	forage	GR		146		45	<0.001(2) <0.001	<0.003 (2) <0.003	<0.005 (2) <0.005	<0.004 (2) <0.004	<0.003 (2) <0.02
	silage					109	<0.007 <0.007 <0.007	<0.004 <0.004 <0.004	<0.02 <0.02 <0.005	<0.003 <0.003 <0.003	<0.02 <0.02 <0.004
	fodder					157	<0.02 <0.02 <0.02	<0.007 <0.007 <0.007	0.031 0.022 0.033	<0.02 <0.02 <0.02	<0.02 <0.02 <0.02
USA, 1992, 92-019 INC	forage	GR		146		45	<0.001	<0.003	<0.005	<0.004	<0.003
	silage					137	<0.007 (2) <0.007	<0.004 (2) <0.004	<0.005(2) 0.005	<0.003 (2) <0.02	<0.004 <0.004
	fodder					191	<0.003 (2) <0.003	<0.007 (2) <0.007	<0.02 (2) <0.005	<0.004 (2) <0.004	2<0.006 <0.006
USA, 1992, 92-019 T-band	forage	GR		146		45	<0.02 (2) <0.02	<0.003 (2) <0.003	<0.005 (2) <0.005	<0.004 (2) <0.004	2<0.003 <0.02
	silage					137	<0.007 <0.007 <0.02	<0.004 <0.004 <0.02	<0.005 <0.005 <0.02	<0.003 <0.02 <0.02	<0.004 <0.004 <0.004
	fodder					191	<0.02	<0.007	<0.02	<0.004	<0.006
USA, 1992, 92-029	forage	GR		157		45	<0.02 (2) <0.02	<0.003 (2) <0.003	<0.005 (2) <0.005	<0.004 (2) <0.004	<0.003 (2) <0.02
	silage					117	<0.007	<0.004	<0.005	<0.003	<0.004
	fodder					153	<0.02	<0.007	<0.02	<0.004	<0.006
USA, 1992, 92-039	forage	GR		135		45	<0.001	<0.003	<0.005	<0.004	<0.003
	silage					102	<0.007	<0.004	<0.005	<0.003	<0.004
	fodder					156	<0.003 (2) <0.003	<0.02 (2) <0.007	<0.005 (2) <0.005	<0.004 (2) <0.004	<0.006 (2) <0.006
USA, 1992, 92-058 INC	forage	GR		157		45	<0.02	<0.003	<0.005	<0.004	<0.003
	silage					129	<0.007	<0.004	<0.005	<0.003	<0.004
	fodder					159	<0.003	<0.007	<0.02	<0.004	<0.006

Country, Year, Reference	Sample	Application				PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha	g ai/hl		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
USA, 1992, 92-058 T-band	forage	GR		157		45	<0.02	<0.003	<0.005	<0.004	<0.003
	silage					129	<0.007 (2) <0.007	<0.004 (2) 0.004	<0.005 (2) <0.005	<0.003 (2) <0.003	<0.004 (2) <0.02
	fodder					159	<0.003 <0.02 <0.003	<0.007 <0.007 <0.007	<0.02 <0.02 <0.005	<0.004 <0.004 <0.004	<0.006 <0.006 <0.02
USA, 1992, 92-076	forage	GR		135		45	<0.02	<0.003	<0.02	<0.004	<0.02
	silage					115	<0.007 (2) <0.02	<0.004 (2) <0.004	<0.02 (2) <0.02	2<0.003 <0.003	<0.02 (2) <0.02
	fodder					151	<0.02 <0.02 <0.02	<0.007 <0.007 <0.007	<0.02 0.024 0.021	<0.02 <0.004 <0.02	<0.02 <0.02 <0.02
USA, 1992, 92-094	forage	GR		146		45	<0.02	<0.003	<0.02	<0.004	<0.02
	silage					121	<0.007 (2) <0.007	<0.004 (2) <0.004	<0.02 (2) <0.005	2<0.003 <0.003	<0.004 (2) <0.004
	fodder					173	<0.02 <0.02 <0.02	<0.02 <0.007 <0.007	0.029 0.042 0.03	<0.004 <0.004 <0.004	<0.02 <0.02 <0.02
USA, 1992, 92-097	forage	GR		146		46	<0.02	<0.003	<0.005	<0.004	<0.02
	silage					130	<0.007	<0.004	<0.005	<0.02	<0.02
	fodder					162	<0.02	<0.007	<0.02	<0.004	<0.02
USA, 1992, 92-101 INC	forage	GR		146		71	<0.02 (2) <0.02	<0.003 (2) <0.003	<0.005 (2) <0.005	<0.004 (2) <0.004	<0.02 (2) <0.003
	silage					126	<0.02	<0.004	<0.02	<0.003	<0.02
	fodder					169	NR	NR	NR	NR	NR
USA, 1992, 92-101 T-band	forage	GR		146		71	<0.02	<0.003	<0.005	<0.004	<0.02
	silage					126	<0.007 (2) <0.02	<0.004 (2) 0.004	<0.02 (2) <0.02	2<0.003 <0.003	<0.02 (2) <0.02
	fodder					169	<0.02 <0.02 <0.02	<0.007 <0.007 <0.007	0.029 0.034 0.038	<0.02 <0.02 <0.02	<0.02 0.02 <0.02
Soil treatment at sowing, 3 replicates (Kowite, 1994)											
USA, 1993, 93-206 T-band	forage	GR		146		45	<0.001	<0.003	<0.005	NR	<0.003
	silage					113	<0.02	<0.004	<0.02		<0.02
	fodder					148	<0.02 <0.02 <0.02	<0.007 <0.02 <0.02	0.022 0.031 0.027		<0.02 <0.02 <0.02
USA, 1993, 93-206 INC	forage	GR		146		45	<0.02 (2) <0.02	<0.003 (2) <0.003	<0.02 (2) <0.02	NR	2<0.003 <0.02
	silage					113	<0.02	<0.004	<0.02		<0.02
	fodder					148	<0.02 <0.02 0.02	<0.007 <0.02 <0.007	0.047 0.045 0.073		0.025 0.024 0.03
USA, 1993. 93-207	forage	GR		146		45	<0.001	<0.003	<0.005	NR	<0.003
	silage					103	<0.02 <0.007 (2)	<0.004 <0.004 (2)	<0.02 <0.005 (2)		<0.02 <0.02 (2)
	fodder					140	<0.02 <0.02 <0.02	<0.02 <0.007 <0.02	<0.02 <0.02 <0.02		<0.02 <0.02 <0.006

Country, Year, Reference	Sample	Application				PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha	g ai/hl		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
USA, 1993, 93-208	forage	GR		146		44	<0.02 <0.02 <0.02	<0.02 <0.003 <0.02	<0.005 <0.005 <0.005	NR	<0.003 <0.02 <0.003
	silage					96	0.022 <0.02 <0.02	≤0.004 <0.004 <0.004	≤0.02 <0.02 0.021		<0.02 <0.02 <0.02
	fodder					125	≤0.02	≤0.007	≤0.02		<0.02
USA, 1993, 93-209	silage	GR		146		96	≤0.007 <0.007 <0.007	≤0.004 <0.004 <0.004	≤0.005 <0.005 <0.005	NR	<0.02 <0.02 <0.004
	fodder					168	≤0.003	≤0.02	≤0.02		<0.006
USA, 1993, 93-210	forage	GR		146		45	<0.02	<0.003	<0.005	NR	<0.003
	silage					106	≤0.007	≤0.004	≤0.005		<0.004
	fodder					128	≤0.02	≤0.02	≤0.02		<0.02
USA, 1993, 93-211	forage	GR		146		45	0.056 0.03 0.03	<0.02 <0.003 <0.003	0.044 0.024 0.027	NR	0.031 0.022 <0.02
	silage					109	≤0.02 <0.02 <0.02	≤0.004 <0.004 <0.004	0.036 <0.02 0.024		<0.02 <0.02 <0.02
	fodder					124	≤0.02 <0.02 <0.02	≤0.007 <0.007 <0.007	0.025 0.022 <0.02		<0.02 <0.02 <0.02
USA, 1993, 93-212 T-band	forage	GR		146		45	<0.02	<0.003	<0.005	NR	<0.003
	silage					115	≤0.007	≤0.004	≤0.005		<0.004
	fodder					171	≤0.003	≤0.007	≤0.005		<0.006
USA, 1993, 93-212 INC	forage	GR		146		45	<0.02	<0.002	<0.005	NR	<0.003
	silage					115	<0.007 (2) ≤0.007	<0.004 (2) ≤0.004	<0.005 (2) ≤0.02		<0.02 (2) <0.02
	fodder					171	≤0.02 (2) <0.02	≤0.007 (2) <0.007	≤0.02 (2) <0.02		<0.02 (2) <0.006
USA, 1993, 93-213 T-band	forage	GR		146		45	<0.02 (2) 0.021	2<0.003 <0.003	<0.02 (2) <0.02	NR	<0.02 (2) <0.02
	silage					129	<0.007 (2) ≤0.007	<0.004 (2) ≤0.02	<0.005 (2) ≤0.005		<0.004 (2) <0.004
	fodder					171	<0.02 (2) 0.02	<0.02 (2) 0.007	<0.02 (2) 0.02		2<0.006 0.006
USA, 1993, 93-213 INC	forage	GR		146		45	0.026 0.025 0.028	<0.003 <0.003 <0.003	<0.02 <0.02 <0.02	NR	<0.02 <0.02 <0.02
	silage					129	<0.007 (2) ≤0.007	<0.004 (2) ≤0.004	<0.005 (2) ≤0.005		<0.004 (2) <0.02
	fodder					171	≤0.02	≤0.02	≤0.02		<0.006
USA, 1993, 93-214 T-band	forage	GR		146		47	<0.02 (2) <0.02	2<0.003 <0.003	<0.02 (2) <0.005	NR	<0.02 (2) <0.02
	silage					103	<0.007 <0.02 ≤0.02	<0.004 <0.004 ≤0.004	<0.005 <0.02 ≤0.02		<0.02 <0.02 <0.02
	fodder					131	<0.02 ≤0.02 <0.003	<0.007 ≤0.007 <0.007	<0.02 ≤0.02 <0.02		<0.006 <0.02 <0.006

Country, Year, Reference	Sample	Application				PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha	g ai/hl		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
USA, 1993, 93-214 INC	forage	GR		146		47	0.024 <0.02 0.022	<0.003 <0.003 <0.003	<0.02 <0.02 <0.02	NR	<0.02 <0.02 <0.02
	silage					103	<0.007 (2) <0.02	<0.004 (2) <0.004	<0.005 (2) <0.02		<0.004 (2) <0.02
	fodder					131	<0.02 (2) <0.02	<0.007 (2) <0.007	<0.02 (2) <0.02		2<0.006 <0.02
USA, 1993, 93-215 T-band	forage	GR		146		45	<0.02 (2) <0.02	2<0.003 <0.003	<0.005 (2) <0.005	NR	<0.02 (2) <0.003
	silage					111	<0.007 (2) <0.007	<0.004 (2) <0.004	<0.005 (2) <0.005		<0.004 (2) <0.02
	fodder					140	<0.02	<0.007	<0.02		<0.006
USA, 1993, 93-215 INC	forage	GR		146		45	<0.02 <0.001 <0.02	<0.003 <0.003 <0.003	<0.005 <0.005 <0.005	NR	<0.003 <0.003 <0.02
	silage					111	<0.007 <0.007 <0.007	<0.004 <0.004 <0.004	<0.02 <0.02 <0.005		<0.02 <0.02 <0.02
	fodder					140	<0.02	<0.007	<0.02		<0.02
Soil treatment at sowing, 2 replicates (Kowite, 1996)											
USA, 1995, 95-0212 GR INC	forage	GR		146		88	<0.02	<0.02	<0.02	NR	<0.02
	fodder					126	0.04 0.032	<0.02 <0.02	0.055 0.051		0.028 0.024
USA, 1995, 95-0212 SS INC	forage	WG		146		88	0.023 <0.02	<0.02 <0.02	0.03 <0.02	NR	<0.02 <0.02
	fodder					126	0.022 0.025	<0.02 <0.02	0.04 0.043		0.02 0.02
USA, 1995, 95-0213 GR INC	forage	GR		146		105	<0.02	<0.004	<0.02	NR	<0.02
	fodder					148	<0.02 <0.02	<0.003 <0.02	<0.02 <0.02		<0.02 <0.02
USA, 1995, 95-0213 SS INC	forage	WG		146		105	<0.005 <0.02	<0.004 <0.004	<0.02 <0.02	NR	<0.02 <0.02
	fodder					148	<0.02	<0.003	<0.02		<0.02
USA, 1995, 95-0214 GR INC	forage	GR		146		105	<0.02	<0.004	<0.02	NR	<0.02
	fodder					153	<0.02 <0.02	<0.003 <0.003	0.034 0.024		<0.02 <0.02
USA, 1995, 95-0214 SS INC	forage	WG		146		105	<0.02	<0.004	<0.02	NR	<0.02
	fodder					153	<0.02 <0.02	<0.003 <0.003	0.021 <0.02		<0.02 <0.02
USA, 1995, 95-0215 GR INC	forage	GR		146		120	<0.02 <0.005	<0.02 <0.004	<0.02 <0.02	NR	<0.02 <0.02
	fodder					159	<0.02 <0.02	<0.003 <0.003	<0.02 <0.008		<0.008 <0.008
USA, 1995, 95-0215 SS INC	forage	WG		146		120	<0.02	<0.004	<0.02	NR	<0.02
	fodder					159	<0.02	<0.003	<0.02		<0.008
USA, 1995, 95-0216 GR INC	forage	GR		146		88	<0.02	<0.004	<0.02	NR	<0.02
	fodder					136	<0.02 <0.02	<0.02 <0.02	0.023 0.025		<0.02 <0.02
USA, 1995, 95-0216 SS INC	forage	WG		146		88	<0.02	<0.004	<0.02	NR	<0.02
	fodder					136	<0.02	<0.02	0.021		<0.02

Country, Year, Reference	Sample	Application				PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha	g ai/hl		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
USA, 1995, 95-0217 GR INC	forage	GR		146		84	<0.02	<0.004	<0.02	NR	<0.02
	fodder					122	<0.02 <0.02	<0.003 <0.003	0.036 0.03		<0.02 <0.02
USA, 1995, 95-0217 SS INC	forage	WG		146		84	<0.02 <0.02	<0.02 <0.02	0.022 <0.02	NR	<0.02 <0.02
	fodder					122	<0.02 <0.02	<0.003 <0.003	0.034 0.051		<0.02 <0.02
USA, 1995, 95-0218 GR INC	forage	GR		146		84	<0.02 <0.02	<0.004 <0.004	0.022 0.029	NR	<0.02 <0.02
	fodder					122	<0.02 <0.02	<0.003 <0.003	0.038 0.048		<0.02 0.03
USA, 1995, 95-0218 SS INC	forage	WG		146		84	<0.02 <0.02	<0.004 <0.004	0.026 0.024	NR	<0.02 <0.02
	fodder					122	<0.02 <0.02	<0.02 <0.02	0.046 0.044		0.024 0.021
USA, 1995, 95-0219 GR INC	forage	GR		146		94	<0.02	<0.02	<0.02	NR	<0.02
	fodder					116	<0.02	<0.003	0.025		<0.02
USA, 1995, 95-0219 SS INC	forage	WG		146		94	<0.02	<0.02	<0.02	NR	<0.02
	fodder					116	<0.02	<0.003	<0.02		<0.02
USA, 1995, 95-0220 GR INC	forage	GR		146		101	<0.02	<0.004	<0.02	NR	<0.02
	fodder					143	<0.02 <0.02	<0.02 <0.02	0.025 0.024		<0.02 <0.02
USA, 1995, 95-0220 SS INC	forage	WG		146		101	<0.02	<0.004	<0.02	NR	<0.02
	fodder					143	0.023 0.022	<0.02 <0.02	0.039 0.041		<0.02 <0.02
USA, 1995, 95-0221 GR INC	forage	GR		146		105	<0.005 <0.005	<0.004 <0.004	<0.02 <0.02	NR	<0.003 <0.02
	fodder					160	<0.02	<0.003	<0.02		<0.02
USA, 1995, 95-0221 SS INC	forage	WG		146		105	<0.005	<0.004	<0.02	NR	<0.003
	fodder					160	<0.02	<0.003	<0.02		<0.02
USA, 1995, 95-0222 GR INC	forage	GR		146		103	<0.02	<0.004	<0.02	NR	<0.02
	fodder					150	<0.02 <0.02	<0.003 <0.003	0.038 <0.02		<0.02 <0.008
USA, 1995, 95-0222 SS INC	forage	WG		146		103	<0.02	<0.004	<0.02	NR	<0.02
	fodder					150	<0.005	<0.003	<0.02		<0.008
USA, 1995, 95-0223 GR INC	forage	GR		146		92	0.022 <0.02	<0.02 <0.004	0.028 0.022	NR	<0.02 <0.02
	fodder					125	<0.02 <0.02	<0.02 <0.02	0.044 0.054		<0.02 <0.02
USA, 1995, 95-0223 SS INC	forage	WG		146		92	0.038 0.029	<0.02 <0.004	0.043 0.032	NR	<0.02 <0.02
	fodder					125	0.032 0.04	<0.02 <0.02	0.098 0.106		0.028 0.029
Seed treatment, 2 replicates (Richard and Muller, 1995d)											
Spain, 1994, 94665SE1	fodder	FS	2500			174	<0.002	<0.002	<0.002	NR	<0.002
Seed treatment, 2 replicates (Maestracci, 1996b)											
Spain, 1994, 95712SE1	fodder	FS	2500			155	<0.005	<0.005	<0.005	<0.005	<0.005
Spain, 1994, 95712SE2	fodder	FS	2500			153	<0.005	<0.005	<0.005	<0.005	<0.005

NR: not reported

*days between sowing and harvest, not between soil spray and harvest

Table 77. Residues in pasture grass after single foliar applications (each trial includes 2 replicates).

Country, Year, Reference	Application		PHI, days	Residues, mg/kg				
	Form	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Keats, 1996 f								
Australia, 1995, 94i55 Clermont Central Queensland	UL	5	0	0.051	<0.002	0.005	<0.002	NR
				0.051	<0.002	0.005	0.002	
			1	0.029	<0.002	0.004	<0.002	
				0.029	<0.002	0.005	<0.002	
			3	0.01	<0.002	0.006	0.002	
				0.011	<0.002	0.006	0.002	
			5	0.009	<0.002	0.007	0.003	
				0.009	<0.002	0.007	0.004	
			7	0.003	<0.002	0.003	<0.002	
				0.005	<0.002	0.003	<0.002	
Australia, 1995, 94i55 Clermont Central Queensland	UL	10	0	0.13	<0.002	0.013	0.003	NR
				0.14	<0.002	0.013	0.003	
			1	0.082	<0.002	0.01	0.003	
				0.071	<0.002	0.012	0.003	
			3	0.017	<0.002	0.01	0.005	
				0.019	<0.002	0.012	0.005	
			5	0.021	<0.002	0.016	0.007	
				0.017	<0.002	0.016	0.008	
			7	0.01	<0.002	0.01	0.004	
				0.011	<0.002	0.01	0.004	
Australia, 1995, 94i55 Clermont Central Queensland	UL	20	0	0.25	0.002	0.029	0.025	NR
				0.17	<0.002	0.029	0.025	
			1	0.13	<0.002	0.022	0.02	
				0.12	<0.002	0.023	0.019	
			3	0.04	<0.002	0.019	0.015	
				0.041	<0.002	0.019	0.015	
			5	0.023	<0.002	0.017	0.012	
				0.021	<0.002	0.017	0.013	
			7	0.011	<0.002	0.008	0.007	
				0.018	<0.002	0.008	0.008	
Australia, 1995, 94i55 Clermont Central Queensland	UL	30	0	0.52	0.003	0.045	0.042	NR
				0.52	0.003	0.041	0.043	
			1	0.29	0.003	0.04	0.036	
				0.29	0.003	0.034	0.037	
			3	0.17	0.002	0.047	0.048	
				0.18	0.003	0.045	0.048	
			5	0.08	<0.002	0.036	0.034	
				0.083	<0.002	0.036	0.035	
			7	0.029	<0.002	0.022	0.022	
				0.028	<0.002	0.023	0.023	
Keats, 1996 k								
Australia, 1995, AUS94i55b Springfield via Orange, NSW	WG	5	0	0.005	<0.002	<0.002	<0.002	NR
				0.005	<0.002	<0.002	<0.002	
			1	0.003	<0.002	<0.002	<0.002	
				0.003	<0.002	<0.002	<0.002	
			3	0.002	<0.002	<0.002	<0.002	
				<0.002	<0.002	<0.002	<0.002	
			5	0.002	<0.002	<0.002	<0.002	
				0.002	<0.002	<0.002	<0.002	
			7	<0.002	<0.002	<0.002	<0.002	
				<0.002	<0.002	<0.002	<0.002	

Country, Year, Reference	Application		PHI, days	Residues, mg/kg				
	Form	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Australia, 1995, AUS94i55b Springfield via Orange, NSW	WG	10	0	0.011	<0.002	<0.002	<0.002	NR
				0.011	<0.002	<0.002	0.002	
			1	0.006	<0.002	<0.002	<0.002	
				0.006	<0.002	<0.002	<0.002	
			3	0.004	<0.002	<0.002	<0.002	
				0.004	<0.002	<0.002	<0.002	
			5	0.003	<0.002	<0.002	<0.002	
				0.003	<0.002	<0.002	<0.002	
			7	0.003	<0.002	<0.002	<0.002	
				0.003	<0.002	<0.002	<0.002	
Australia, 1995, AUS94i55b Springfield via Orange, NSW	WG	20	0	0.021	<0.002	<0.002	0.004	NR
				0.02	<0.002	<0.002	0.004	
			1	0.014	<0.002	<0.002	0.003	
				0.014	<0.002	<0.002	0.003	
			3	0.009	<0.002	<0.002	0.003	
				0.009	<0.002	<0.002	0.002	
			5	0.005	<0.002	<0.002	0.002	
				0.005	<0.002	<0.002	0.002	
			7	0.006	<0.002	<0.002	<0.002	
				0.005	<0.002	<0.002	<0.002	
Australia, 1995, AUS94i55b Springfield via Orange, NSW	WG	30	0	0.032	<0.002	<0.002	0.005	NR
				0.032	<0.002	<0.002	0.004	
			1	0.02	<0.002	<0.002	0.005	
				0.02	<0.002	<0.002	0.005	
			3	0.014	<0.002	<0.002	0.003	
				0.014	<0.002	<0.002	0.004	
			5	0.007	<0.002	<0.002	0.003	
				0.007	<0.002	<0.002	0.003	
			7	0.008	<0.002	<0.002	0.002	
				0.009	<0.002	<0.002	0.002	
Keats, 1996 g								
Australia, 1995, AUS95i55R Binguy New South Wales	UL	5	1	0.11	<0.002	0.009	0.003	NR
				0.091	<0.002	0.009	0.003	
			3	0.03	<0.002	0.011	0.005	
				0.026	<0.002	0.011	0.005	
			5	0.021	<0.002	0.014	0.007	
				0.019	<0.002	0.014	0.007	
			7	0.007	0.006	0.007	<0.002	
				0.009	<0.002	0.007	0.003	
Australia, 1995, AUS95i55R Binguy New South Wales	UL	10	1	0.23	<0.002	0.021	0.005	NR
				0.2	<0.002	0.01	0.003	
			3	0.045	<0.002	0.01	0.006	
				0.039	<0.002	0.022	0.01	
			5	0.045	<0.002	0.033	0.016	
				0.042	<0.002	0.031	0.016	
			7	0.027	<0.002	0.02	0.008	
				0.029	<0.002	0.021	0.008	
Australia, 1995, AUS95i55R Binguy New South Wales	UL	15	1	0.42	0.003	0.044	0.034	NR
				0.38	0.003	0.038	0.027	
			3	0.14	0.004	0.033	0.021	
				0.13	0.004	0.025	0.022	
			5	0.066	<0.002	0.023	0.018	
				0.057	<0.002	0.031	0.019	
			7	0.028	<0.002	0.015	0.014	
				0.037	<0.002	0.016	0.014	

Country, Year, Reference	Application		PHI, days	Residues, mg/kg				
	Form	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Australia, 1995, AUS95i55R Binguy New South Wales	UL	30	1	0.78	0.004	0.06	0.054	NR
				0.71	0.004	0.053	0.051	
			3	0.44	0.004	0.07	0.077	
				0.44	0.004	0.067	0.067	
			5	0.22	0.002	0.056	0.044	
				0.21	0.002	0.046	0.022	
			7	0.075	<0.002	0.031	0.031	
				0.073	<0.002	0.031	0.03	
Keats, 1996 l								
Australia, 1995, AUS94i55cR Gingin Western Autralia	SC	5	0	0.18	0.005	0.004	0.042	NR
				0.18	0.004	0.005	0.044	
			1	0.067	0.005	0.005	0.05	
				0.065	0.005	0.005	0.05	
			3	0.032	0.008	0.004	0.026	
				0.031	0.008	0.004	0.026	
			5	0.004	0.004	<0.002	0.004	
				0.003	0.004	<0.002	0.004	
			7	0.003	0.004	<0.002	0.003	
				0.003	0.004	<0.002	0.004	
Australia, 1995, AUS94i55cR Gingin Western Australia	SC	10	0	0.36	0.014	0.017	0.15	NR
				0.36	0.015	0.018	0.15	
			1	0.27	0.014	0.01	0.13	
				0.27	0.014	0.011	0.14	
			3	0.17	0.015	0.017	0.15	
				0.18	0.015	0.018	0.16	
			5	0.072	0.018	0.01	0.058	
				0.069	0.017	0.008	0.056	
			7	0.005	0.005	<0.002	0.006	
				0.005	0.004	<0.002	0.006	
Australia, 1995, AUS94i55cR Gingin Western Autralia	SC	20	0	0.44	0.027	0.052	0.30	NR
				0.44	0.024	0.058	0.29	
			1	0.39	0.014	0.023	0.18	
				0.41	0.018	0.041	0.21	
			3	0.24	0.026	0.047	0.27	
				0.24	0.026	0.047	0.28	
			5	0.22	0.033	0.059	0.36	
				0.21	0.024	0.043	0.31	
			7	0.14	0.031	0.038	0.23	
				0.14	0.034	0.042	0.24	
Australia, 1995, AUS94i55cR Gingin Western Australia	SC	30	0	0.5	0.056	0.1	0.27	NR
				0.5	0.056	0.1	0.25	
			1	0.46	0.046	0.082	0.24	
				0.46	0.045	0.086	0.24	
			3	0.26	0.031	0.073	0.21	
				0.26	0.031	0.076	0.20	
			5	0.23	0.024	0.056	0.23	
				0.24	0.025	0.056	0.23	
			7	0.23	0.027	0.077	0.27	
				0.23	0.028	0.077	0.26	

Country, Year, Reference	Application		PHI, days	Residues, mg/kg				
	Form	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Keats, 1996 j								
Australia, 1996, AUS94i55d Springfield via Orange, NSW	SC	5	0	0.009	<0.002	<0.002	<0.002	NR
				0.009	<0.002	<0.002	<0.002	
			1	0.005	<0.002	<0.002	<0.002	
				0.005	<0.002	<0.002	<0.002	
			3	0.004	<0.002	<0.002	<0.002	
				0.004	<0.002	<0.002	<0.002	
			5	0.002	<0.002	<0.002	<0.002	
				0.002	<0.002	<0.002	<0.002	
			7	0.002	<0.002	<0.002	<0.002	
				0.002	<0.002	<0.002	<0.002	
Australia, 1996, AUS94i55d Springfield via Orange, NSW	SC	10	0	0.015	<0.002	<0.002	<0.002	NR
				0.015	<0.002	<0.002	<0.002	
			1	0.009	<0.002	<0.002	<0.002	
				0.009	<0.002	<0.002	<0.002	
			3	0.006	<0.002	<0.002	<0.002	
				0.006	<0.002	<0.002	<0.002	
			5	0.004	<0.002	<0.002	<0.002	
				0.004	<0.002	<0.002	<0.002	
			7	0.003	<0.002	<0.002	<0.002	
				0.004	<0.002	<0.002	<0.002	
Australia, 1996, AUS94i55d Springfield via Orange, NSW	SC	15	0	0.038	<0.002	<0.002	0.004	NR
				0.039	<0.002	<0.002	0.004	
			1	0.023	<0.002	<0.002	0.003	
				0.023	<0.002	<0.002	0.003	
			3	0.014	<0.002	<0.002	0.002	
				0.015	<0.002	<0.002	0.002	
			5	0.008	<0.002	<0.002	0.002	
				0.008	<0.002	<0.002	0.002	
			7	0.008	<0.002	<0.002	<0.002	
				0.009	<0.002	<0.002	<0.002	
Australia, 1996, AUS94i55d Springfield via Orange, NSW	SC	30	0	0.087	<0.002	<0.002	0.006	NR
				0.072	<0.002	<0.002	0.005	
			1	0.04	<0.002	<0.002	0.005	
				0.043	<0.002	<0.002	0.005	
			3	0.026	<0.002	<0.002	0.004	
				0.025	<0.002	<0.002	0.004	
			5	0.011	<0.002	<0.002	0.003	
				0.012	<0.002	<0.002	0.003	
			7	0.013	<0.002	<0.002	0.002	
				0.012	<0.002	<0.002	0.002	

Country, Year, Reference	Application		PHI, days	Residues, mg/kg				
	Form	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Keats, 1998e								
Australia, 1998, 98NST08 Mt. Mclaren Queensland	UL	1.25	0	0.035	<0.002	0.005	0.002	NR
				0.041	0.003	0.003	0.006	
			2	0.032	<0.002	<0.002	0.004	
				0.026	0.004	0.004	0.004	
			4	0.014	0.004	0.003	<0.002	
				0.023	0.003	0.005	0.003	
			7	0.009	<0.002	0.005	<0.002	
				0.007	<0.002	0.005	<0.002	
			15	0.004	<0.002	0.003	<0.002	
				0.004	<0.002	0.003	<0.002	
			21	0.002	<0.002	<0.002	<0.002	
				0.003	<0.002	0.002	<0.002	
			28	<0.002	<0.002	<0.002	<0.002	
				<0.002	<0.002	<0.002	<0.002	
Australia, 1998, 98NST08 Mt. Mclaren Queensland	UL	2.5	0	0.067	0.002	0.004	0.005	NR
				0.058	0.002	0.004	0.009	
			2	0.029	0.004	0.002	0.01	
				0.033	0.004	0.006	0.014	
			4	0.018	0.002	0.007	0.004	
				0.024	0.002	0.009	0.008	
			7	0.021	0.002	0.015	<0.002	
				0.021	0.002	0.013	<0.002	
			15	0.008	<0.002	0.003	<0.002	
				0.008	<0.002	0.003	<0.002	
			21	0.007	<0.002	0.006	0.002	
				0.005	<0.002	0.006	0.002	
			28	<0.002	<0.002	<0.002	<0.002	
				<0.002	<0.002	<0.002	<0.002	
Australia, 1998, 98NST08 Mt. Mclaren Queensland	UL	5.0	0	0.20	0.009	0.008	0.02	NR
				0.22	0.003	0.004	0.019	
			2	0.09	0.004	0.003	0.02	
				0.098	0.003	0.005	0.024	
			4	0.059	0.003	0.005	0.006	
				0.047	0.005	0.009	0.01	
			7	0.027	0.002	0.006	<0.002	
				0.027	0.002	0.011	<0.002	
			15	0.014	<0.002	0.009	0.003	
				0.02	0.002	0.011	0.004	
			21	0.02	0.002	0.007	0.003	
				0.021	0.002	0.008	0.003	
			28	<0.002	<0.002	<0.002	<0.002	
				<0.002	<0.002	<0.002	<0.002	

Country, Year, Reference	Application		PHI, days	Residues, mg/kg				
	Form	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Australia, 1998, 98NST08 Mt. Mclaren Queensland	UL	7.5	0	0.29	0.01	0.012	0.024	NR
				0.31	0.014	0.006	0.034	
			2	0.19	0.008	0.007	0.035	
				0.21	0.012	0.013	0.045	
			4	0.089	0.011	0.026	0.01	
				0.079	0.005	0.012	0.01	
			7	0.073	<0.002	0.035	0.002	
				0.073	0.005	0.034	0.005	
			15	0.022	0.002	0.015	0.004	
				0.018	0.002	0.013	0.003	
			21	0.027	0.003	0.012	0.004	
				0.024	0.003	0.013	0.004	
			28	<0.002	<0.002	<0.002	<0.002	
				<0.002	<0.002	<0.002	<0.002	
Keats, 1998f								
Australia, 1998, 98NST10 Clermont Queensland	UL	1.25	0	0.026	<0.002	0.004	0.002	NR
				0.03	0.002	0.003	0.004	
			2	0.022	<0.002	0.004	0.014	
				0.018	0.003	0.004	0.01	
			4	0.01	0.002	0.002	0.004	
				0.009	0.003	0.005	0.003	
			7	0.007	<0.002	<0.002	<0.002	
				0.009	<0.002	<0.002	0.003	
			16	<0.002	<0.002	<0.002	<0.002	
				<0.002	<0.002	<0.002	<0.002	
			21	<0.002	<0.002	<0.002	<0.002	
				<0.002	<0.002	<0.002	<0.002	
			28	<0.002	<0.002	<0.002	<0.002	
				<0.002	<0.002	<0.002	<0.002	
Australia, 1998, 98NST10 Clermont Queensland	UL	2.5	0	0.045	0.004	0.004	0.006	NR
				0.053	0.004	0.004	0.01	
			2	0.034	0.002	0.008	0.011	
				0.028	0.002	0.004	0.009	
			4	0.01	0.002	0.01	0.004	
				0.018	0.003	0.006	0.004	
			7	0.007	<0.002	<0.002	0.003	
				0.011	<0.002	<0.002	<0.002	
			16	<0.002	<0.002	<0.002	<0.002	
				<0.002	<0.002	<0.002	<0.002	
			21	<0.002	<0.002	<0.002	<0.002	
				<0.002	<0.002	<0.002	<0.002	
			28	<0.002	<0.002	<0.002	<0.002	
				<0.002	<0.002	<0.002	<0.002	

Country, Year, Reference	Application		PHI, days	Residues, mg/kg				
	Form	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Australia, 1998. 98NST10 Clermont Queensland	UL	5.0	0	0.13	0.005	0.009	0.016	NR
				0.14	0.009	0.013	0.012	
			2	0.07	0.003	0.013	0.016	
				0.078	0.005	0.009	0.02	
			4	0.03	0.002	0.002	0.008	
				0.038	0.006	0.002	0.004	
			7	0.016	0.002	0.002	0.003	
				0.008	0.002	0.002	0.003	
			16	0.004	<0.002	0.003	<0.002	
				0.004	<0.002	0.003	<0.002	
			21	0.002	<0.002	<0.002	<0.002	
				0.002	<0.002	<0.002	<0.002	
			28	<0.002	<0.002	<0.002	<0.002	
				<0.002	<0.002	<0.002	<0.002	
Australia, 1998, 98NST10 Clermont Queensland	UL	7.5	0	0.16	0.01	0.01	0.018	NR
				0.18	0.01	0.014	0.02	
			2	0.10	0.006	0.003	0.03	
				0.12	0.012	<0.002	0.026	
			4	0.049	0.007	0.009	0.01	
				0.057	0.005	0.011	0.008	
			7	0.014	<0.002	0.005	0.003	
				0.023	0.004	0.003	0.005	
			16	0.003	<0.002	0.002	<0.002	
				0.003	<0.002	0.003	<0.002	
			21	0.005	<0.002	0.003	<0.002	
				0.005	<0.002	0.005	0.002	
			28	<0.002	<0.002	<0.002	<0.002	
				<0.002	<0.002	<0.002	<0.002	
Richard and Muller, 1995c								
Mauritania, 1994, Mauritania 94686XX1	UL	11.04	2	0.095	<0.01	0.011	<0.01	<0.01
				0.011	<0.01	<0.01	<0.01	<0.01
				0.047	<0.01	0.028	<0.01	<0.01
				0.044	<0.01	0.029	<0.01	<0.01
				0.26	0.024	0.120	0.025	<0.01
			9	<0.01	<0.01	<0.01	<0.01	<0.01
				<0.01	<0.01	0.012	<0.01	<0.01
				0.016	<0.01	0.021	<0.01	<0.01
				0.031	<0.01	0.04	<0.01	<0.01
				0.023	<0.01	0.047	<0.01	<0.01
				<0.01	<0.01	0.014	<0.01	<0.01
			16	<0.01	<0.01	<0.01	<0.01	<0.01
				<0.01	<0.01	0.012	<0.01	<0.01
				<0.01	<0.01	0.017	<0.01	<0.01
				0.034	<0.01	0.077	<0.01	<0.01
				0.02	<0.01	0.018	<0.01	<0.01
				<0.01	<0.01	<0.01	<0.01	<0.01
			23	<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	<0.01	<0.01	<0.01	<0.01
				<0.01	<0.01	<0.01	<0.01	<0.01

Country, Year, Reference	Application		PHI, days	Residues, mg/kg				
	Form	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
			30	<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
				0.014	<0.01	0.025	<0.01	<0.01
				0.013	<0.01	0.034	<0.01	<0.01
				<0.01	<0.01	<0.01	<0.01	<0.01
Yslan, 1999								
Russia, 1998	EC	4	0	0.20	<0.01	0.04	0.015	NR
982008STA1				0.26	<0.01	0.041	<0.01	
Levokoumsk			1	0.068	<0.01	0.044	0.02	
(STRAVOPOL				0.14	<0.01	0.058	0.017	
KRAI) Russia North			3	0.046	<0.01	0.05	0.02	
				0.039	<0.01	0.044	0.017	
			7	0.019	<0.01	0.019	<0.01	
				NA	NA	NA	NA	
			14	0.118	<0.01	0.018	<0.01	
				NA	NA	NA	NA	
			21	<0.01	<0.01	<0.01	<0.01	
				NA	NA	NA	NA	
			28	<0.01	<0.01	<0.01	<0.01	
				<0.01	<0.01	<0.01	<0.01	
Viljoen and van Zyl, 1998								
South Africa, 1997, RS97101 De Ar Hanover grassveld	UL	7.5	1 h	3.8	0.14	0.45	<0.01	<0.01
				5.9	0.13	0.68	<0.01	<0.01
			1	3.0	0.04	0.82	0.04	<0.01
				2.6	0.04	0.70	0.01	0.01
			3	0.55	<0.01	0.26	0.11	<0.01
				0.55	<0.01	0.26	0.16	<0.01
			7	0.17	<0.01	0.33	0.18	<0.01
				0.17	<0.01	0.33	0.19	<0.01
			14	0.04	<0.01	0.21	0.06	<0.01
				0.04	<0.01	0.22	0.06	<0.01
			21	0.21	<0.01	0.32	0.13	<0.01
				0.2	<0.01	0.28	0.13	<0.01
South Africa, 1997, RS97101 De Ar Hanover grassveld		15	1 h	8.5	0.19	0.87	<0.01	<0.01
				8.7	0.15	0.81	<0.01	<0.01
			1	2.9	0.03	0.67	<0.01	<0.01
				4.2	0.06	1.0	0.07	0.01
			3	2.1	<0.01	0.78	0.14	<0.01
				2.1	<0.01	0.80	0.14	<0.01
			7	1.9	<0.01	1.0	0.41	<0.01
				1.8	<0.01	1.0	0.41	<0.01
			14	0.67	<0.01	0.61	0.26	<0.01
				0.66	<0.01	0.61	0.26	<0.01
			21	0.33	<0.01	0.48	0.22	<0.01
				0.36	<0.01	0.49	0.22	<0.01

Country, Year, Reference	Application		PHI, days	Residues, mg/kg				
	Form	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
South Africa, 1997, RS97101 De Ar Hanover bossieveld	UL	7.5	1 h	0.69	0.61	0.46	<0.01	0.42
				0.67	0.64	0.44	<0.01	0.37
			1	0.60	0.01	0.37	<0.01	0.19
				0.60	<0.01	0.37	<0.01	0.19
			3	0.40	0.09	0.11	<0.01	0.02
				0.39	0.09	0.11	<0.01	0.03
			7	0.06	0.04	0.12	0.01	<0.01
				0.06	0.04	0.14	0.02	<0.01
			14	0.04	0.02	<0.01	<0.01	0.16
				0.03	0.02	<0.01	<0.01	0.19
			21	<0.05	<0.01	0.02	<0.01	<0.01
				NA	<0.01	0.02	<0.01	<0.01
South Africa, 1997, RS97101 De Ar Hanover bossieveld	UL	15	1 h	0.21	0.33	0.19	<0.01	0.24
				0.21	0.30	0.19	<0.01	0.24
			1	0.21	0.17	0.22	<0.01	0.11
				0.22	0.17	0.22	<0.01	0.11
			3	0.98	0.12	0.16	0.65	<0.01
				1.1	0.20	0.21	0.73	<0.01
			7	0.07	0.04	0.07	0.07	0.08
				0.08	0.04	0.07	0.07	0.09
			14	0.04	<0.01	0.04	0.81	<0.01
				0.04	<0.01	0.06	0.85	<0.01
			21	0.04	0.10	0.25	<0.01	0.04
				0.04	0.10	0.25	<0.01	0.04
South Africa, 1997, RS97101 De Ar Hanover mixedveld	UL	7.5	1 h	2.1	<0.01	0.03	<0.01	0.04
				2.1	<0.01	0.03	<0.01	0.04
			1	1.4	<0.01	0.04	<0.01	0.04
				1.4	<0.01	0.34	<0.01	0.04
			3	0.76	0.09	<0.01	0.06	0.07
				0.73	0.11	<0.01	0.05	0.07
			7	1.1	<0.01	<0.01	0.15	0.07
				0.82	<0.01	<0.01	0.13	0.07
			14	0.38	<0.01	<0.01	0.06	0.07
				0.71	<0.01	<0.01	0.07	0.10
			21	0.43	<0.01	<0.01	0.05	0.09
				0.44	<0.01	<0.01	0.05	0.09
South Africa, 1997, RS97101 De Ar Hanover mixedveld	UL	15	1 h	2.8	<0.01	0.03	<0.01	0.03
				2.8	<0.01	0.02	<0.01	0.03
			1	2.6	<0.01	0.01	<0.01	0.09
				2.6	<0.01	0.01	<0.01	0.10
			3	2.3	0.06	<0.01	<0.01	0.09
				2.3	<0.01	<0.01	<0.01	0.09
			7	1.9	0.11	<0.01	0.18	0.04
				1.9	0.10	<0.01	0.18	0.04
			14	0.86	<0.01	0.09	<0.01	0.15
				0.82	<0.01	0.09	0.14	0.15
			21	0.17	<0.01	0.13	0.02	0.11
				0.33	<0.01	0.17	<0.01	0.09

NR: not reported NA: not analysed

Table 78. Residues in barley straw after single seed treatments.

Country, Year, Reference	Sample	Application		PHI,	Residues, mg/kg				
		Form	g ai/t	days	Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Seed treatment, 2 replicates (Muller, 1995a)									
France, 1993-1994, 94501OR1	straw	FS	1500	260	<0.01	<0.01	<0.01	NR	<0.01
France, 1993-1994, 94501AM1	straw	FS	1000 1500	271 271	0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	0.024 0.024 0.038 0.032	NR	<0.01 0.01 0.014 0.01
France, 1993-1994, 94501RS1	straw	FS	1500	260	<0.01	<0.01	<0.01	NR	<0.01
France, 1993-1994, 94501DJ1	straw	FS	1000 1500	250 250	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	0.016 0.023 0.015 0.013	NR	<0.01 <0.01 <0.01 <0.01
France, 1993-1994, 94501LY1	straw	FS	1000 1500	249 249	0.014 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	0.013 0.014 0.012 0.014	NR	<0.01 <0.01 <0.01 <0.01

NR: not reported

Table 79. Residues in rice straw. All single applications.

Commodity, Country, Year, Reference	Application				PHI,	Residues, mg/kg				
	Form	g ai/t	g ai/ha	No.	days	Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Seed treatment, 2 replicates (Keats, 1996e)										
Australia, 1996, AK96059	FS		25 50 100	1	167	<0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002	<0.002 <0.002 0.003 0.005	NR
Seed treatment, 3 replicates (Keats, 1996h)										
Australia, 1996, AK96060	FS		25 50 100	1	215	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	NR
Seed treatment, 2 replicates (Keats, 1996i)										
Australia, 1996, AK96061	FS		12.5 25 50	1	144	<0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002	<0.002 <0.002 0.007 0.008	NR
Seed treatment (Maestracci, 1997c)										
France, 1996, 96561AV1	FS	130		1	143	<0.002	<0.002	<0.002	<0.002	<0.002
Seed treatment (Maestracci, 1998e)										
France, 1997, 97545AV1 variety Thaibonnet	FS	130		1	149	<0.005	<0.005	<0.005	<0.005	NR
France, 1997, 97545AV2 variety Ariete	FS	130		1	149	<0.005	<0.005	<0.005	<0.005	NR
Seed treatment (Maestracci, 1998f)										
France, 1997, 97546AV1 variety Ariete	FS	130		1	133	<0.005	<0.005	<0.005	<0.005	NR
France, 1997, 97546AV2 variety Thaibonnet	FS	130		1	133	<0.005	<0.005	<0.005	<0.005	NR
Seed treatment, 2 replicates (Yslan and Baudet, 1999a)										
Spain, 1998, 98639TR1	FS	263	52.6	1	133	<0.002	<0.002	<0.002	<0.002	NR
Seed treatment, 2 replicates (Mede, 1996b)										
USA, 1995[1], 95-0248AR	FS		56	1	119	<0.01 <0.01	<0.01 <0.01	0.017 0.012	<0.003 <0.003	<0.01 <0.01

Commodity, Country, Year, Reference	Application				PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
USA, 1995[1], 95-0249AR	FS		56	1	134	<0.01 <0.01	<0.003 <0.01	<0.01 0.012	<0.01 <0.01	<0.003 <0.003
USA, 1995[1], 95-0250CA	FS		56	1	139	<0.01 <0.003	<0.003 <0.003	<0.003 <0.003	<0.003 <0.003	<0.003 <0.003
USA, 1995[1], 95-0251CA	FS		56	1	139	<0.003 <0.003	<0.01 <0.01	<0.003 <0.003	<0.01 <0.003	<0.003 <0.003
USA, 1995[1], 95-0252LA	FS		56	1	107	<0.01	<0.01	0.011	<0.01	<0.01
USA, 1995[1], 95-0253LA	FS		56	1	112	<0.01 <0.01	<0.01 <0.01	0.01 <0.01	<0.01 <0.01	<0.003 <0.003
USA, 1995[1], 95-0254MS	FS		56	1	128	<0.01 <0.01	<0.01 <0.01	0.024 0.014	<0.01 <0.01	0.014 0.011
USA, 1995[1], 95-0255TX	FS		56	1	119	<0.003 <0.01	<0.003 <0.01	<0.01 0.01	<0.003 <0.003	<0.003 <0.003
USA, 1995[1], 95-0256MS	FS		56	1	130	<0.01	<0.01	<0.01	<0.003	<0.003
USA, 1995[1], 95-0257LA	FS		56	1	109	0.012 <0.01	<0.01 <0.01	0.02 0.015	<0.01 <0.01	0.014 <0.01
Seed treatment, 2 replicates (Mede, 1997)										
USA, 1996[1], 10392-01	FS		54	1	126	<0.003	<0.003	<0.003	<0.01	NR
USA, 1996[1], 10392-02	FS		58	1	125	<0.01 <0.01	<0.01 <0.01	0.012 0.013	<0.003 <0.003	NR
USA, 1996[1], 10392-03	FS		57	1	129	<0.01 <0.01	<0.01 <0.01	<0.01 0.014	<0.003 <0.003	NR
USA, 1996[1], 10392-04	FS		56	1	110	<0.003	<0.003	<0.003	<0.003	NR
USA, 1996[1], 10392-05	FS		56	1	117	<0.01	<0.01	<0.01	<0.003	NR
USA, 1996[1], 10392-07	FS		56	1	128	<0.01	<0.01	<0.01	<0.003	NR
USA, 1996[1], 10392-08	FS		57	1	138	<0.01	<0.01	<0.003	<0.01	NR
Seed box treatment, 3 replicates (Anon., 1994)										
Japan, 1993, I.E.T. Official residue trial, Ushuku	GR		0.5 g ai/box	1	132	0.01 0.01 0.01	0.09 0.06 0.06	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01
Japan, 1993, I.E.T. Official residue trial, Shiga	GR		0.5 g ai/box	1	141	0.04 0.03 0.03	0.19 0.16 0.12	0.03 0.03 0.02	0.01 0.01 0.01	0.01 0.01 0.01
Seed box treatment, 2 replicates (Anon., 1995a)										
Japan, 1995, Nihon Noyaku	GR		0.5 g ai/box	1	123	0.04	<0.01	<0.01	<0.01	<0.01
Seed box treatment, 2 replicates (Anon., 1995b)										
Japan, 1995, Nissan, Fukui	GR		0.5 g ai/box	1	140	<0.01	0.01	<0.01	<0.01	<0.01
Japan, 1995, Nissan, Mie	GR		0.5 g ai/box	1	118	0.01	0.04	0.02	<0.01	<0.01
Soil pre-plant incorporation broadcast treatment, 2 replicates (Mede, 1996b)										
USA, 1995[1], 95-0248AR	WG		56	1	119	<0.01 <0.01	<0.01 <0.01	0.012 0.017	<0.003 <0.003	<0.01 <0.01
USA, 1995[1], 95-0249AR	WG		56	1	134	<0.01	<0.003	<0.01	<0.003	<0.003
USA, 1995[1], 95-0250CA	WG		56	1	141	<0.01	<0.003	<0.003	<0.003	<0.003
USA, 1995[1], 95-0251CA	WG		56	1	140	<0.01 <0.003	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.003 <0.003
USA, 1995[1], 95-0252LA	WG		56	1	112	0.016 <0.01	<0.01 <0.01	0.015 0.014	<0.003 <0.01	0.019 0.014
USA, 1995[1], 95-0253LA	WG		56	1	114	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.003	<0.003 <0.003
USA, 1995[1], 95-0254MS	WG		56	1	128	<0.003 <0.01	<0.01 <0.01	<0.01 0.013	<0.003 <0.003	<0.01 0.011
USA, 1995[1], 95-0255TX	WG		56	1	119	<0.01 <0.003	<0.01 <0.01	<0.01 <0.01	<0.003 <0.01	<0.003 <0.003
USA, 1995[1], 95-0256MS	WG		56	1	130	<0.01	<0.01	<0.01	<0.003	<0.003
USA, 1995[1], 95-0257LA	WG		56	1	110	<0.01 <0.01	<0.01 <0.01	0.014 0.012	<0.01 <0.01	<0.01 <0.01

Commodity, Country, Year, Reference	Application				PHI, days	Residues, mg/kg				
	Form	g ai/t	g ai/ha	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Soil pre-plant incorporation broadcast treatment, 2 replicates (Mede, 1997)										
USA, 1996[1], 10392-01	SC		57	1	133	<0.01	<0.01	<0.01	<0.003	NR
USA, 1996[1], 10392-02	SC		57	1	125	<0.01	<0.01	<0.01	<0.003	NR
USA, 1996[1], 10392-03	SC		57	1	136	<0.003 <0.01	<0.01 <0.01	<0.01 <0.01	<0.003	NR
USA, 1996[1], 10392-04	SC		56	1	113	<0.003	<0.003	<0.003	<0.003	NR
USA, 1996[1], 10392-05	SC		57	1	120	<0.01	<0.01	<0.01	<0.003	NR
USA, 1996[1], 10392-07	SC		59	1	128	<0.01	<0.01	<0.01	<0.003	NR
USA, 1996[1], 10392-08	SC		56	1	143	<0.01	<0.01	<0.01	<0.01	NR
Foliar treatment, 3 replicates (Maycey *et al*., 1994c)										
Thailand, 1992/1993, 92/276 Supanuri Rice Research Station	SC		50	1	49	0.039 0.061 0.042	0.01 0.01 0.009	0.028 0.047 0.059	0.048 0.075 0.051	NR
Thailand, 1992/1993, 92/276 Supanuri Rice Research Station	SC		50	1	45	0.017 0.006 0.014	0.006 <0.005 0.005	0.016 0.01 0.011	0.028 0.013 0.017	NR
Thailand, 1992/1993, 92/276 Supanuri	SC		50	1	48	0.038 0.072 0.13	0.009 0.014 0.03	0.044 0.053 0.18	0.046 0.068 0.2	NR
Thailand, 1992/1993, 92/276 Nontaburi	SC		50	1	45	0.041 0.022 0.09	0.006 0.005 0.012	0.041 0.059 0.095	0.045 0.032 0.095	NR
Foliar treatment, 3 replicates, (Maycey *et al*., 1994a)										
Philippines, 1992, 92/277 Luzon	SC		50	1	51	0.022 0.027 0.018	0.005 0.009 0.006	0.037 0.043 0.027	0.065 0.084 0.059	NR
Foliar treatment, 3 replicates (Maycey *et al*., 1994b)										
Taiwan, 1993, 92/275 Chitong Li	SC		50	1	79	<0.005	<0.005	<0.005	<0.005	NR
Broadcast treatment after transplanting followed by foliar treatment, 3 replicates, (Maycey *et al*., 1994a)										
Philippines, 1992, 92/277 Luzon	GR SC		50	2	51	0.014 0.018 0.02	0.009 0.008 <0.005	0.024 0.042 0.039	0.046 0.053 0.049	NR
Broadcast treatment into flooded paddy, 3 replicates (Maycey *et al*., 1994a)										
Philippines, 1992, 92/277 Luzon	GR		50	1	81	0.009 0.006 0.008	<0.005 <0.005 0.005	0.008 0.008 0.01	0.018 0.025 0.021	NR
Broadcast treatment into flooded paddy, 3 replicates (Maycey *et al*., 1994b)										
Taiwan, 1993, 92/275 Hsinung Li	GR		50	1	89	<0.005	<0.005	<0.005	<0.005	NR
Broadcast treatment into flooded paddy, (Maycey *et al*., 1994c)										
Thailand, 1992/1993, 92/276 Supanuri Rice Research Station, **replicate 1**	GR		50	1	79	<0.005	0.008	<0.005	0.01	NR
Thailand, 1992/1993, 92/276 Supanuri Rice Research Station, **replicate 2**	GR		50	1	75	<0.005	0.008	0.006	0.012	NR
Thailand, 1992/1993, 92/276 Supanuri	GR		50	1	79	<0.005 <0.005 <0.005	0.011 0.012 0.013	0.008 <0.005 <0.005	0.021 0.017 0.015	NR
Thailand, 1992/1993, 92/276 Nontaburi	GR		50	1	75	<0.005 <0.005 <0.005	<0.005 <0.005 <0.005	<0.005 <0.005 0.006	<0.005 <0.005 0.006	NR

NR: not reported

[1]residues reported as ND (not detected) in the original report are shown as <0.00x, i.e. the minimum limit of detection (MLD)

Table 80. Residues in sorghum forage and straw from trials in Australia. All single applications.

Year, Reference	Sample	Application			PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Foliar treatment at stage BBCH 85, 2 replicates (Keats, 1996n)										
1995, AK96074	forage	UL		10	0	0.019	0.003	0.002	<0.002	NR
						0.017	0.003	<0.002	<0.002	
					1	0.016	0.003	0.008	<0.002	
						0.016	0.003	0.007	<0.002	
					3	0.011	0.002	0.002	<0.002	
						0.011	0.002	<0.002	<0.002	
					7	0.002	<0.002	<0.002	<0.002	
						0.003	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
	straw				35	<0.002	<0.002	<0.002	<0.002	
					48	<0.002	<0.002	<0.002	<0.002	
1995, AK96074	forage	UL		20	0	0.065	0.005	<0.002	<0.002	NR
						0.055	0.005	<0.002	<0.002	
					1	0.019	0.002	0.003	<0.002	
						0.018	0.002	0.002	<0.002	
					3	0.007	0.003	0.003	<0.002	
						0.007	<0.002	0.002	<0.002	
					7	0.004	<0.002	<0.002	<0.002	
						0.004	<0.002	<0.002	<0.002	
					21	0.002	<0.002	<0.002	<0.002	
						0.002	<0.002	<0.002	<0.002	
	straw	UL		20	35	0.004	<0.002	0.003	<0.002	
						0.002	<0.002	0.002	<0.002	
					48	<0.002	<0.002	<0.002	<0.002	
Foliar treatment at stage BBCH 75 (Keats, 1996o)										
1996, AK96075	forage	UL		10	0	0.067	<0.002	0.003	<0.002	NR
					1	0.057	<0.002	0.004	<0.002	
					3	0.038	<0.002	0.003	<0.002	
					7	0.018	<0.002	0.003	<0.002	
					17	0.005	<0.002	0.003	<0.002	
	straw				42	<0.002	<0.002	<0.002	<0.002	
1996, AK96075	forage	UL		20	0	0.13	0.002	<0.002	<0.002	NR
					1	0.14	<0.002	<0.002	<0.002	
					3	0.078	<0.002	<0.002	<0.002	
					7	0.075	<0.002	<0.002	<0.002	
					17	0.005	<0.002	<0.002	<0.002	
	straw				42	0.002	<0.002	<0.002	<0.002	
Foliar treatment at maturity, BBCH 87, 2 replicates (Keats, 1996p)										
1996, AK96076	forage	UL		10	0	0.021	0.003	<0.002	<0.002	NR
						0.019	0.003	<0.002	<0.002	
					1	0.013	0.002	0.002	<0.002	
						0.013	0.002	<0.002	<0.002	
					3	0.015	0.003	0.003	<0.002	
						0.009	0.002	<0.002	<0.002	
					7	0.002	<0.002	<0.002	<0.002	
						0.003	<0.002	<0.002	<0.002	
					21	0.002	<0.002	<0.002	<0.002	
						<0.002	<0.002	<0.002	<0.002	
	straw				42	<0.002	<0.002	<0.002	<0.002	
1996, AK96076	forage	UL		20	0	0.054	0.009	0.002	<0.002	NR
						0.052	0.009	0.002	<0.002	
					1	0.019	0.003	<0.002	<0.002	
						0.022	0.004	0.002	<0.002	
					3	0.017	0.003	<0.002	<0.002	
						0.017	0.003	0.002	<0.002	
					7	0.008	<0.002	0.002	<0.002	
						0.007	<0.002	0.002	<0.002	
					21	0.007	<0.002	0.002	<0.002	
						0.006	<0.002	0.002	<0.002	
	straw				42	0.004	<0.002	0.002	<0.002	
						0.004		0.002		

Year, Reference	Sample	Application			PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Foliar treatment at maturity, BBCH 87, 2 replicates (Keats, 1998a)										
1998, AK98024	forage	SC		1.25	0	0.072	0.002	0.002	0.002	NR
						0.08	0.003	0.003	0.002	
					4	0.004	<0.002	<0.002	<0.002	
					7	0.004	<0.002	<0.002	<0.002	
					15	<0.002	<0.002	<0.002	<0.002	
						0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
						0.002	<0.002	<0.002	<0.002	
					29	<0.002	<0.002	<0.002	<0.002	
1998, AK98024	forage	SC		2.5	0	0.12	0.006	0.004	0.009	NR
						0.13	0.007	0.004	0.01	
					4	0.02	<0.002	0.003	0.003	
						0.018	<0.002	0.002	0.003	
					7	0.007	<0.002	<0.002	<0.002	
						0.006	<0.002	<0.002	<0.002	
					15	<0.002	<0.002	0.002	<0.002	
						0.002	<0.002	<0.002	<0.002	
					21	0.002	<0.002	<0.002	<0.002	
						0.002	<0.002	<0.002	<0.002	
					29	<0.002	<0.002	<0.002	<0.002	
1998, AK98024	forage	SC		5.0	0	0.37	0.015	0.022	0.025	NR
						0.39	0.015	0.023	0.034	
					4	0.048	0.002	<0.002	<0.002	
						0.043	0.002	<0.002	<0.002	
					7	0.011	0.002	<0.002	<0.002	
						0.01	0.002	0.003	<0.002	
					15	0.002	<0.002	0.002	<0.002	
						0.003	<0.002	<0.002	<0.002	
					21	0.002	<0.002	<0.002	0.002	
					29	<0.002	<0.002	<0.002	<0.002	
Foliar treatment at maturity, BBCH 87, 2 replicates (Keats, 1998b)										
1998, AK98025	forage	UL		1.25	0	0.049	0.002	<0.002	0.005	NR
						0.053	0.003	<0.002	0.004	
					2	0.01	0.002	<0.002	0.002	
						0.009	<0.002	<0.002	0.002	
					4	0.004	<0.002	<0.002	0.002	
						0.003	<0.002	<0.002	<0.002	
					7	0.006	<0.002	0.004	<0.002	
						0.005	<0.002	0.004	<0.002	
					15	<0.002	<0.002	<0.002	<0.002	
					21	0.002	<0.002	<0.002	<0.002	
					28	<0.002	<0.002	<0.002	<0.002	
1998, AK98025	forage	UL		2.5	0	0.067	0.004	<0.002	0.007	NR
						0.072	0.004	<0.002	0.01	
					2	0.011	0.002	<0.002	0.002	
						0.012	<0.002	<0.002	0.002	
					4	0.014	0.002	<0.002	0.003	
						0.016	0.002	<0.002	<0.002	
					7	0.007	<0.002	0.006	<0.002	
						0.007	<0.002	0.007	<0.002	
					15	0.003	<0.002	0.004	<0.002	
						0.004	<0.002	0.004	<0.002	
					21	0.005	<0.002	0.005	<0.002	
					28	<0.002	<0.002	<0.002	<0.002	
1998, AK98025	forage	UL		5.0	0	0.11	0.005	0.005	0.028	NR
						0.1	0.007	0.003	0.032	
					2	0.039	0.002	0.005	0.009	
						0.035	<0.002	0.003	0.011	
					4	0.035	0.002	0.002	0.004	
						0.039	<0.002	0.002	0.003	
					7	0.01	0.004	0.017	<0.002	
						0.009	0.005	0.023	<0.002	
					15	0.008	<0.002	0.005	<0.002	
					21	0.008	<0.002	0.002	<0.002	
						0.008	<0.002	0.003	<0.002	
					28	<0.002	<0.002	<0.002	<0.002	

Year, Reference	Sample	Application			PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
1998, AK98025	forage	UL		7.5	0	0.27	0.009	0.006	0.042	NR
						0.27	0.011	0.01	0.04	
					2	0.067	0.002	0.006	0.012	
						0.073	0.002	0.008	0.015	
					4	0.059	0.008	0.002	0.009	
						0.065	0.004	0.002	0.011	
					7	0.061	0.005	0.034	0.002	
						0.032	0.004	0.034	0.002	
					15	0.015	0.002	0.011	0.002	
						0.02	<0.002	0.012	0.003	
					21	0.018	0.002	0.011	0.002	
						0.017	0.002	0.011	0.002	
					28	<0.002	<0.002	<0.002	<0.002	
Foliar treatment at stage BBCH 73, 2 replicates (Keats, 1998c)										
1998, AK98027	forage	UL		1.25	0	0.029	<0.002	<0.002	0.002	NR
						0.033	<0.002	<0.002	0.002	
					2	0.009	<0.002	<0.002	0.004	
						0.013	<0.002	0.003	<0.002	
					4	0.004	<0.002	<0.002	<0.002	
						0.003	<0.002	<0.002	<0.002	
					7	0.003	<0.002	0.002	<0.002	
						0.005	<0.002	0.002	<0.002	
					15	<0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
					28	<0.002	<0.002	<0.002	<0.002	
1998, AK98027	forage	UL		2.5	0	0.047	0.002	<0.002	0.005	NR
						0.053	0.003	<0.002	0.004	
					2	0.011	<0.002	0.002	0.002	
						0.013	<0.02	0.002	0.002	
					4	0.014	<0.002	<0.002	<0.002	
						0.006	0.002	<0.002	<0.002	
					7	0.007	0.003	0.002	<0.002	
						0.005	<0.002	<0.002	<0.002	
					15	0.002	<0.002	<0.002	<0.002	
					21	<0.002	<0.002	<0.002	<0.002	
					28	<0.002	<0.002	<0.002	<0.002	
1998, AK98027	forage	UL		5.0	0	0.13	0.002	0.002	0.01	NR
						0.12	0.003	0.003	0.014	
					2	0.039	<0.002	0.002	0.012	
						0.041	0.003	0.005	0.008	
					4	0.019	0.002	0.002	0.003	
						0.022	<0.002	0.002	0.002	
					7	0.01	0.002	0.006	0.003	
						0.014	0.002	0.012	0.002	
					15	0.002	<0.002	0.003	<0.002	
					21	<0.002	<0.002	0.002	<0.002	
					28	<0.002	<0.002	<0.002	<0.002	
1998, AK98027	forage	UL		7.5	0	0.32	0.005	0.004	0.019	NR
						0.32	0.006	0.01	0.023	
					2	0.073	<0.002	0.007	0.015	
						0.069	0.003	0.006	0.013	
					4	0.03	0.002	0.002	0.004	
						0.038	0.006	0.002	0.008	
					7	0.022	<0.002	0.011	0.002	
						0.03	0.003	0.015	0.002	
					15	0.003	<0.002	0.003	<0.002	
						0.002	<0.002	0.004	<0.002	
					21	<0.002	<0.002	0.002	<0.002	
						<0.002	<0.002	0.003	<0.002	
					28	<0.002	<0.002	<0.002	<0.002	
Seed treatment before sowing, GAP pending, 2 replicates (Keats, 1998g)										
1998, AK98030	forage	FS	750		35	<0.002	<0.002	<0.002	<0.002	NR
			1500			<0.002	<0.002	<0.002	<0.002	
	straw		750		138	<0.002	<0.002	<0.002	<0.002	
			1500			<0.002	<0.002	<0.002	<0.002	
Seed treatment before sowing, GAP pending, 2 replicates (Keats, 1998h)										
1998, AK98031	forage	FS	750		33	<0.002	<0.002	<0.002	<0.002	NR
			1500			<0.002	<0.002	<0.002	<0.002	

Year, Reference	Sample	Application			PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
	straw		750		104	<0.002	<0.002	<0.002	<0.002	
			1500			<0.002	<0.002	<0.002	<0.002	

NR: not reported

Table 81. Residues in wheat straw. All single applications.

Crop, Country, Year, Reference	Sample	Application				PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha	g ai/hl		Fipronil	MB 45950	MB 46136	MB 46513	RPA 200766
Seed treatment, 2 replicates (Muller, 1994k)											
Wheat, winter, France 1992/3, 93-507E1	forage[1]	FS	1000			121	0.016	<0.01	<0.01	NR	<0.01
							0.016	<0.01	<0.01		0.01
						182	<0.01	<0.01	<0.01		<0.01
	straw					251	<0.01	<0.01	<0.01	NR	<0.01
Seed treatment, 2 replicates (Muller, 1996j)											
Wheat, spring, France, 1995, 95-518RS1	forage	FS	500			74	<0.01	<0.01	<0.01	NR	<0.01
	straw					131	<0.01	<0.01	<0.01		<0.01
Seed treatment, 2 replicates (Richard and Muller, 1994h)											
Wheat , winter, France 1992/3, 93-508B1	straw	FS	1000			279	<0.01	<0.01	<0.01	NR	0.017
							<0.01	<0.01	<0.01		0.02
			1500				0.017	<0.01	0.011		0.017
							0.023	<0.01	0.012		0.015
Wheat, winter, France 1992/3, 93-508C1	straw	FS	1500			286	<0.01	<0.01	<0.01	NR	<0.01
Wheat, winter, France 1992/3, 93-508D1	straw	FS	1000			269	<0.01	<0.01	<0.01	NR	<0.01
			1500				<0.01	<0.01	<0.01		<0.01
							<0.01	<0.01	0.011		<0.01
Wheat, winter, France 1992/3, 93-508E1	straw	FS	1500			264	<0.01	<0.01	<0.01	NR	<0.01
Wheat, winter, France 1992/3, 93-508F1	straw	FS	1500			268	<0.01	<0.01	<0.01	NR	<0.01
Wheat, winter, France 1992/3, 93-508K1	straw	FS	1000			226	<0.01	<0.01	<0.01	NR	<0.01
			1500				<0.01	<0.01	<0.01		<0.01
Seed treatment, 2 replicates (Muller, 1995b)											
Wheat, winter, France, 1993/4, 94-500BX1	straw	FS	1000			244	<0.01	<0.01	0.013	NR	<0.01
							0.01	<0.01	<0.01		<0.01
			1500				<0.01	<0.01	<0.01		<0.01
							0.012	<0.01	0.013		<0.01
Wheat, winter, France, 1993/4, 94-500RN1	straw	FS	1000			260	<0.01	<0.01	<0.01	NR	<0.01
			1500				<0.01	<0.01	0.06		0.012
							0.011	<0.01	0.036		0.01
Wheat, winter, France, 1993/4, 94-500AM1	straw	FS	1000			286	<0.01	<0.01	<0.01	NR	<0.01
			1500				<0.01	<0.01	0.023		0.01
							<0.01	<0.01	0.025		0.011
Wheat, winter, France, 1993/4, 94-500DJ1	straw	FS	1000			262	<0.01	<0.01	<0.01	NR	<0.01
							<0.01	<0.01	0.01		<0.01
			1500				<0.01	<0.01	0.015		<0.01
							<0.01	<0.01	0.019		<0.01
Wheat, winter, France, 1993/4, 94-500LY1	straw	FS	1000			245	<0.01	<0.01	<0.01	NR	0.014
							<0.01	<0.01	<0.01		0.012
			1500				<0.01	<0.01	<0.01		0.013
							0.011	<0.01	<0.01		0.01
Wheat, winter, France, 1993/4, 94-500AV1	straw	FS	1500			245	<0.01	<0.01	<0.01	NR	<0.01

Crop, Country, Year, Reference	Sample	Application				PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha	g ai/hl		Fipronil	MB 45950	MB 46136	MB 46513	RPA 200766
Seed treatment, 2 replicates (Muller, 1996k)											
Wheat, spring, France, 1995, 95-507BX1	straw	FS	500			140	0.011 0.014	<0.01 <0.01	0.014 <0.01	NR	<0.01 <0.01
Wheat, spring, France, 1995, 95-507AM1	straw	FS	500			128	<0.01	<0.01	<0.01	NR	<0.01
Wheat, spring, France, 1995, 95-507RS1	straw	FS	500			131	<0.01	<0.01	<0.01	NR	<0.01
Wheat, spring, France, 1995, 95-507LY1	straw	FS	500			145	0.011 <0.01	<0.01 <0.01	<0.01 <0.01	NR	<0.01 <0.01
Foliar treatment, 2 replicates (Muller, 1996a)											
Wheat, winter, Poland, 1995, 95795PL1 *evaluated acc. to Czech/Slovak GAP*	straw	WG		20	6.67	44	0.017 0.014	<0.005 <0.05	0.015 0.006	0.029 0.013	<0.005 <0.005
Foliar treatment, no reference (summary table, no GLP report available)											
Wheat, winter, Russia, 1997, Krasnodar	straw	EC		24	12	27	<0.01	NR	NR	NR	NR
Wheat, winter, Russia, 1997, Rostov	straw	EC		20	10	25	<0.01	NR	NR	NR	NR
Wheat, winter, Russia, 1997, Voronesh	straw	EC		20	10	38	<0.01	NR	NR	NR	NR
Wheat, spring, Russia, 1998, Volgograd	straw	EC		21	10.5	49	<0.01	NR	<0.01	<0.01	NR
Wheat, spring, Russia, 1998, Saratov	straw	EC		21	10.5	49	<0.01	NR	<0.01	<0.01	NR

[1] data from green plant residues, early March and early May samplings, should cover 15-20 cm stage to stem elongation
NR: not reported

Table 82. Residues in cotton plants and gin trash.

Country, Year, Reference	Sample	Application					PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	MB 46513	RPA 200766
Seed treatment, 2 replicates (Keats, 1997i)												
Australia, 1995, AK97018 Clare, QLD	plant	FS	5000 10 000			1	169	0.007 0.006 0.01 0.009	<0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002	0.002 0.002 0.002 0.002	NR
Foliar treatment, 2 replicates (Keats, 1997e)												
Australia, 1995, AK97016 Breeza, NSW	Plant (trash)	EC		200		4	14 24 28	0.64 0.29 0.093 0.12 0.16 0.19	0.21 0.08 0.024 0.035 0.041 0.058	0.079 0.002 <0.002 0.004 0.004 0.005	0.82 0.58 0.29 0.30 0.31 0.36	NR
Seed treatment followed by foliar treatments, 2 replicates (Keats, 1997f)												
Australia, 1996, AK97015 Kincora Queensland	Plant	FS + SC + ULV	5000	 25 25		1 1 1	113	0.003 0.003	<0.002 <0.002	0.011 0.009	0.002 0.002	NR
Australia, 1996, AK97015 Kincora Queensland	plant	FS + SC + ULV	10 000	 50 50		1 1 1	113	0.004 0.004	<0.002 <0.002	0.009 0.008	0.002 0.002	NR

Country, Year, Reference	Sample	Application					PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	MB 46513	RPA 200766
Australia,	plant	FS	10 000			1	113	0.01	0.002	0.024	0.008	NR
1996,		+						0.01	<0.002	0.022	0.007	
AK97015		SC		100		1						
Kincora		+										
Queensland		ULV		100		1						
Australia,	plant	FS	5000			1	4	0.048	0.004	0.3	0.016	NR
1996,		+						0.053	0.004	0.35	0.017	
AK97015		SC		25		1	7	0.057	0.006	0.30	0.01	
Kincora		+						0.052	0.005	0.27	0.009	
Queensland		ULV		25		3	14	0.023	0.002	0.33	0.013	
								0.021	0.002	0.31	0.013	
							21	0.019	<0.002	0.076	0.006	
								0.018	<0.002	0.078	0.005	
							28	0.016	<0.002	0.18	0.004	
								0.017	<0.002	0.17	0.004	
Australia,	plant	FS	10 000			1	4	0.33	0.012	1.5	0.059	NR
1996,		+						0.29	0.011	1.3	0.059	
AK97015		SC		50		1	7	0.078	0.004	0.62	0.02	
Kincora		+						0.074	0.003	0.52	0.018	
Queensland		ULV		50		3	14	0.068	0.003	0.32	0.013	
								0.066	0.003	0.34	0.014	
							21	0.047	0.002	0.4	0.015	
								0.046	0.005	0.38	0.017	
							28	0.032	0.004	0.3	0.011	
								0.027	<0.002	0.27	0.011	
Australia,	plant	FS	10 000			1	4	0.41	0.005	1.3	0.032	NR
1996,		+						0.35	0.004	1.1	0.024	
AK97015		SC		100		1	7	0.36	0.007	1.6	0.086	
Kincora		+						0.34	0.007	1.5	0.085	
Queensland		ULV		100		3	14	0.11	0.003	1.0	0.034	
								0.10	0.002	0.89	0.049	
							21	0.084	0.002	0.59	0.022	
								0.077	0.002	0.49	0.019	
							28	0.019	<0.002	0.13	0.005	
								0.015	<0.002	0.072	0.004	
Seed treatment followed by foliar treatments, 2 replicates (Keats, 1997g)												
Australia,	plant	FS	5000			1	109	<0.002	<0.002	<0.002	<0.002	NR
1996,		+										
AK97014		SC		25		1						
Breeza		+										
NSW		ULV		25		1						
Australia,	plant	FS	10 000			1	109	<0.002	<0.002	<0.002	<0.002	NR
1996,		+										
AK97014		SC		50		1						
Breeza		+										
NSW		ULV		50		1						
Australia,	plant	FS	10 000			1	109	<0.002	<0.002	<0.002	<0.002	NR
1996,		+										
AK97014		SC		100		1						
Breeza		+										
NSW		ULV		100		1						
Australia,	plant	FS	5000			1	0	0.87	0.01	0.062	0.006	NR
1996,		+						0.94	0.01	0.056	0.006	
AK97014		SC		25		1	6	0.17	0.006	0.08	0.016	
Breeza		+						0.14	0.006	0.10	0.016	
NSW		ULV		25		3	15	0.064	0.003	0.073	0.012	
								0.057	0.002	0.075	0.011	
							31	0.025	<0.002	0.005	0.003	
								0.013	<0.002	0.004	0.003	
Australia,	plant	FS	10 000			1	0	1.7	0.03	0.069	0.005	NR
1996,		+						1.3	0.014	0.033	0.003	
AK97014		SC		50		1	6	0.064	0.004	0.086	0.008	
Breeza		+						0.054	0.003	0.079	0.006	
NSW		ULV		50		3	15	0.05	0.002	0.087	0.006	
								0.044	0.002	0.089	0.005	
							31	0.027	0.002	0.024	0.005	
								0.025	<0.002	0.016	0.005	
Australia,	plant	FS	10 000			1	0	2.5	0.024	0.076	0.004	NR
1996,		+						2.1	0.016	0.055	0.003	
AK97014		SC		100		1	6	0.15	0.005	0.12	0.006	
Breeza		+						0.09	0.005	0.15	0.005	

Country, Year, Reference	Sample	Application					PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	MB 46513	RPA 200766
NSW		ULV		100		3	15	0.059	0.003	0.10	0.005	
								0.069	0.003	0.17	0.012	
							31	0.045	0.002	0.032	0.002	
								0.038	<0.002	0.03	0.003	
Single foliar treatments, 2 replicates (Lynch, 1998)												
Australia,	plant	SC		35.4	44.8	1	0	0.086	<0.002	0.056	0.024	NR
1998,								0.09	<0.002	0.061	0.023	
AK99029						1	15	0.053	0.004	0.053	0.015	
Jambin								0.058	0.003	0.058	0.009	
Queensland						1	30	0.01	0.003	0.036	0.008	
								0.014	0.004	0.042	0.007	
Single						1	43	0.004	<0.002	0.032	0.003	
applications								0.005	<0.002	0.028	0.003	
to separate						1	57	<0.002	<0.002	0.026	<0.002	
plots								<0.002	<0.002	0.021	<0.002	
						1	71	<0.002	<0.002	0.022	<0.002	
								<0.002	<0.002	0.016	<0.002	
						1	85	<0.002	<0.002	0.008	<0.002	
								<0.002	<0.002	0.006	<0.002	
						1	99	<0.002	<0.002	<0.002	<0.002	
Australia,	gin	SC		35.4	44.8	1	0	0.26	0.01	0.031	0.011	NR
1998,	trash							0.28	0.01	0.038	0.014	
AK99029						1	15	0.16	0.012	0.043	0.02	
Jambin								0.15	0.012	0.036	0.015	
Queensland						1	30	0.098	0.009	0.032	0.009	
								0.086	0.006	0.027	0.006	
Single						1	43	0.035	0.005	0.021	<0.002	
applications								0.028	0.003	0.013	<0.002	
to separate						1	57	0.008	<0.002	0.008	<0.002	
plots								0.005	<0.002	0.004	<0.002	
						1	71	0.003	<0.002	0.003	<0.002	
								<0.002	<0.002	0.005	<0.002	
						1	85	<0.002	<0.002	0.002	<0.002	
						1	99	<0.002	<0.002	<0.002	<0.002	
Australia,	plant	SC		71.4	92.7	1	0	0.36	0.028	0.05	0.034	NR
1998,								0.38	0.023	0.042	0.044	
AK99029						1	15	0.23	0.014	0.066	0.047	
Jambin								0.26	0.018	0.085	0.059	
Queensland						1	30	0.13	0.007	0.062	0.012	
								0.14	0.004	0.062	0.005	
Single						1	43	0.06	0.002	0.052	0.003	
applications								0.033	<0.002	0.048	<0.002	
to separate						1	57	0.011	<0.002	0.044	<0.002	
plots								0.008	<0.002	0.032	<0.002	
						1	71	0.003	<0.002	0.024	<0.002	
								0.003	<0.002	0.012	<0.002	
						1	85	<0.002	<0.002	0.008	<0.002	
								<0.002	<0.002	0.005	<0.002	
						1	99	<0.002	<0.002	0.003	<0.002	
								<0.002	<0.002	<0.002	<0.002	
Australia,	gin	SC		71.4	92.7	1	0	0.52	0.024	0.031	0.016	NR
1998,	trash							0.5	0.03	0.038	0.022	
AK99029						1	15	0.26	0.019	0.042	0.03	
Jambin								0.29	0.023	0.06	0.029	
Queensland						1	30	0.13	0.016	0.057	0.01	
								0.16	0.019	0.049	0.014	
Single						1	43	0.036	0.009	0.042	0.003	
applications								0.041	0.012	0.035	0.004	
to separate						1	57	0.02	0.004	0.036	<0.002	
plots								0.013	<0.002	0.027	<0.002	
						1	71	0.007	<0.002	0.03	<0.002	
								0.003	<0.002	0.023	<0.002	
						1	85	<0.002	<0.002	0.012	<0.002	
								<0.002	<0.002	0.008	<0.002	
						1	99	<0.002	<0.002	0.003	<0.002	
								<0.002	<0.002	<0.002	<0.002	
Single foliar treatments, 2 replicates (Lynch, 1999)												
Australia,	plant	SC		25	28.4	1	0	0.062	0.003	0.017	0.018	NR
1998,								0.059	0.004	0.014	0.016	
AK99032						1	15	0.017	<0.002	0.011	0.013	
Wee Waa								0.023	<0.002	0.012	0.013	
NSW						1	29	0.003	<0.002	0.009	0.009	

Country, Year, Reference	Sample	Application					PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	MB 46513	RPA 200766
								0.005	<0.002	0.008	0.01	
Single						1	43	<0.002	<0.002	0.004	<0.002	
applications								<0.002	<0.002	0.003	<0.002	
to separate						1	57	<0.002	<0.002	0.002	<0.002	
plots						1	71	<0.002	<0.002	<0.002	<0.002	
						1	85	<0.002	<0.002	<0.002	<0.002	
						1	99	<0.002	<0.002	<0.002	<0.002	
Australia,	gin	SC		25	28.4	1	0	0.25	0.014	0.026	0.02	NR
1998,	trash							0.26	0.016	0.022	0.017	
AK99032						1	15	0.071	0.009	0.011	0.016	
Wee Waa								0.078	0.003	0.009	0.014	
NSW						1	29	0.008	<0.002	0.009	0.01	
								0.005	<0.002	0.012	0.012	
Single						1	43	0.002	<0.002	0.007	0.003	
applications								<0.002	<0.002	0.006	0.004	
to separate						1	57	<0.002	<0.002	0.002	<0.002	
plots								<0.002	<0.002	<0.002	<0.002	
						1	71	<0.002	<0.002	<0.002	<0.002	
						1	85	<0.002	<0.002	<0.002	<0.002	
						1	99	<0.002	<0.002	<0.002	<0.002	
Australia,	plant	SC		50	56.8	1	0	0.13	0.008	0.016	0.006	NR
1998,								0.019	0.009	0.025	0.015	
AK99032						1	15	0.075	0.004	0.021	0.018	
Wee Waa								0.057	0.005	0.017	0.016	
NSW						1	29	0.007	0.002	0.016	0.011	
								0.004	<0.002	0.012	0.011	
Single						1	43	0.002	<0.002	0.008	0.002	
applications								0.003	<0.002	0.007	<0.002	
to separate						1	57	0.002	<0.002	0.003	<0.002	
plots								<0.002	<0.002	0.002	<0.002	
						1	71	<0.002	<0.002	0.002	<0.002	
						1	85	<0.002	<0.002	<0.002	<0.002	
						1	99	<0.002	<0.002	<0.002	<0.002	
Australia,	gin	SC		50	56.8	1	0	0.45	0.02	0.023	0.025	NR
1998,	trash							0.46	0.02	0.025	0.024	
AK99032						1	15	0.16	0.011	0.2	0.016	
Wee Waa								0.15	0.007	0.18	0.008	
NSW						1	29	0.018	0.002	0.017	0.013	
								0.01	<0.002	0.019	0.016	
Single						1	43	0.007	<0.002	0.013	0.007	
applications								0.005	<0.002	0.01	0.008	
to separate						1	57	0.003	<0.002	0.007	0.003	
plots								0.004	<0.002	0.009	<0.002	
						1	71	0.002	<0.002	0.005	<0.002	
								0.003	<0.002	0.004	<0.002	
						1	85	<0.002	<0.002	0.003	<0.002	
								<0.002	<0.002	<0.002	<0.002	
						1	99	<0.002	<0.002	<0.002	<0.002	
Soil treatment in-furrow at sowing followed by foliar treatments, or foliar only (Norris, 1997a)												
USA, 1995, 95-0023NC	gin trash	WG		168 inc + 84 foliar		1 2	44	<0.04 <0.04	<0.04 <0.04	0.33 0.26	0.48 0.43	<0.05 <0.05
USA, 1995, 95-0023NC	gin trash	WG		84 foliar		4	44	<0.04 <0.1	<0.04 <0.04	0.37 0.41	0.5 0.6	<0.05 <0.05
USA, 1995, 95-0026AR	gin trash	WG		168 inc + 84 foliar		1 2	45	<0.1 <0.1	<0.04 <0.04	0.17 0.21	0.59 0.64	<0.05 <0.05
USA, 1995, 95-0026AR	gin trash	WG		84 foliar		4	45	0.11 <0.1	<0.04 <0.04	0.42 0.43	1.3 1.1	<0.05 <0.05
USA, 1995, 95-0027MS	gin trash	WG		168 inc + 84 foliar		1 2	44	<0.1 <0.1	<0.04 <0.04	<0.1 <0.1	0.23 0.25	<0.05 <0.05
USA, 1995, 95-0027MS	gin trash	WG		84 foliar		4	44	<0.1 <0.1	<0.04 <0.04	0.27 0.19	0.51 0.47	<0.05 <0.05

Country, Year, Reference	Sample	Application					PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	MB 46513	RPA 200766
USA, 1995, 95-0028LA	gin trash	WG		168 inc + 84 foliar		1 2	45	<0.04 <0.04	<0.04 <0.04	0.25 0.29	0.61 0.68	<0.05 <0.05
USA, 1995, 95-0028LA	gin trash	WG		84 fol		4	45	<0.1 <0.1	<0.04 <0.04	0.449 0.435	1.0 1.1	<0.05 <0.05
USA, 1995, 95-0029TX	gin trash	WG		168 inc + 84 foliar		1 2	43	0.11 <0.1	<0.04 <0.04	0.21 0.2	0.17 0.16	<0.05 <0.05
USA, 1995, 95-0029TX	gin trash	WG		84 foliar		4	43	0.12 0.13	<0.1 <0.04	0.37 0.40	0.39 0.41	<0.05 <0.05
USA, 1995, 95-0030TX	gin trash	WG		168 inc + 84 foliar		1 2	44	<0.04	<0.04	<0.05	<0.04	<0.05
USA, 1995, 95-0030TX	gin trash	WG		84 foliar		4	44	<0.04	<0.04	<0.05	<0.04	<0.05
USA, 1995, 95-0031OK	gin trash	WG		168 inc + 84 foliar		1 2	44	<0.04 <0.04	<0.04 <0.04	<0.05 <0.1	<0.04 <0.04	<0.05 <0.05
USA, 1995, 95-0031OK	gin trash	WG		84 foliar		4	44	<0.04	<0.04	<0.05	<0.1	<0.05
USA, 1995, 95-0032TX	gin trash	WG		168 inc + 84 foliar		1 2	46	<0.1 <0.1	<0.04 <0.04	0.17 <0.05	<0.1 <0.1	<0.05 <0.05
USA, 1995, 95-0032TX	gin trash	WG		84 foliar		4	46	<0.1 0.1	<0.04 0.04	<0.05 0.17	<0.1 0.18	<0.05
USA, 1995, 95-0033TX	gin trash	WG		168 inc + 84 foliar		1 2	46	<0.04	<0.04	<0.05	<0.1	<0.05
USA, 1995, 95-0033TX	gin trash	WG		84 foliar		4	46	<0.04	<0.04	<0.05	<0.1	<0.05
USA, 1995, 95-0034AZ	gin trash	WG		168 inc + 84 foliar		1 2	45	0.29 0.31	<0.04 <0.04	0.31 0.30	0.49 0.48	<0.05 <0.05
USA, 1995, 95-0034AZ	gin trash	WG		84 foliar		4	45	0.36 0.4	<0.1 <0.1	0.56 0.6	0.86 0.83	<0.05 <0.05
USA, 1995, 95-0035CA	gin trash	WG		168 inc + 84 foliar		1 2	45	0.57 0.62	0.12 0.13	0.33 0.38	1.7 2.0	<0.05 <0.05
USA, 1995, 95-0035CA	gin trash	WG		84 foliar		4	45	1.4 1.6	0.23 0.25	0.95 1.0	4.4 4.7	<0.05
USA, 1995, 95-0036CA	gin trash	WG		168 inc + 84 foliar		1 2	46	0.52 0.43	0.1 <0.1	0.46 0.37	1.7 1.8	<0.05 <0.05
USA, 1995, 95-0036CA	gin trash	WG		84 foliar		4	46	0.36 0.58	<0.1 0.11	0.24 0.5	1.3 2.4	<0.05
Foliar treatment, 2 replicates (Norris, 1997b)												
USA, 1995, 95-0276GA	gin trash	WG		56		6	43	0.32 0.23	<0.1 <0.1	0.79 0.62	0.89 0.77	<0.05
USA, 1995, 95-0277LA	gin trash	WG		56		6	45	0.11 0.1	<0.04	0.52 0.56	1.0 1.2	<0.05
USA, 1995, 95-0278TX	gin trash	WG		56		6	46	<0.1 <0.1	<0.1 <0.1	0.12 0.11	0.1 0.11	<0.05
USA, 1995, 95-0279TX	gin trash	WG		56		6	44	0.11 0.11	<0.1 <0.1	0.15 0.14	0.18 0.18	<0.05
USA, 1995, 95-0280CA	gin trash	WG		56		6	46	1.6 1.6	0.25 0.25	1.2 1.2	4.8 4.6	<0.05
Foliar treatment, 2 replicates (Norris, 1998)												
USA, 1996, 10669-01NC	gin trash	EC		56		6	43	<0.1 <0.1	<0.1 <0.1	0.37 0.23	0.24 0.15	NR
USA, 1996,	gin	EC		56		6	44	0.13	<0.1	0.67	0.5	NR

Country, Year, Reference	Sample	Application					PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	MB 46513	RPA 200766
10669-02GA	trash							0.2	<0.1	1.1	0.9	
USA, 1996, 10669-03AR	gin trash	EC		56		6	45	<0.1 <0.1	<0.1 <0.1	0.15 0.28	0.11 0.29	NR
USA, 1996, 10669-04AR	gin trash	EC		56		6	46	<0.1 <0.1	<0.1 <0.1	0.22 0.32	0.21 0.31	NR
USA, 1996, 10669-05MS	gin trash	EC		56		6	45	<0.1 <0.1	<0.1 <0.1	0.63 0.3	0.34 0.15	NR
USA, 1996, 10669-06LA	gin trash	EC		56		6	45	<0.1 <0.1	<0.1 <0.1	0.36 0.45	0.45 0.54	NR
USA, 1996, 10669-07TX	gin trash	EC		56		6	47	<0.1 <0.1	<0.04 <0.04	0.55 0.5	0.47 0.4	NR
USA, 1996, 10669-08TX	gin trash	EC		56		6	45	<0.1 0.12	<0.1 <0.1	0.29 0.57	0.19 0.36	NR
USA, 1996, 10669-09OK	gin trash	EC		56		6	45	<0.04 <0.04	<0.04 <0.04	<0.1 <0.05	<0.1 <0.1	NR
USA, 1996, 10669-10TX	gin trash	EC		56		6	47	<0.1 <0.1	<0.04 <0.04	<0.1 <0.1	<0.1 <0.1	NR
USA, 1996, 10669-11TX	gin trash	EC		56		6	46	<0.1 <0.1	<0.04 <0.04	<0.1 <0.1	<0.1 <0.1	NR
USA, 1996, 10669-12AZ	gin trash	EC		56		6	45	0.23 0.34	<0.04 <0.1	0.53 0.80	0.45 0.8	NR
USA, 1996, 10669-13CA	gin trash	EC		56		6	44	0.16 0.27	<0.1 <0.1	0.57 0.97	0.64 1.4	NR
USA, 1996, 10669-14CA	gin trash	EC		56		6	45	0.26 0.19	<0.1 <0.1	1.5 0.96	1.0 0.7	NR
Foliar treatment, GAP pending, 2 replicates (Gough, 1999)												
USA, 1997, 13499-01 Kerman CA	gin trash	EC		56		3	75	<0.04 <0.01	<0.04 <0.004	<0.1 0.11	0.22 0.30	NR
USA, 1997, 13499-01 Kerman CA	gin trash	EC		56		4	75	<0.1 <0.1	<0.04 <0.004	<0.1 0.16	0.12 0.38	NR
USA, 1997, 13499-02 Tulare CA	gin trash	EC		56		3	75	<0.1 <0.1	<0.1 <0.1	0.32 0.29	0.32 0.27	NR
USA, 1997, 13499-02 Tulare CA	gin trash	EC		56		4	75	<0.1 <0.1	<0.1 <0.1	0.22 0.29	0.25 0.34	NR
USA, 1997, 13499-03 Brawley CA	gin trash	EC		56		3	76	<0.1 <0.1	<0.04 <0.04	0.12 <0.1	0.31 0.24	NR
USA, 1997, 13499-03 Brawley CA	gin trash	EC		56		4	76	<0.1 <0.1	<0.04 <0.04	0.14 0.17	0.39 0.45	NR
Foliar treatment, GAP pending, 2 replicates (Macy, 1998)												
USA, 1997, 13501-01AR	gin trash	EC		56		3	58	<0.04 <0.04	<0.04 <0.04	0. 16 0. 11	<0.1 <0.1	NR
USA, 1997, 13501-01AR	gin trash	EC		56		4	58	<0.04 <0.04	<0.04 <0.04	0. 17 0.15	<0.1 <0.1	NR
USA, 1997, 13501-02AR	gin trash	EC		56		3	61	<0.04 <0.04	<0.04 <0.04	0.1 0.21	0.15 <0.1	NR
USA, 1997, 13501-02AR	gin trash	EC		56		4	61	<0.04 <0.04	<0.04 <0.04	0.14 0.13	<0.1 <0.1	NR
USA, 1997, 13501-03LA	gin trash	EC		56		3	60	<0.04	<0.04	<0.1	<0.1	NR
USA, 1997, 13501-03LA	gin trash	EC		56		4	60	<0.04 <0.04	<0.04 <0.04	<0.1 <0.1	<0.1 0.14	NR
USA, 1997, 13501-04LA	gin trash	EC		56		3	61	<0.04	<0.04	<0.1	<0.1	NR
USA, 1997, 13501-04LA	gin trash	EC		56		4	61	<0.04 <0.04	<0.04 <0.04	0.19 0.2	0.15 0.16	NR
USA, 1997, 13501-05MS	gin trash	EC		56		3	61	<0.04 <0.04	<0.04 <0.04	<0.1 0.12	<0.1 <0.1	NR

Country, Year, Reference	Sample	Application					PHI, days	Residues, mg/kg				
		Form	g ai/t	g ai/ha	g ai/hl	No.		Fipronil	MB 45950	MB 46136	MB 46513	RPA 200766
USA, 1997, 13501-05MS	gin trash	EC		56		4	61	<0.04 <0.04	<0.04 <0.04	0.1 0.19	<0.1 0.13	NR
USA, 1997, 13501-06AR	gin trash	EC		56		3	57	<0.04 <0.04	<0.04 <0.04	0.1 <0.1	0.17 0.11	NR
USA, 1997, 13501-06AR	gin trash	EC		56		4	57	<0.04 <0.04	<0.04 <0.04	0.15 0.13	0.16 0.14	NR

NR: not reported
inc: soil incorporation

Table 83. Residues in sunflower forage and straw from seed treatment (each trial with 2 replicates), Australia, all single applications.

Year, Reference	Application			PHI, days	Residues, mg/kg				
	Sample	Form	g ai/ ton		Fipronil	MB 45950	MB 46136	fipronil- desulfinyl	RPA 200766
(Keats, 1998l)									
1997, AK 98032, 96i38gR	forage	FS	750 1500	36	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
	straw		750 1500	113	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	
(Keats, 1998k)									
1998, AK 98035, 96i38bR	forage	FS	750 1500	40	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
	straw		750 1500	144	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	
(Keats, 1998i)									
1998, AK 98033, 97i004a	forage	FS	750 1500	53	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	0.002 <0.002	NR
	straw		750 1500	138	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	
(Keats, 1998j)									
1998, AK 98034, 97i004b	forage	FS	750 1500	22	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
	straw		750 1500	112	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	

NR: not reported

Table 84. Residues in sugar cane leaves resulting from soil and foliar applications in Australia.

Year, Reference, Location	Application			PHI, days	Residues, mg/kg				
	Form	g ai/ha	No.		Fipronil	MB 45950	MB 46136	fipronil- desulfinyl	RPA 200766
Foliar spray, 2 replicates (Keats, 1997m)									
1995, AUS94i74r Mowilyan Queensland	WG	100	1	181	<0.002 0.002	<0.002 <0.002	<0.002 <0.002	<0.002 <0.002	NR
1995, AUS94i74r Mowilyan Queensland	WG	200	1	181	0.002 0.002	<0.002 <0.002	<0.002 <0.002	0.003 0.002	NR
1995, AUS94i74r Mowilyan Queensland	WG	50	2	134	0.002 0.002	<0.002 <0.002	<0.002 <0.002	0.002 0.002	NR
1995, AUS94i74r Mowilyan Queensland	WG	100	2	53	0.005 0.007	<0.002 <0.002	<0.002 <0.002	0.002 0.002	NR
Soil treatment and stool spray, 2 replicates (Keats, 1997k)									
1996, AUS94i74cr Kurrimine Beach Queensland	WG + SC	100 soil 50 foliar	1 + 2	95	<0.002	<0.002	<0.002	<0.002	NR

Year, Reference, Location	Application			PHI, days	Residues, mg/kg				
	Form	g ai/ha	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
1996, AUS94i74cr Kurrimine Beach Queensland	WG + SC	200 soil 100 foliar	1 + 2	95	<0.002	<0.002	<0.002	<0.002	NR
1996, AUS94i74cr Kurrimine Beach Queensland	WG + SC	400 soil 200 foliar	1 + 2	95	<0.002	<0.002	<0.002	<0.002	NR
Foliar application, 2 replicates (Keats, 1997k)									
1997, 97NST14 Kurrimine Beach Queensland	SC	75	1	101	<0.002	<0.002	<0.002	<0.002	NR
Foliar application to base on cane stalks, 2 replicates (Keats, 1997c)									
1996, AUS94i74br Rocky Point Queensland	WG	50	2	0	0.11	0.003	0.005	0.002	NR
					0.11	0.004	0.005	0.002	
				1	0.22	0.009	0.02	0.036	
					0.18	0.008	0.014	0.032	
				3	0.093	0.004	0.009	0.016	
					0.096	0.004	0.011	0.016	
				5	0.12	0.004	0.008	0.007	
					0.13	0.004	0.008	0.006	
				7	0.058	0.003	0.008	0.013	
					0.061	0.003	0.009	0.012	
				14	0.01	<0.002	0.004	0.006	
					0.009	<0.002	0.004	0.005	
				26	0.015	<0.002	0.004	0.003	
					0.014	<0.002	0.004	0.003	
1996, AUS94i74br Rocky Point Queensland	WG	100	2	0	0.34	0.008	0.012	0.005	NR
					0.33	0.008	0.014	0.005	
				1	0.26	0.007	0.016	0.016	
					0.23	0.007	0.015	0.015	
				3	0.21	0.01	0.048	0.059	
					0.2	0.01	0.048	0.059	
				5	0.15	0.007	0.023	0.033	
					0.17	0.008	0.023	0.035	
				7	0.15	0.004	0.012	0.014	
					0.14	0.005	0.013	0.016	
				14	0.097	0.004	0.01	0.014	
					0.099	0.004	0.01	0.015	
				26	0.079	0.005	0.019	0.026	
					0.058	0.004	0.015	0.017	
1996, AUS94i74br Rocky Point Queensland	WG	100	1	0	0.7	0.012	0.021	0.006	NR
					0.75	0.012	0.022	0.005	
				1	0.54	0.01	0.025	0.013	
					0.5	0.011	0.02	0.012	
				3	0.46	0.019	0.061	0.035	
					0.57	0.018	0.058	0.028	
				5	0.19	0.01	0.029	0.015	
					0.2	0.01	0.032	0.02	
				7	0.075	0.003	0.009	0.008	
					0.067	0.002	0.007	0.009	
				14	0.038	0.002	0.007	0.003	
					0.029	0.002	0.007	0.003	
				26	0.018	0.002	0.007	0.003	
					0.016	0.002	0.006	0.003	

Year, Reference, Location	Application			PHI, days	Residues, mg/kg				
	Form	g ai/ha	No.		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
1996, AUS94i74br	WG	200	1	0	0.86	0.014	0.026	0.006	NR
Rocky Point					0.94	0.016	0.025	0.005	
Queensland				1	1.1	0.023	0.053	0.056	
					1.2	0.023	0.054	0.056	
				3	0.51	0.015	0.039	0.062	
					0.5	0.014	0.038	0.059	
				5	0.39	0.017	0.061	0.098	
					0.36	0.016	0.063	0.103	
				7	0.32	0.015	0.046	0.093	
					0.32	0.017	0.046	0.091	
				14	0.19	0.007	0.02	0.031	
					0.17	0.007	0.017	0.028	
				26	0.085	0.004	0.016	0.033	
					0.072	0.005	0.015	0.026	

NR: not reported

Animal feeding studies

<u>Cows</u>. Three feeding studies were reported on the transfer of residues of fipronil into meat, animal fat, meat by-products and milk, and a further study for residues of fipronil-desulfinyl in lactating animals.

In the first study (Byrd, 1994a) groups of three lactating cows were dosed daily with fipronil by bolus at a rate equivalent to 0.04, 0.13 or 0.43 ppm in the diet for 35 days. Milk was collected twice daily. Equal samples of morning and evening milk were combined and analysed for fipronil, MB 45950 and MB 46136. Fipronil-derived residues reached a plateau after 25 days at the high-dose, and consisted almost entirely of MB 46136. MB 45950 was detected in a single milk sample and trace amounts of fipronil (<0.01 mg/kg) at the high dose. The results are shown in Table 85.

Table 85. Residues in milk, mg/kg (Byrd, 1994a).

Day	0.04 ppm dose			0.13 ppm dose			0.43 ppm dose		
	fipronil	MB 45950	MB 46136	fipronil	MB 45950	MB 46136	fipronil	MB 45950	MB 46136
0	ND (3)[1]	ND (3)	ND (3)	ND (3)	ND (3)	ND (3)	ND (3)	ND (3)	ND (3)
1	ND (3)	ND (3)	ND (3)	ND (3)	ND (3)	<0.01 (3)[2]	<0.01 (3)	ND (3)	<0.01 (3)
3	ND (3)	ND (3)	<0.01 (3)	ND ND <0.01	ND ND ND	<0.01 <0.01 <0.01	<0.01 (3)	ND (3)	<0.01 (3)
7	ND ND ND	ND ND ND	<0.01 ND <0.01	ND (3)	ND (3)	<0.01 (3)	<0.01 <0.01 <0.01	ND ND <0.01	0.014 0.013 0.023
12	ND (3)	ND (3)	<0.01 (3)	ND (3)	ND (3)	0.013 <0.01 <0.01	<0.01 <0.01 <0.01	ND ND ND	0.025 0.019 0.025
15	ND (3)	ND (3)	<0.01 (3)	ND (3)	ND (3)	0.016 <0.01 <0.01	<0.01 <0.01 <0.01	ND ND ND	0.024 0.025 0.027
20	ND (3)	ND (3)	<0.01 (3)	ND (3)	ND (3)	0.014 <0.01 0.01	<0.01 <0.01 <0.01	ND ND ND	0.025 0.026 0.029
25	ND (3)	ND (3)	<0.01 (3)	ND (3)	ND (3)	0.012 <0.01 0.013	<0.01 <0.01 <0.01	ND ND ND	0.030 0.024 0.040
29	ND (3)	ND (3)	<0.01 (3)	ND (3)	ND (3)	0.012 <0.01 0.012	ND <0.01 ND	ND ND ND	0.030 0.038 0.052
34	ND (3)	ND (3)	<0.01 (3)	ND (3)	ND (3)	0.018 <0.01 0.013	<0.01 <0.01 <0.01	ND ND ND	0.035 0.032 0.041

[1] ND: not detectable [2] <0.01: residue lower than the LOQ, traces detected

After 35 days the cows were slaughtered and the liver, kidney, fat and muscle were collected for analysis in triplicate from each animal. Fat contained the highest concentration of fipronil residues, but only at levels slightly above the LOQ in the high-dose group. Most of the residue was MB 46136. The results are shown in Table 86.

Table 86. Residues in cow tissues, mg/kg (Byrd, 1994a).

Sample	0.04 ppm group			0.13 ppm group			0.43 ppm group		
	fipronil	MB 45950	MB 46136	Fipronil	MB 45950	MB 46136	Fipronil	MB 45950	MB 46136
Muscle	ND[1]	ND	<0.01[2]	ND	ND	0.01	ND	ND	ND
	ND	ND	<0.01	ND	ND	0.015	ND	ND	ND
	ND	ND	<0.01	ND	ND	0.01	ND	ND	ND
Liver	ND	ND	0.013	ND	ND	0.061	ND	ND	0.16
	ND	ND	0.011	ND	ND	0.039	ND	ND	0.122
	ND	ND	0.012	ND	ND	0.046	ND	ND	0.117
Kidney	ND	ND	<0.01	ND	ND	0.014	<0.01	ND	0.034
	ND	ND	<0.01	<0.01	ND	<0.01	<0.01	ND	0.027
	ND	ND	<0.01	<0.01	ND	0.01	<0.01	ND	0.027
Fat	<0.01	ND	0.046	<0.01	<0.01	0.218	0.042	<0.01	0.546
	<0.01	ND	0.036	<0.01	<0.01	0.134	0.031	<0.01	0.446
	<0.01	ND	0.063	<0.01	<0.01	0.146	0.026	<0.01	0.413

[1]ND: not detectable
[2] <0.01: residue lower than the LOQ, traces detected

In a second study (Tew, 1999) two cows were dosed with fipronil at a rate equivalent to 1 ppm in the feed (dry weight basis) for 20 days. Milk samples were collected on 4 non-consecutive days before and on the 1st, 2nd, 4th, 7th and 19th days after the last dose. Milk fat was separated from whole milk collected on the last day of dosing by centrifuging. Whole milk and milk fat samples were analysed for residues of fipronil, MB 46136 and MB 45950 to determine the rate at which residues decreased in whole milk and the degree to which fipronil was concentrated in the fat. Analysis of samples from days 14-20 of dosing confirmed that residues had reached a plateau, and throughout the study consisted almost entirely of the metabolite MB 46136. The mean plateau residue of days 14,16, 18 and 20 was 0.033 mg/kg). No measurable residues of fipronil or MB 45950 (LOQ 0.003 mg/kg) were found in any whole milk samples.

Residues in the milk decreased slowly at the end of dosing. After one week MB 46136 residues were approximately 66% of their plateau level. Samples 19 days after the last dose had decreased to near the LOQ with average MB 46136 residues of 0.004 mg/kg. The calculated half-life of fipronil was 5.2 days.

The residues in milk fat samples were compared to those in day 20 whole milk. Similarly, residues consisted mainly of MB 46136 but because the residue was concentrated, minor amounts of MB 45950 (0.006 mg/kg) were detected. Average total residues as MB 46136 equivalents in whole milk and milk fat from day 20 were 0.039 mg/kg and 0.552 mg/kg respectively, indicating a concentration factor of 14.2.

In a feeding study by Keats (1998) in Australia to determine maximum residues in rangeland cattle oversprayed and then fed on fipronil-treated pasture grasses and the rate at which the residues decreased in various edible tissues, 32 cows were divided into 10 groups of 3 animals (groups 1-10), plus two control animals. On day 1, groups 1-10 were sprayed once dermally at the proposed commercial rate of 2.5 g ai/ha and the total applied to each was 0.75 mg as fipronil, calculated on an average hide area of 3 m^2. The treated cows were then dosed orally once a day with fipronil for up to 14 days. Animals from groups 1-7 and groups 8-10 were dosed with capsules at rates to give dietary exposures corresponding to twice the commercial rate (5 g ai/ha) and the commercial rate of 2.5 g ai/ha respectively. The fipronil concentration in the capsules decreased from day 1 to 14, corresponding to the fipronil levels found in Australian pasture decline studies. The doses were as follows:

Day	**Fipronil/capsule (mg)**	
	Groups 8-10	Groups 1-7
1	2.5	5.0
2	1.9	3.9
3	1.4	2.7
4	0.8	1.5
5	0.7	1.5
6	0.7	1.4
7	0.6	1.1
8	0.4	0.8
9	0.4	0.7
10	0.3	0.6
11	0.3	0.5
12	0.2	0.4
13	0.2	0.4
14	0.1	0.3

The details of dosing are shown in Table 87. Fipronil, MB 45950 and MB 46136 were measured in renal, subcutaneous and abdominal fat, loin, round and diaphragm muscle, kidney and liver at 3 time points through the 14 days (3 animals per time point) and at 4 time points through the depuration period (again 3 animals per time point) at the double rate, and twice during uptake and once during depuration at the lower rate. The samples were stored at ≤-20°C and analysed within 2 months.

Table 87. Details of dosing in rangeland cattle study (Keats, 1998).

Group (3 animals/group)	Dermal dose (mg/animal)	Days dosed orally	Oral dose (mg/animal)	Pre-slaughter depuration (days)
1	0.75	5	14.6	0
2	0.75	10	19.2	0
3	0.75	14	20.8	0
4	0.75	14	20.8	6
5	0.75	14	20.8	13
6	0.75	14	20.8	20
7	0.75	14	20.8	27
8	0.75	10	9.7	0
9	0.75	14	10.5	0
10	0.75	14	10.5	13
Control	0	0	0	NA

Samples from the control animal contained no quantifiable residues. No residues of fipronil-desulfinyl were found, indicating that the photodegradation product, if formed on the animal after over-spray, will not be absorbed to any measurable extent. The highest residues were in the fat followed by liver in all dose groups, with very little residue transferred to muscles or kidneys (Tables 88-89).

Table 88. Residues in cattle fat, mg/kg (Keats, 1998).

Group	Renal fat			Abdominal fat			Subcutaneous fat		
	fipronil	MB 45950	MB 46136	fipronil	MB 45950	MB 46136	fipronil	MB 45950	MB 46136
Double dose									
1	0.027	0.008	0.078	0.024	0.007	0.061	0.026	0.005	0.064
	0.028	0.008	0.092	0.029	0.009	0.091	0.030	0.009	0.091
	0.035	0.013	0.059	0.029	0.009	0.044	0.026	0.009	0.050
2	0.018	0.004	0.078	0.021	0.005	0.083	0.021	0.003	0.08
	0.022	0.007	0.097	0.021	0.007	0.09	0.012	0.002	0.045
	0.02	0.005	0.11	0.014	0.003	0.081	0.017	0.003	0.088

Group	Renal fat			Abdominal fat			Subcutaneous fat		
	fipronil	MB 45950	MB 46136	fipronil	MB 45950	MB 46136	fipronil	MB 45950	MB 46136
3	0.009	0.005	0.154	0.009	0.005	0.145	0.01	0.004	0.156
	0.009	0.003	0.109	0.009	0.003	0.111	0.009	0.002	0.128
	0.012	0.002	0.069	0.012	0.002	0.068	0.011	0.002	0.048
4	0.004	0.005	0.067	0.004	0.004	0.065	0.006	0.006	0.065
	0.002	0.002	0.068	0.006	0.002	0.064	0.008	0.002	0.072
	0.003	0.002	0.077	0.002	0.002	0.079	0.006	0.002	0.067
5	<0.002	0.002	0.089	0.002	0.002	0.087	0.003	0.003	0.094
	0.003	0.002	0.084	0.003	0.002	0.071	0.004	0.002	0.057
	0.002	<0.002	0.045	<0.002	<0.002	0.033	0.003	<0.002	0.046
6	0.003	<0.002	0.034	<0.002	<0.002	0.040	<0.002	<0.002	0.033
	<0.002	<0.002	0.031	<0.002	<0.002	0.020	<0.002	<0.002	0.033
	<0.002	<0.002	0.059	0.002	<0.002	0.055	<0.002	<0.002	0.069
7	<0.002	<0.002	0.023	<0.002	<0.002	0.028	<0.002	<0.002	0.031
	<0.002	<0.002	0.08	<0.002	<0.002	0.063	0.002	<0.002	0.07
	0.002	<0.002	0.037	<0.002	<0.002	0.035	0.002	0.002	0.048
Single dose									
8	0.011	0.005	0.085	0.009	0.002	0.08	0.006	<0.002	0.051
	0.01	0.004	0.05	0.01	0.004	0.045	0.011	0.003	0.04
	0.01	0.006	0.062	0.011	0.007	0.064	0.012	0.006	0.08
9	0.008	0.004	0.066	0.007	0.002	0.057	0.007	<0.002	0.056
	0.01	0.005	0.085	0.01	0.005	0.070	0.008	0.002	0.066
	0.004	<0.002	0.034	0.005	<0.002	0.041	0.007	<0.002	0.052
10	<0.002	<0.002	0.033	<0.002	<0.002	0.030	<0.002	<0.002	0.026
	<0.002	<0.002	0.026	<0.002	<0.002	0.026	<0.002	<0.002	0.03
	<0.002	<0.002	0.036	<0.002	<0.002	0.034	0.002	<0.002	0.03

Table 89. Residues in cattle tissues, mg/kg (Keats, 1998).

Group	Diaphragm muscle			Loin and round muscle			Liver			Kidney		
	fipronil	MB 45950	MB 46136	fipronil	MB 45950	MB 46136	fipronil	MB 45950	MB 46136	fipronil	MB 45950	MB 46136
Double dose												
1	0.004	<0.002	0.011	<0.002	<0.002	0.002	<0.002	<0.002	0.018	0.002	<0.002	0.006
	<0.002	<0.002	0.011	<0.002	<0.002	0.002	<0.002	<0.002	0.01	0.003	<0.002	0.003
	0.003	0.002	0.004	<0.002	<0.002	<0.002	<0.002	<0.002	0.005	<0.002	<0.002	0.003
2	0.002	<0.002	0.006	<0.002	<0.002	<0.002	<0.002	<0.002	0.004	0.002	<0.002	0.003
	0.003	<0.002	0.006	<0.002	<0.002	0.002	<0.002	<0.002	<0.002	0.002	<0.002	0.002
	<0.002	<0.002	0.005	<0.002	<0.002	0.002	0.002	<0.002	0.003	<0.002	<0.002	0.002
3	<0.002	<0.002	0.002	<0.002	<0.002	<0.002	<0.002	<0.002	0.01	<0.002	<0.002	0.003
	<0.002	<0.002	0.003	<0.002	<0.002	<0.002	<0.002	<0.002	0.006	<0.002	<0.002	0.004
	<0.002	<0.002	0.002	<0.002	<0.002	0.002	<0.002	<0.002	0.002	<0.002	<0.002	<0.002
4	<0.002	<0.002	0.007	<0.002	<0.002	0.002	<0.002	<0.002	0.005	<0.002	<0.002	0.005
	<0.002	<0.002	0.004	<0.002	<0.002	<0.002	0.002	<0.002	0.006	<0.002	<0.002	0.006
	<0.002	<0.002	0.006	<0.002	<0.002	0.002	<0.002	<0.002	0.007	<0.002	<0.002	0.002
5	<0.002	<0.002	0.004	<0.002	<0.002	<0.002	<0.002	<0.002	0.020	<0.002	<0.002	0.006
	<0.002	<0.002	<0.002	<0.002	<0.002	0.003	<0.002	<0.002	0.01	0.002	<0.002	0.005
	<0.002	<0.002	0.002	<0.002	<0.002	0.002	<0.002	<0.002	0.005	<0.002	<0.002	0.003
6	<0.002	<0.002	0.002	<0.002	<0.002	<0.002	<0.002	<0.002	0.002	<0.002	<0.002	<0.002
	<0.002	<0.002	0.003	<0.002	<0.002	0.003	0.002	<0.002	0.002	<0.002	<0.002	<0.002
	<0.002	<0.002	0.003	<0.002	<0.002	0.002	<0.002	<0.002	0.005	<0.002	<0.002	0.002
7	<0.002	<0.002	0.002	<0.002	<0.002	<0.002	<0.002	<0.002	0.002	<0.002	<0.002	<0.002
	<0.002	<0.002	0.002	<0.002	<0.002	<0.002	<0.002	<0.002	0.002	0.002	<0.002	0.002
	<0.002	<0.002	0.002	<0.002	<0.002	<0.002	<0.002	<0.002	0.002	<0.002	<0.002	0.002
Single Dose												
8	<0.002	<0.002	0.003	<0.002	<0.002	0.002	<0.002	<0.002	0.003	<0.002	<0.002	0.002
	<0.002	<0.002	0.003	<0.002	<0.002	0.002	<0.002	<0.002	0.004	<0.002	<0.002	0.002
	0.002	<0.002	0.003	<0.002	<0.002	0.002	<0.002	<0.002	0.003	<0.002	<0.002	0.002
9	<0.002	<0.002	0.003	<0.002	<0.002	0.002	<0.002	<0.002	0.003	<0.002	0.002	0.002
	<0.002	<0.002	0.003	<0.002	<0.002	0.002	<0.002	<0.002	0.003	<0.002	<0.002	0.002
	<0.002	<0.002	0.002	<0.002	<0.002	0.002	<0.002	<0.002	0.003	<0.002	<0.002	0.002
10	<0.002	<0.002	0.002	<0.002	<0.002	0.002	<0.002	<0.002	0.006	<0.002	<0.002	0.002
	<0.002	<0.002	<0.002	<0.002	<0.002	0.002	<0.002	<0.002	0.002	<0.002	<0.002	0.002
	<0.002	<0.002	0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	0.003

In a study by Williams (1997) groups of three lactating cows were dosed daily with fipronil-desulfinyl by bolus at a rate equivalent to 0.025, 0.075, 0.3 or 1 ppm in the diet for 35 days. An additional animal was administered the highest dose and depurated for 7 days after the 35 days. Milk was collected twice daily on 10 days during the dosing period at pre-determined times and on days 37, 39 and 42 from the single depuration animal. Equal portions of morning and evening milk samples were combined and analysed for fipronil-desulfinyl. Milk fat samples were prepared from day 35 high-dose samples for analysis.

Fipronil-desulfinyl reached a plateau in milk in the 15- to 20-day interval, and the concentration of the analyte paralleled the administered dose in all but the high-dose group (Table 90). fipronil-desulfinyl residues were mainly in the milk fat rather than the skimmed milk, by a factor of about 16.

Table 90. Plateau residues of fipronil-desulfinyl in whole milk (Williams, 1997).

Group	Measured fipronil-desulfinyl dietary level (ppm)	fipronil-desulfinyl mean plateau concentration (mg/kg)	fipronil-desulfinyl range at day 35 (mg/kg)
1	0	<0.002	Not detected
2	0.025	0.003	0.003-0.005
3	0.076	0.008	0.009-0.01
4	0.31	0.027	0.026-0.031
5[1]	1.03	0.058	0.046-0.072
5 (d)	1.03	0.072 at day 35	0.027 at day 42[2]

[1] average of 4 animals
[2] milk level in 1 high-dose animal after 7 days of depuration

After 35 days of dosing, all treated cows except the depuration animal were slaughtered and the liver, kidney, fat and muscle collected for analysis. The depuration cow was slaughtered and sampled 7 days later. The results reported are the means of 2 analyses. Of the tissues examined, fat and liver contained significant levels of fipronil-desulfinyl and muscle and kidney very little (Table 91). The tissue levels in the depurated animal, in conjunction with the milk residues, support a half-life value for MB 46136 in lactating cows of somewhat less than one week.

Table 91. Fipronil-desulfinyl residues in the tissues of lactating cows, mg/kg (Williams, 1997).

Sample	0.025 ppm group	0.076 ppm group	0.3 ppm group	1.03 ppm group[1]	1.03 ppm (dep)[2]
Muscle	<0.002	0.005	0.015	0.028	0.018
	<0.002	0.003	0.011	0.035	
	0.003	0.003	0.019	0.037	
Liver	0.038	0.094	0.27	0.61	0.25
	0.036	0.069	0.25	0.49	
	0.033	0.098	0.28	0.59	
Kidney	0.004	0.013	0.044	0.12	0.052
	0.005	0.012	0.030	0.094	
	0.006	0.01	0.041	0.093	
Fat	0.039	0.12	0.41	1.06	0.48
	0.039	0.091	0.35	0.79	
	0.043	0.094	0.33	1.07	

[1]excluding the depurated cow
[2]depurated cow

Hens. Byrd (1994b) dosed laying hens, ten per dose group, daily by bolus at rates equivalent to 0.01 ppm, 0.031 ppm, or 0.103 ppm in the diet for 42 days. Eggs were collected throughout and residues reached a plateau after approximately 15 days. The hens were killed after the last doses and liver, skin with adhering fat and muscle were collected. The eggs and tissues were analysed for fipronil, MB 46136 and MB 45950.

Average MB 46136 residues in eggs in the medium- and high-dose groups reached a plateau at about 25-28 days. Those from the low-dose group contained ≤0.01 mg/kg throughout, except from one bird at 25 days (0.013 mg/kg). No MB 45950 was observed in eggs from any group and only trace amounts of fipronil (<0.01 mg/kg) in the high-dose group (Table 92).

Table 92. Residues in eggs, mg/kg (Byrd, 1994b).

Day	0.01 ppm dose			0.031 ppm dose			0.103 ppm dose		
	fipronil	MB 45950	MB 46136	fipronil	MB 45950	MB 46136	fipronil	MB 45950	MB 46136
0	ND (3) [1]	ND (3)	ND (3)	ND (3)	ND (3)	ND (3)	ND (3)	ND (3)	ND (3)
1	ND (3)	ND (3)	ND (3)	ND (3)	ND (3)	ND (3)	ND (3)	ND (3)	ND (3)
3	ND (3)	ND (3)	<0.01 (3)	ND (3)	ND (3)	ND (3)	ND (3)	ND (3)	<0.01 (3) [2]
7	ND (3)	ND (3)	<0.01 (3)	ND (3)	ND (3)	<0.01 (3)	<0.01 (3)	ND (3)	0.033 0.025 0.026
12	ND (3)	ND (3)	<0.01 (3)	ND (3)	ND (3)	0.013 0.01 0.013	<0.01 (3)	ND (3)	0.049 0.040 0.039
15	ND (3)	ND (3)	<0.01 (3)	ND (2)	ND (2)	0.018 0.019	<0.01 (3)	ND (3)	0.051 0.045 0.042
20	ND (3)	ND (3)	<0.01 (3)	ND (3)	ND (3)	0.019 0.02 0.015	<0.01 (3)	ND (3)	0.102 0.081 0.091
25	ND (3)	ND (3)	<0.01 0.013 <0.01	ND (3)	ND (3)	0.023 0.021 0.022	<0.01 (3)	ND (3)	0.116 0.092 0.096
28	ND (3)	ND (3)	<0.01 0.01 <0.01	ND (3)	ND (3)	0.029 0.027 0.03	<0.01 (3)	ND (3)	0.115 0.077 0.083
34	ND (3)	ND (3)	<0.01 0.012 <0.01	ND (3)	ND (3)	0.036 0.017 0.02	<0.01 (3)	ND (3)	0.097 0.092 0.087
41	ND (3)	ND (3)	<0.01 (3)	ND (3)	ND (3)	0.021 0.029 0.023	<0.01 (3)	ND (3)	0.112 0.086 0.09

[1]ND: not detectable
[2] <0.01: residue lower than the LOQ, traces detected

Residues of MB 46136 in the low-dose group were at or below 0.01 mg/kg in muscle and liver and 0.013 mg/kg in skin with adhering fat. At all doses MB 46136 was present at much higher levels in the skin with adhering fat than in the other tissues. The total residue in the fat was almost entirely MB 46136 with fipronil constituting less than 10% in the high-dose group (Table 93).

Table 93. Residues in the tissues of laying hens, mg/kg (Byrd, 1994b).

Sample	0.01 ppm group			0.031 ppm group			0.103 ppm group		
	fipronil	45950	46136	fipronil	45950	46136	fipronil	45950	46136
Muscle	ND (3) [1]	ND (3)	<0.01 (3)	ND (3)	ND (3)	<0.01 (3) [2]	ND (3)	ND (3)	0.014 0.012 0.01
Liver	ND (3)	ND (3)	<0.01 (3)	<0.01 ND ND	ND (3)	0.019 0.020 0.020	<0.01 (3)	ND (3)	0.071 0.067 0.069
Skin with fat	ND (3)	ND (3)	0.013 0.013 0.014	ND <0.01 <0.01	ND ND <0.01	0.046 0.057 0.060	<0.01 (3)	ND (3)	0.161 0.208 0.204

[1]ND: not detectable
[2] <0.01: residue lower than the LOQ, traces detected

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No information.

In processing

Potatoes (Tables 94-95). In a processing study by Macy (1997) potatoes in Washington state, USA, were foliar-sprayed four times with a 200 g/l SC formulation at an exaggerated rate of 280 g ai/ha/application (total rate 1120 g ai/ha) and the tubers harvested 28 days after the last application. All potatoes were washed and inspected for culls.

For chips, potatoes were peeled using an abrasive peeler, trimmed by hand, sliced into chips and placed in warm water. Slices were fried in a restaurant-style deep-fat fryer and drained.
For flakes, potatoes were steam-peeled, scrubbed to remove skins, hand trimmed, cut into 12 mm slices and then washed. Slices were then cooked in a pre-cooker, culled and cooked in a steam cooker. The cooked potatoes were mashed, mixed with additives, dried and broken into flakes.

The peels removed in the flake process were hydraulically pressed and mixed with cut trim waste to make up wet peel for analysis.

Table 94 shows the results (mean of 2 analysed samples).

Table 94. Residues in potatoes and their processed fractions (Macy, 1997).

Location,, Year, Reference	Application			PHI,	Commodity	Residues, mg/kg				
	Form	g ai/ha	No.	days		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
USA (WA), 1996 96V11660[1]	SC	280	4	28	Tuber	0.003	ND	<0.003	0.003	NR
					Chips	<0.003	ND (2)	ND (2)	ND (2)	NR
					Flakes	<0.002	ND (2)	<0.003 (2)	ND (2)	NR
					Wet peel	0.03	0.009	0.008	0.024	NR

[1] <0.003 means residues were detected at >MLD but <LOQ
ND: not detectable (<MLD)
NR: not reported

In another US processing study Macy (1996) sprayed potatoes in Washington state in furrow at 1120 g ai/ha and then 4 times foliarly at 280 g ai/ha/application (total rate applied 2240 g ai/ha, at highly exaggerated rates for both application methods) with an 800 g/kg WG formulation. Tubers were harvested 28 days after the last application. The same processing procedures as above were used. The results are shown in Table 95 (mean of 2 analysed samples).

Table 95. Residues in potatoes and their processed fractions, Washington state, USA, 1996 (Macy, 1996).

Reference	Application			PHI,	Commodity	Residues, mg/kg				
	Form	g ai/ha	No.	days		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
US95V03R	WG	1120 soil, 280 foliar	4	28	tuber	0.034	0.003	0.008	0.011	NR
					chips	0.003	ND	0.008	ND	NR
					flakes	0.011	0.001	0.002	0.001	NR
					wet peel	0.252	0.019	0.084	0.035	NR

ND: not detectable
NR: not reported

In two studies of 8 trials each (Maestracci, 1998; Yslan and Baudet, 1999) on potatoes in Germany 4 foliar applications of WG formulation were made at a seasonal rate corresponding to 80 g ai/ha, with a 28-day PHI. Tuber samples were harvested with attached soil that was removed by washing in cold tap water. Tubers were dried with paper towels and peeled. The wet peel and peeled tubers were frozen separately for subsequent analysis. Residues of the parent compound and the metabolites in all unpeeled tuber samples were below the LOQ, and were undetectable in peeled potatoes.

Rice. A rice processing study was planned in the USA but after a fivefold application rate there were no residues above the LOQ of 0.01 mg/kg in rice grain samples so the processed products were not analysed (Mede, 1996a).

Maize (Table 96). In a maize processing study by Kowite (1993b) in the USA single in-furrow applications of 20 g/kg granules at an exaggerated application rate of 2912 g ai/ha (20 times the US label rate) were made at planting. At harvest, triplicate samples were taken from treated and control plots for use in dry and wet milling processes. The procedures are outlined below.

- *Dry milling:* The moisture content of the grain was between 20 and 28% by weight. The maize samples were dried and cleaned by aspiration and screening. The light impurities from aspiration were classified as grain dust. The cleaned grain was moisture-adjusted and impact-milled to produce hull, grits, meal, flour and germ. The germ was heat-conditioned, flaked and pressed in an expeller to liberate most of the crude oil. The residual crude oil in the solid material (presscake) exiting the expeller was later extracted with hexane. The solvent was removed from the presscake and the crude oils recovered from the expeller and solvent extraction were combined and refined.

- *Wet milling:* The dried and cleaned grain was steeped in water and then milled to recover germ, hull, coarse gluten-starch, gluten and starch in that order. After drying, the processing was completed as before.

Validation of the method with fortification levels of 0.01 mg/kg for each compound and commodity, except starch (0.02 mg/kg), gave recoveries of >75%. The results are shown in Table 96. Undetectable residues are recorded as less than the reported minimum limit of detection (MLD); actual values between the MLD and LOQ (0.01 for all samples, except starch 0.02 mg/kg) are reported as found.

Table 96. Residues in maize and its processed fractions, Nebraska, USA, 1992. Ref. 92-059 (Kowite, 1993b).

Application			PHI, days	Commodity	Residues, mg/kg				
Form	g ai/ha	No.			Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
GR	2912	1	179	Whole seed Dry milling	3<0.002	3<0.002	3<0.003	3<0.002	3<0.003
				Grit Dry milling	3<0.002	3<0.002	3<0.003	3<0.002	3<0.003
				Meal Dry milling	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.003 <0.003 <0.003	**0.004**[1] **0.01**[1] **0.005**[1] **UTC: 0.007**	<0.003 <0.003 <0.003
				Flour Dry milling	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.003 <0.003 <0.003	**0.002**[2] **0.018**[2] <0.002 **UTC: <0.002**	<0.003 <0.003 <0.003
				Crude oil Dry milling	<0.002 **0.002**[1] <0.002 **UTC: 0.005**	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.003 <0.003 <0.003	<0.004 <0.004 <0.004
				Refined oil Dry milling	**0.002**[1] **0.003**[1] **0.002**[1] **UTC: 0.005**	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.003 <0.003 <0.003	**0.03**[1] **0.005**[1] **0.0007**[1] **UTC: 0.027**
				Grain dust Dry milling	3<0.002	3<0.002	3<0.003	3<0.002	3<0.003
				Whole seed Wet milling	3<0.002	3<0.002	3<0.003	3<0.002	3<0.003
				Starch Wet milling	3<0.004	3<0.003	3<0.004	3<0.003	3<0.006
				Crude oil Wet milling	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.002 <0.002 <0.002	<0.003 <0.003 <0.003	**0.006** **0.005** **0.004** **UTC: <0.004**
				Refined oil Wet milling	**0.003** <0.002 <0.002 **UTC: <0.002**	<0.002 <0.002 <0.002	<0.003 <0.003 <0.003	<0.003 <0.003 <0.003	**0.016** **0.006** **0.018** **UTC: <0.004**
				Grain dust Wet milling	**0.004** **0.004** **0.003** **UTC: <0.002**	<0.002 <0.002 <0.002	**0.004** **0.004** **<0.003** **UTC: <0.003**	<0.002 <0.002 <0.002	<0.003 <0.003 <0.003

[1] Stated to be contamination
[2] Thought to be contamination (variability of the results and improbability of photolytic reaction occurring during processing make the findings suspect)
UTC: untreated control sample

Cotton seed (Table 97). In a processing study in the USA by Norris (1995) duplicate plots were treated with an in-furrow spray at 1680 g ai/ha followed by 4 foliar applications each at 420 g ai/ha with 800 WG formulation (5-10 times normal rates for both foliar and soil applications). The cotton was harvested 45 days after the last foliar application and ginned. The resulting cotton seed was processed by

- delinting
- hulling and separation of seed kernels from hulls

- expansion and solvent extraction of kernels, separation into meal and crude oil
- addition of alkali and refining of crude oil

The LOQ for each compound in cotton seed as well as the processed fractions was 0.01 mg/kg. Residues below the LOQ but above the MLD were reported as the calculated value in the analytical report.

A limited processing study (2 trials) was carried out by Carringer (1998) in Mexico to produce crude oil from cotton seed from plants treated with 6 applications at the exaggerated rate of 250 g ai/ha. No evidence of concentration was observed.

Table 97. Residues in cotton seed and its processed fractions (Norris, 1995). All WG formulations

Location, Year, Reference	Application		PHI, days	Commodity	Residues, mg/kg				
	g ai/ha	No.			Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
USA (Texas), 1994 94-0353 Replicate 1 Norris, 1995	1680 soil 420 foliar	1 + 4	45	Seed	0.056	0.021	0.07	0.173	<0.004
				Meal	<0.002	<0.001	<0.002	0.004	<0.002
				Hulls	0.008	0.003	0.008	0.021	<0.002
				Crude oil	0.009	0.044	0.013	0.041	<0.005
				Refined oil	0.01	0.005	0.016	0.039	<0.005
USA (Texas), 1994 94-0353 Replicate 2 Norris, 1995	1680 soil 420 foliar	1 + 4	45	Seed	0.049	0.017	0.066	0.0143	<0.004
				Meal	<0.002	<0.002	0.003	0.007	<0.002
				Hulls	0.014	0.003	0.013	0.0029	<0.002
				Crude oil	0.017	0.006	0.019	0.061	<0.005
				Refined oil	0.013	0.006	0.019	0.054	<0.005
Mexico, 1996 12046-01 Carringer, 1998	250	6	54	Seed	<0.01	<0.002	<0.01	<0.01	NR
				Crude oil	<0.01	<0.001	<0.003	<0.01	NR
Mexico, 1996 12046-02 Carringer, 1998	250	6	46	Seed	0.037	<0.01	0.037	0.067	NR
				Crude oil	<0.01	<0.01	<0.01	<0.01	NR

NR: not reported

<u>Sunflower seed</u> (Table 98). In various residue trials in Southern Europe residues were determined in the oil extracted from the seeds and the seed-cake solid residue.

Table 98. Residues in sunflower seeds and their processed fractions.

Country, Year, Reference	Application				PHI,	Commodity	Residues, mg/kg			
	Form	g ai/t	g ai/ha	No.	days		Fipronil	MB 45950	MB 46136	RPA 200766
France, 1990 Leovillle 91-215, LA19I27 Maiano and Muller, 1991	GR		200	1	147	Seed Oil Seed cake	NR <0.01 (2) 0.01 <0.01	NR <0.01 (2) <0.01 <0.01	NR <0.01 (2) <0.01 <0.01	NR <0.01 (2) <0.01 <0.01
France, 1990 Chadenac 91-215, LA19I27 Maiano and Muller, 1991	GR		200	1	151	Seed Oil Seed cake	NR <0.01 (2) <0.01 (2)	NR <0.01 (2) <0.01 (2)	NR <0.01 (2) <0.01 (2)	NR <0.01 (2) <0.01 (2)
France, 1990 91-215, LA19I27 St. Gilles Maiano and Muller, 1991	GR		200	1	140	Seed Oil Seed cake	NR <0.01 (2) <0.01 (2)	NR <0.01 (2) <0.01 (2)	NR <0.01 (2) <0.01 (2)	NR <0.01 (2) <0.01 (2)

Country, Year, Reference	Application				PHI, days	Commodity	Residues, mg/kg			
	Form	g ai/t	g ai/ha	No.			Fipronil	MB 45950	MB 46136	RPA 200766
France, 1992 92-142 part 2, XA192R84 Muller, 1994	GR		294	1	146	Seed Oil Seed cake	NR <0.002 (2) <0.002 (2)	NR <0.002 (2) <0.002 (2)	NR <0.005 (2) <0.005 (2)	NR <0.005 (2) NR
France, 1992 92-142 part 2, XH192R84 Muller, 1994	GR		206 295	1	137	Seed Oil Seed cake	NR <0.002 (2) <0.002 (2)	NR <0.002 (2) <0.002 (2)	NR <0.005 (2) <0.005 0.006	NR <0.005 (2) NR
France, 1992 92-142 part 2, XK192R84 Muller, 1994	GR		200 300	1	140	Seed Oil Seed cake	NR <0.002 (2) <0.002 (2)	NR <0.002 (2) <0.002 (2)	NR <0.005 (2) <0.005 (2)	NR <0.005 (2) NR
France, 1993 R93567A2 Muller, 1994 g	FS	3750		1	147	Seed Oil Seed cake	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)
France, 1993 R93567H1 Muller, 1994 g	FS	3750		1	166	Seed Oil Seed cake	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)
France, 1993 R93567K1 Muller, 1994 g	FS	3750		1	89	Seed Oil Seed cake	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) 0.011, 0.013 <0.002 (2)
France, 1995 95537DJ1 Maestracci, 1996	GR		190	1	154	Seed Oil Seed cake	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)
France, 1995 95537TL1 Maestracci, 1996	GR		187	1	164	Seed oil Seed cake	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)
Italy, 1995 95743BO1 Maestracci, 1997	GR		153	1	153	Seed Oil Seed cake	NR <0.002 (2) <0.002 (2)	NR <0.002 (2) <0.002 (2)	NR <0.002 (2) <0.002 (2)	NR <0.002 (2) <0.002 (2)
Italy, 1995 95743BO2 Maestracci, 1997	GR		150	1	154	Seed Oil Seed cake	NR <0.002 <0.002	NR <0.002 <0.002	NR <0.002 <0.002	NR <0.002 <0.002
Italy, 1995 95743BO3 Maestracci, 1997	GR		150	1	154	Seed Oil Seed cake	NR <0.002 (2) <0.002 (2)	NR <0.002 (2) <0.002 (2)	NR <0.002 (2) <0.002 (2)	NR <0.002 (2) <0.002 (2)
Spain, 1994 94667SE1 Muller, 1995	FS	10,00 0		1	133	Seed Oil Seed cake	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) 0.002 <0.002 <0.002 (2)	<0.002 (2) 0.002 0.002 <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)
Spain, 1996 96637M1 Maestracci, 1997	FS	10,00 0		1	192	Seed Oil Seed cake	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) 0.002 <0.002	<0.002 (2) <0.002 (2) <0.002 (2)
Spain, 1996 96637SE1 Maestracci, 1997	FS	10,00 0		1	164	Seed Oil Seed cake	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)	<0.002 (2) <0.002 (2) <0.002 (2)

NR: not reported

Sugar cane (Table 99). A number of studies were conducted in Australia to measure residues in various processed fractions of sugar cane treated with fipronil by soil or foliar application.

Table 99. Residues in sugar cane and its processed fractions.

Country, Year, Reference	Application			PHI, days	Commodity	Residues, mg/kg				
	Form	g ai/ha	No.			Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Australia, 1996 AUS94i74br Rocky Point Queensland Keats, 1997b	WG	100 foliar	2	116	Cane	NR	NR	NR	NR	NR
					Bagasse	0.003 0.002	<0.002 (2)	0.002 <0.002	<0.002 (2)	NR
					Juice	<0.002 0.002	<0.002 (2)	<0.002 (2)	<0.002 (2)	NR
					Molasses	<0.002 (2)	<0.002 (2)	<0.002 (2)	<0.002 (2)	NR
					Sugar	<0.002 (2)	<0.002 (2)	<0.002 (2)	<0.002 (2)	NR

Country, Year, Reference	Application			PHI,	Commodity	Residues, mg/kg				
	Form	g ai/ha	No.	days		Fipronil	MB 45950	MB 46136	fipronil-desulfinyl	RPA 200766
Australia, 1996 AUS94i74cr Kurrimine Beach Queensland Keats, 1997k	WG + SC	100 soil 50 foliar	1 + 2	95	Cane	0.002 0.002	<0.002 (2)	0.002 0.002	<0.002 (2)	NR
					Bagasse	0.004 0.004	<0.002 (2)	0.003 0.002	<0.002 (2)	
					Juice	<0.002 (2)	<0.002 (2)	<0.002 (2)	<0.002 (2)	
					Cane	0.002 0.003	<0.002 (2)	0.003 0.003	0.002 0.002	NR
					Bagasse	0.004 0.004	<0.002 (2)	0.004 0.003	0.002 <0.002	
					Juice	<0.002 (2)	<0.002 (2)	<0.002 (2)	<0.002 (2)	
					Cane	0.005 0.005	<0.002 (2)	0.006 0.006	0.008 0.008	NR
					Bagasse	0.005 0.006	<0.002 (2)	0.004 0.005	0.003 0.004	
					Juice	0.003 0.003	<0.002 (2)	<0.002 (2)	<0.002 (2)	
Australia, 1995 AUS94i74r Mowilyan Queensland Keats, 1997m	WG	50 foliar	2	134	Cane	<0.002 0.002	<0.002 (2)	<0.002 (2)	<0.002 (2)	NR
					Bagasse	<0.002 0.002	<0.002 (2)	<0.002 0.002	<0.002 (2)	
					Juice	0.002 <0.002	<0.002 (2)	<0.002 (2)	<0.002 (2)	
		100 foliar	2	53	Cane	0.025 0.02	<0.002 (2)	0.008 0.007	0.003 0.003	NR
					Bagasse	0.013 0.018	0.002 0.002	0.003 0.005	0.003 0.003	
					Juice	0.004 0.005	<0.002 (2)	<0.002 (2)	<0.002 (2)	
		100 foliar	1	181	Cane	0.002 0.002	<0.002 (2)	0.002 0.002	<0.002 (2)	NR
					Bagasse	0.004 0.004	<0.002 (2)	0.002 <0.002	<0.002 (2)	
					Juice	<0.002 (2)	<0.002 (2)	<0.002 (2)	<0.002 (2)	
		200 foliar	1	181	Cane	0.002 0.002	<0.002 (2)	0.002 0.002	0.002 0.002	NR
					Bagasse	<0.002 (2)	<0.002 (2)	<0.002 (2)	0.002 0.002	
					Juice	<0.002 (2)	<0.002 (2)	<0.002 (2)	<0.002 (2)	
Australia, 1996 97NST14 Kurrimine Beach Queensland Keats, 1997k	SC	75 foliar	1	101	Cane	<0.002 (2)	<0.002 (2)	<0.002 (2)	<0.002 (2)	NR
					Bagasse	0.002 0.002	<0.002 (2)	<0.002 (2)	0.002 0.002	NR
					Juice	0.002 0.002	<0.002 (2)	<0.002 (2)	<0.002 (2)	NR
					Molasses	<0.002 (2)	<0.002 (2)	<0.002 (2)	<0.002 (2)	NR
					Sugar	<0.002 (2)	<0.002 (2)	<0.002 (2)	<0.002 (2)	NR

NR: not reported

Residues in the edible portion of food commodities

Data are reported in “Fate of residues in storage and processing”.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No monitoring or enforcement data were received.

NATIONAL MAXIMUM RESIDUE LIMITS

Information, as of 7 March 2000, on world-wide national MRLs submitted by the manufacturer is tabulated below.

Country	Commodity	MRL (mg/kg)	Definition of the residue
Australia	Bananas	0.01	Sum of Fipronil, MB 45950 and MB 46136
	Brassica vegetables (head cabbage, cauliflower, broccoli, Brussels sprouts, kohlrabi)	0.05 (temporary)	Sum of Fipronil, MB 45950, MB 46136 and fipronil-desulfinyl
	Cotton (seed, crude oil, meal, hull)	0.01	Sum of Fipronil, MB 45950, MB 46136 and fipronil-desulfinyl
	Edible offal	0.02	
	Eggs	0.02	
	Meat of mammals (fat)	0.05	
	Milk (fat)	0.01	
	Mushrooms	0.02	Sum of Fipronil, MB 45950 and MB 46136
	Peanuts (nut, crude oil, forage, fodder)	0.01	Sum of Fipronil, MB 45950 and MB 46136
	Potatoes	0.01	Sum of Fipronil, MB 45950 and MB 46136
	Poultry, edible offal	0.01	
	Poultry meat (fat)	0.02	
	Rice	0.005	Sum of Fipronil, MB 45950 and MB 46136
	Sugar cane (cane, fodder)	0.01	Sum of Fipronil, MB 45950 and MB 46136
	Sweet potatoes	0.01	Sum of Fipronil, MB 45950 and MB 46136
Brazil	Cotton	0.01	Fipronil
	Potatoes	0.05	Fipronil
	Rice	0.01	Fipronil
	Sugar cane	0.01	Fipronil
Belgium	Cereals	0.02	Sum of Fipronil and MB 46136
Czech Republic	Potatoes	0.01	
France	Bananas	0.01	Fipronil
	Maize (grain, silage)	0.01	Fipronil
	Sugar beet	0.01	Fipronil
	Sunflower	0.01	Fipronil
	Cereals	0.01	
Hungary	Cereals	0.05	
	Maize grain	0.05	
	Potatoes	0.05	
	Sugar beet	0.05	
India	Cabbage	0.05	
	Chillies	0.05	
Italy	Maize grain Maize silage	0.01 0.02	
	Sugar beet (root)	0.02	
	Sunflower seed	0.01	
	Potatoes	0.03	
	Tomatoes	0.01	
Japan	Rice	0.01	
	Water	0.0005	
Korea	Cucumber	0.06	
	Rice	0.01	
Mexico	Maize	0.01	
Peru	Cotton seed	0.01	
	Grapes	0.05	
	Mangold	0.05	
	Potatoes	0.05	
	Tomatoes	0.05	
Russia	Cereals	0.005	
	Potatoes	0.005	
South Africa	Citrus	0.05 (at LOQ)	Sum of Fipronil, MB 45950, MB 46136, fipronil-desulfinyl and RPA 200766
	Mango	0.05 (at LOQ)	Sum of Fipronil, MB 45950, MB 46136, fipronil-desulfinyl and RPA 200766

Country	Commodity	MRL (mg/kg)	Definition of the residue
Spain	Potatoes	0.01	Sum of Fipronil, MB 45950, MB 46136 and RPA 200766
Switzerland	Cereals	0	Fipronil
	Maize	0.01	Fipronil
Taiwan	Cabbage (headed leafy vegetables)	0.1	Sum of Fipronil and MB 46136
	Cabbage (non-headed leafy vegetables)	0.5	Sum of Fipronil and MB 46136
	Rice	0.01	
USA	Maize forage Maize grain Maize stover	0.15 0.02 0.3	Sum of Fipronil, MB 45950 and MB 46136
	Eggs	0.03	
	Fat - Cattle, goat, horse, sheep - Hog - Poultry	 0.4 0.03 0.05	
	Liver - Cattle, goat, horse, sheep - hog	 0.1 0.02	
	Meat - Cattle, goat, horse, sheep - Hog - Poultry	 0.04 0.01 0.02	
	Meat by-products, poultry	0.02	
	Meat by-products (except liver) - cattle, goat, horse, sheep - hog	 0.04 0.01	
	Milk (fat, reflecting 0.05 mg/kg in whole milk)	1.5	
	Rice grain	0.04	Sum of Fipronil, MB 45950, MB 46136 and fipronil-desulfinyl
	Rice straw	0.1	Sum of Fipronil, MB 45950, MB 46136 and fipronil-desulfinyl
Uzbekistan	Cabbage	0.01	
Venezuela	Cabbage	0.5	
	Cotton seed	<0.01	
	Rice	0.01	

APPRAISAL

Fipronil belongs to a new class of insecticides known as phenylpyrazoles and was first reviewed by the 1997 JMPR for toxicology only. The compound was identified by the 1998 CCPR as a candidate for the residue evaluation of a new compound by the 2000 JMPR. The evaluation was postponed to the Meeting in 2001.

The manufacturer sent the Meeting information on metabolism in animals and plants, environmental fate in soil and water, methods of residue analysis, stability of residues in stored analytical samples, uses, supervised trials and processing data as well as national MRLs. Information on national GAP was provided by the governments of Australia, The Netherlands and Poland.

Pure fipronil is a white powder with a melting-point of 203 °C and low volatility. It has limited solubility in water and medium–high solubility in certain organic solvents. The log P_{OW} for the parent and relevant metabolites of 3.5–4 suggests that bioaccumulation may occur.

The parent, metabolites and degradation products are identified by the code numbers shown below.

Code	Chemical name
fiponil (MB 46030)	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulfinylpyrazole
MB 45950	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylthiopyrazole
MB 45897	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)pyrazole
MB 46136	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4 trifluoromethylsulfonylpyrazole
fipronil-desulfinyl (MB 46513)	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylpyrazole
MB 46400	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)pyrazole-4-carboxylic acid
RPA 106889	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)pyrazole-3,4-dicarboxylic acid
RPA 104615	5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)pyrazole-4-sulfonic acid
RPA 105320	5-amino-3-carbamoyl-1-(2,6-dichloro-4-trifluoromethylphenyl)-4 trifluoromethylsulfonylpyrazole
RPA 105048	5-amino-3-carbamoyl-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylpyrazole
RPA 200761	5-amino-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulfinylpyrazole-3-carboxylic acid
RPA 200766	5-amino-3-carbamoyl-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulfinylpyrazole

Metabolism

Animals

The absorption, distribution, metabolism and excretion of [phenyl ring ^{14}C]fipronil and its toxicologically relevant photodegradation product fipronil-desulfinyl were studied in rats, goats and hens.

Parent fipronil: Rats were given a single dose of 4 or 150 mg/kg bw or 4 mg/kg bw [^{14}C]fipronil after pretreatment with 14 daily non-radiolabelled doses. After absorption, metabolism was rapid, and no unmetabolized fipronil was detected in any tissues or urine. Most of the radiolabel was eliminated in faeces, which contained unchanged [^{14}C]fipronil and metabolites, suggesting both bilary elimination of absorbed fipronil (metabolized) and elimination of unabsorbed fipronil. This observation indicates that some metabolites are probably excreted in the bile. The tissue concentrations of total radioactive residues (TRR) were high 7 days after dosing, with the highest levels in fat. The main residue in fat and other tissues examined was fipronil-sulfone.

Goats were given seven daily oral doses of [^{14}C]fipronil by capsule, equivalent to 0.05, 2 or 10 ppm in the diet (dry matter basis). Animals given the lowest and highest concentrations excreted the radiolabel extensively, mainly in the faeces. In contrast, those at 2 ppm appeared to retain a greater proportion of the administered dose. After administration at 0.05 ppm in the diet, 83% of the total dose was recovered, most (64%) being found in faeces. Much of the remaining radiolabel was estimated to have been retained in tissues (18%). At this concentration, no radiolabel was detected in urine, and negligible amounts were recovered in milk (0.86%). At the concentration of 2 ppm, a total of 50% of the radiolabel was recovered, most of which was sequestered in tissues (25%), with 2.5%, 18% and 4.6% in urine, faeces and milk, respectively. At the nominal concentration of 10 ppm, 77% of the administered radiolabel was recovered, principally in faeces (61%), with the remainder in urine (6.6%), milk (1.3%) and tissues (7.4%).

Consistent with the lipophilic nature of the compound and its metabolites, most of the radiolabelled residues were found in fat, providing supporting evidence that the radiolabel that was not recovered was retained in the animal. The parent compound was the main residue in milk and fat in animals at the highest concentration, representing 0.099 and 1.4 mg/kg, respectively. The metabolites fipronil-thioether and fipronil-sulfone were also present in these samples. Although the individual components of the TRR in kidney and muscle represented < 0.05 mg/kg fipronil equivalents, the parent compound and fipronil-sulfone were also present. In the liver, the main metabolite was fipronil-sulfone (0.46 mg/kg fipronil equivalents), representing 53% of TRR; compounds identified in smaller amounts were RPA 200076 (0.098 mg/kg fipronil equivalents) and the parent compound (0.013 mg/kg).

Hens were given repeated oral doses of [^{14}C]fipronil by capsule at a concentration of 0.05, 2 or 10 ppm in the diet (dry matter basis). Approximately 52–58% of the administered radiolabel was eliminated, principally in the excreta. Plateau levels for both the excretion of radiolabel and residue concentrations in egg yolk and white were close to being attained. The high concentrations in egg yolk, skin and fat were consistent with the lipophilic nature of the compound. The metabolite fipronil-sulfone was identified as the principal component of the TRR in eggs and tissues at all concentrations.

The fate of fipronil has been shown to be similar in all species studied. It is relatively well absorbed and extensively distributed in the tissues, with a preference for tissues with a high lipid content. Faeces and then urine were the major routes of elimination of fipronil. Its biotransformation involved changes in the functional groups attached to the pyrazole ring. The compounds identified in faeces and urine were the parent and the fipronil-sulfone, the amide (RPA 200766) derived from the nitrile group, a reduction product (fipronil-thioether), a cleavage product (MB 45897) of the sulfone and its derivatives formed by further cleavage. The fipronil-sulfone was the main compound in eggs and tissues. Parent compound and fipronil-sulfone were identified as major compounds in milk and fat.

Fipronil-desulfinyl: Rats: The absorption, distribution, metabolism and excretion of [^{14}C]fipronil-desulfinyl were studied in rats that received either a single oral dose of 1 or 10 mg/kg bw or 14 daily oral doses of unlabelled fipronil-desulfinyl at 1 mg/kg bw per day followed by a single oral radiolabelled dose. Much more of the dose was eliminated in the faeces (46–70%) than in the urine with all dosing regimens. Appreciable quantities of residues were found in the tissues 1 week after treatment, the highest concentrations being present in fat and fatty tissues. Numerous metabolites or conjugates of fipronil-desulfinyl were present in urine and faeces. Biotransformation of the compound involved changes at the functional groups attached to the pyrazole ring. Only unchanged fipronil-desulfinyl was identified in the liver, fat, skin and residual carcass.

Goats were given repeated oral doses of [^{14}C]fipronil-desulfinyl by capsule at concentrations equivalent to 0.05, 2 and 10 ppm in the diet (dry matter basis) for 7 days. Excretion was mainly in the faeces, the percentage excreted declining with decreasing dose. Plateau levels appeared to have been attained after 104 h on the basis of measurements of radiolabel in milk. The high concentrations in fat were consistent with the lipophilic nature of the compound. Fipronil-desulfinyl was identified as the principal component of the TRR in milk and tissues at all concentrations.

Hens received 14 daily doses of [^{14}C]fipronil-desulfinyl by capsule at concentrations equivalent to 0.05, 2 and 10 ppm in the diet (dry matter basis). Approximately 53–71% of the administered radiolabel was eliminated in the excreta. Measurements of radiolabel in eggs indicated that plateau levels had been attained by the end of the dosing period. The high concentrations in egg yolk, omental fat, and skin with fat were consistent with the lipophilic nature of the compound. Fipronil-desulfinyl was identified as the principal component of the TRR in egg and tissues at all concentrations.

The metabolic pathway of the photodegradation product fipronil-desulfinyl in livestock is consistent with that in rats. Fipronil-desulfinyl is metabolized to more polar derivatives or forms polar conjugates, which are excreted. Unmetabolized fipronil-desulfinyl is distributed to eggs, milk, and/or tissues, the highest concentrations being found in fat, consistent with the lipophilic nature of the molecule. These results indicate that only unchanged fipronil-desulfinyl has the potential to transfer to animal substrates in measurable quantities.

Plants

The metabolism of [phenyl ring-^{14}C]fipronil was investigated after application to the soil or to the aerial part of the plant.

Studies of metabolism after soil incorporation were carried out on maize, sugar beet, cotton and sunflowers. Quantitative analysis of radiolabel showed that the uptake of soil-applied fipronil by plants is low (< 5% on the basis of the total radiolabel measured in whole plants at harvest). Analysis of extracts of maize forage samples revealed fipronil, fipronil-sulfone and amide RPA 200766 as the major metabolites; RPA 200761 was also found. In samples taken at harvest, sugar beet and sunflower leaves, maize fodder and cotton foliage contained two common metabolites, fipronil-sulfone and RPA 200766, in addition to various amounts of the parent compound. RPA 105320 and MB 45897 were identified only in beet leaves; RPA 200761 was identified in maize fodder and cotton foliage.

With regard to edible plant parts, fipronil-sulfone and RPA 200766 were present in sugar beet, but only amide RPA 200766 was found in field maize grain. Investigation of sunflower seed extract revealed a complex mixture of substances different from those found in the leaves; a number of components each representing < 0.01 mg/kg were separated. Cotton seed was not analysed as it was found to contain < 0.01 mg/kg of the TRR.

In summary, identification of residues in plant tissues after soil incorporation of fipronil showed that the metabolism proceeded mainly by oxidation to fipronil-sulfone and hydrolysis to amide RPA 200766. Further hydrolysis of metabolites RPA 200766 and fipronil-sulfone can also occur. Very small amounts of fipronil-thioether can be formed by reduction, but in no case was it found at > 5% of the TRR

Studies of metabolism after application by foliar spray were carried out on cabbage, rice, cotton and potato. Radiolabelled residues were quantified in all plant parts. In addition to the formation of previously known fipronil metabolites by oxidation (fipronil-sulfone), reduction (fipronil-thioether) and hydrolysis (RPA 200766, RPA 200761), the photodegradates fipronil-desulfinyl and RPA 104615 were shown to be possible terminal residues after foliar application of fipronil. The main residues found consistently after foliar application were the parent and fipronil-desulfinyl; lesser amounts of fipronil-thioether and fipronil-sulfone were also formed.

Environmental fate

Soil

The photolytic degradation of [^{14}C]fipronil was studied after surface application to a clay loam soil. Fipronil degraded rapidly in both the control (no irradiation) and irradiated phase of the study, with estimated half-times of 49 and 34 days, respectively. The enhanced degradation stimulated by photolysis yielded the photodegradates RPA 104615 and fipronil-desulfinyl, which were also observed after aqueous photolysis but not in the dark control experiments nor in studies of hydrolysis (dark).

Aerobic soil degradation of [^{14}C]fipronil in various soils (sandy loam, sandy clay loam, sand) resulted in DT_{50} values of 40–308 days, depending on the soil type and temperature. The main breakdown product of fipronil in all cases was RPA 200766 (30–47%). Fipronil-sulfone was also identified as a significant degradate (about 20%). Fipronil-thioether was found, but at levels < 10%; RPA 105320 and MB 45897 were present at very low levels. Polar metabolites not previously found appeared in the later stages of the study and were generated in significant amounts (5.9–29.2%, collectively). These metabolites occurred at higher concentrations in the sandy clay loams than in the other soils. The polar metabolites were identified as acid homologues of fipronil and its metabolites, the result of hydrolysis of nitrile to amide and to carboxylic acid.

In a study of adsorption and desorption in soil, fipronil, fipronil-thioether and fipronil-desulfinyl showed medium–low mobility, and fipronil-sulfone was classified as having low mobility to immobility.

Studies of rotational crops were carried out with [^{14}C]fipronil at recommended use rates for soil incorporation or surface treatment. The results were consistent with the established pathways of environmental degradation and plant metabolism.

[^{14}C]Fipronil incorporated into soil at 157 g ai/ha was taken up at a low rate by carrot, radish, lettuce, mustard, sorghum and wheat. Only cereal forage and fodder contained concentrations of residues > 0.01 mg/kg.

After application of [^{14}C]fipronil to soil surface at 369 g ai/ha, neither fipronil nor its relevant metabolites were found in cereal grains 30–365 days later. Further, residues were not found in root crops or leafy vegetables 5 months after treatment.

A field study on radish, soya bean, pea, mustard, lettuce, sorghum and wheat confirmed that soil-surface application at 340 g ai/ha would result in low residues of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone in the vegetative portions of crops, and none in grains. At plant-back intervals of 119–367 days after treatment, concentrations of residues of the parent and its relevant metabolites ranging from < 0.005 to 0.026 mg/kg were found. Only at a short plant-back interval of 31 days were residues found in leafy and root crops (< 0.002–0.016 mg/kg).

Water–sediment systems

A study of the fate and behaviour of [^{14}C]fipronil in two water–sediment systems showed that the major degradate under aerobic conditions was the fipronil-thioether (80–88% of applied radiolabel). The DT_{50} values were 6–14 days in water, 48–75 days in sediment and 22–32 days in the total systems. Under anaerobic conditions, decomposition resulted mainly in the formation of fipronil-thioether and the amide RPA 200766, accounting for 32 to 47% of applied radiolabel, respectively. The DT_{50} value for fipronil under anaerobic conditions was 123 days.

In an investigation of the fate and behaviour of the photodegradation product [^{14}C]fipronil-desulfinyl in two water–sediment test systems (Manningtree, UK, and Ongar, UK), it was found that any fipronil-desulfinyl reaching or formed in the water after an application of fipronil moved to the sediment at an initially rapid rate. The degradation (principally hydrolysis) of the compound then proceeded steadily in both water and sediment phases. The movement of the compound from water to sediment and the degradation resulted in DT_{50} values of 4.2 days and 9.9 days and DT_{90} values of 174 days and 146 days in the two test systems.

Methods of analysis

Plant material is extracted with acetonitrile or water:acetone, and the crude extract is purified by liquid–liquid partition and column chromatography (e.g. silica gel, alumina, Florisil or C18 cartridge). Determination is conducted by GLC with an ECD, MSD or electrochemical detector. The methods have been validated for fipronil, fipronil-thioether, fipronil-sulfone, fipronil-desulfinyl and RPA 200766 in numerous matrices. The LOQs of all compounds ranged from 0.002 mg/kg in e.g. cereal grains, banana and potato to 0.01 mg/kg in cereal straw and forage.

The analytical methods for animal products follow the same steps described above. Numerous validation studies resulted in LOQs for fipronil, fipronil-desulfinyl, fipronil-thioether and MB 56136 of 0.002– 0.01 mg/kg in bovine muscle, milk, liver, kidney, fat and eggs.

The multi-residue analytical method DFG S19, suitable for enforcement, was modified and successfully validated for the determination of residues of fipronil and its metabolites (fipronil-thioether, fipronil-sulfone, fipronil-desulfinyl) in plants and animal products at 0.002 mg/kg per analyte (LOQ) and 0.02 mg/kg per analyte (10 x LOQ).

Methods were developed for the analysis of fipronil, fipronil-sulfone, fipronil-thioether, fipronil-desulfinyl, and RPA 200766 in soil. The residues are extracted from soil with acetonitrile:acetone (70:30). The sample is centrifuged, the extract is dried with Na_2SO_4, and the analytes are adsorbed onto activated charcoal and eluted with acetonitrile. The residues are quantified by GC with ECD. The LOQ is 0.005 mg/kg for all compounds.

Stability of residues in stored analytical samples

Studies of the stability of fipronil, fipronil-thioether and fipronil-sulfone on animal products (milk, liver, kidney, muscle, fat, eggs) under storage conditions indicated that they are stable at –10 °C for at least 3 months. Studies of stability in storage were also reported for residues of fipronil, fipronil-thioether, fipronil-sulfone and fipronil-desulfinyl in lettuce, potato, broccoli, cabbage, cauliflower, maize (grain, forage, fodder, oil, starch) and cotton (seed, hulls, meal, oil, gin trash). The residues were shown to be stable at –20 °C for 12–24 months.

Definition of the residue

Toxicological background

Fipronil was evaluated for toxicology by the 1997 and the 2000 JMPR. The 1997 Meeting concluded that the toxicity of the mammalian metabolites is comparable to or substantially less than that of fipronil. Because the photodegradation product fipronil-desulfinyl is of toxicological concern but not a mammalian metabolite of fipronil, it was reviewed separately.

After considering additional data, the 2000 JMPR established a group ADI of 0–0.0002 mg/kg bw for fipronil and fipronil-desulfinyl, alone or in combination. The acute RfD established by the 1997 JMPR of 0.003 mg/kg bw for fipronil and fipronil-desulfinyl, alone or in combination, was confirmed. Other toxicologically significant compounds are fipronil-sulfone and fipronil-thioether. The 2000 JMPR concluded that the metabolite RPA 200766 is significantly less toxic than fipronil, the acknowledged relevant metabolites fipronil-thioether and fipronil-sulfone and the degradation product fipronil-desulfinyl. Therefore, RPA 200766 should not be relevant for dietary risk assessment.

Plant material

Studies of plant metabolism have shown that, after soil incorporation, residues of the parent and fipronil-sulfone represent most of the total residues, the concentrations of fipronil-thioether usually being low.

In studies of foliar metabolism, most of the residues in edible plant parts (cabbage, potato tubers) consisted of the parent compound and fipronil-desulfinyl, whereas in animal feed items (rice straw, husk, bran), the parent compound, fipronil-sulfone, fipronil-desulfinyl and fipronil-thioether were the residues most relevant for consideration.

The results of supervised residue trials indicated that the parent compound is the main component of the residue. The Meeting concluded that fipronil is a good indicator compound for enforcement purposes for plant commodities. The Meeting considered that, for the purposes of long-term and short-term dietary risk assessment, the residue should be defined as the sum of fipronil, fipronil-sulfone, fipronil-desulfinyl and fipronil-thioether, calculated as fipronil.

Animal products

In a study of metabolism in goats, fipronil, fipronil-sulfone and fipronil-thioether were the principal compounds. In a study of metabolism in laying hens, fipronil-sulfone was identified as the major component of the TRR in eggs and tissues. The results of studies in which fipronil was fed to cows and hens showed that most of the residues in milk, eggs and tissues consisted of fipronil-sulfone.

The Meeting concluded that the definition of residue for enforcement purposes should be the sum of fipronil and fipronil-sulfone, expressed as fipronil. For the purposes of long-term and short-term dietary risk assessment, the residue should be defined as the sum of fipronil, fipronil-sulfone, fipronil-desulfinyl and fipronil-thioether, calculated as fipronil.

The residue definitions are thus:

- for compliance with MRLs for plant commodities: fipronil
- for compliance with MRLs for animal commodities: sum of fipronil and fipronil-sulfone, expressed as fipronil.
- for estimation of long-term and short-term dietary intake from plant and animal commodities: sum of fipronil, fipronil-desulfinyl, fipronil-sulfone and fipronil-thioether, expressed as fipronil.

The Meeting concluded that the residue is fat-soluble.

Results of supervised trials

The residues reported in supervised trials consisted of three (after soil treatment) or four (after foliar spray) components. The studies of metabolism and the supervised trials showed that after soil incorporation, residues of parent and fipronil-sulfone represented most of the total residues. After foliar uses (including soil surface treatment; broadcast treatment of flooded paddy rice), most of the residues in edible plant parts consisted of fipronil and fipronil-desulfinyl, whereas those in animal feed items were fipronil, fipronil-sulfone and fipronil-desulfinyl. If the concentrations of all components are below the LOQ (or MLD[1]), a reasonable assumption is that the concentrations of combined residues are:

- after soil incorporation and seed treatment, for food and feed commodities *(banana, potato, sugar beet, barley, wheat, maize, rice, sweet corn, sorghum, sugar cane, sunflower seed, sugar beet leaves or tops, maize forage and fodder, cereal straw)*: lower than the combined LOQs for fipronil and fipronil-sulfone;
- after foliar and soil surface use and treatment of flooded paddy rice, for food commodities *(banana, flowerhead brassicas, head cabbage, potato, rice, sorghum, cotton seed)*: lower than the combined LOQs for fipronil and fipronil-desulfinyl;
- after foliar and soil surface use and treatment of flooded paddy rice, for feed items *(pasture grass, cereal straw, sorghum forage and fodder, cotton gin trash)* and sugar cane: lower than the combined LOQs for fipronil, fipronil-sulfone and fipronil-desulfinyl.

When the concentration of one component is above and the other below the LOQ, that of the combined residue is assumed to be close to the measurable component plus the LOQ of the other. To indicate that one of the results was a real measurement, the Meeting agreed to express the sum of the values as a real figure (e.g. < 0.002 + 0.004 mg/kg = 0.006 mg/kg). The method for calculating the total residue in various situations is illustrated below.

Fipronil	Fipronil-sulfone or fipronil-desulfinyl	Total
< 0.002	< 0.002	< 0.004
< 0.002	0.004	0.006
0.003	0.005	0.008

The concentrations of residues of fipronil (437.2 g/mol) and the metabolites fipronil-thioether (421.1 g/mol, factor 1.04), fipronil-sulfone (453.1 g/mol, factor 0.965) and fipronil-desulfinyl (389.02 g/mol, factor 1.1) are given in the evaluation tables for the individual compounds but were calculated

[1]For data on maize and rice grain in the USA only: when no detectable residues were found, “less than the minimum limit of detection (MLD) ” is shown in the evaluation tables.

in the appraisal according to the respective residue definition. The LOQs of the individual compounds are not corrected by these factors.

Banana: Common practice worldwide is to apply 300–400 g ai/ha fipronil around the base of the banana stem or plant with or without incorporation into the soil. The number of treatments and the PHI are not specified in most countries.

Two studies, each of 300 and 600 g ai/ha, and one study with 800 g ai/ha were available from Australia. As separate plots were treated on different dates and the bananas were harvested at different times (PHI, 0–166 days), these were considered to be individual trials. In all 10 trials conducted according to Australian GAP (stem and soil surface treatment, 300 g ai/ha), the concentrations of fipronil, fipronil-thioether, fipronil-sulfone and fipronil-desulfinyl residues were no higher than the LOQ of 0.002 mg/kg.

Five studies of soil treatment in Guadeloupe (10 trials, 1 x 400 kg ai/ha; three trials, 2 x 400 g ai/ha) with PHIs of 0–149 days were carried out according to French GAP. A further soil treatment trial was carried out according to GAP (400 g ai/ha, soil broadcast at base of plant) in Cameroon. The ranked orders of concentrations of residues after stem and soil surface spray were < 0.002 (10) mg/kg for fipronil and < 0.004 (10) mg/kg for the sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone (calculated as fipronil). The ranked orders of concentrations of residues after soil incorporation were < 0.002 (12) and 0.003 mg/kg for fipronil and < 0.004 (12) and 0.005 mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

The combined results of 23 trials with foliar spray and soil incorporation were, in ranked order (median underlined), < 0.002 (22) and 0.003 mg/kg for fipronil and < 0.004 (22) and 0.005 mg/kg for the sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

The Meeting estimated the following residue levels in bananas: maximum residue level (fipronil), 0.005 mg/kg; STMR (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.004 mg/kg; highest residue (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.005 mg/kg.

Foliar spray use of fipronil on broccoli and cauliflower is registered in Australia with a maximum GAP of 4 x 48 g ai/ha and a PHI of 7 days. Numerous trials were carried out in Australia, but only one trial on broccoli and two on cauliflower were conducted in accordance with the maximum GAP (4 or 5 x 48 g ai/ha; PHI, 7 days). The results of further trials with two to ten applications at an interval of 7 days showed that the number of applications is of secondary relevance to the residue concentration. Therefore, further trials with 48 g ai/ha and a PHI of 7 days were used for evaluation, two on broccoli (six or ten treatments) and four on cauliflower (two, eight or nine treatments). The ranked orders of concentrations of residues after foliar spray were < 0.002 (2), 0.002, 0.003 (3), 0.005, 0.006 and 0.008 mg/kg for fipronil and < 0.004 (2), 0.004, 0.005 (3), 0.007, 0.008 and 0.01 mg/kg for the sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

Foliar spray use of fipronil in head cabbage is registered in Australia (4 x 24–48 g ai/ha; PHI, 7 days), in New Zealand (4 x 24 g ai/ha; PHI, 7 days), the Philippines (6–8 x 25–50 g ai/ha; PHI, 7 days) and other countries in Asia and Latin America. Supervised trials with various numbers of applications were available from Australia and New Zealand. The Meeting noted that the number of applications is of secondary importance for the concentration of residue, and therefore the results from trials with two, four and eight applications were combined.

In four of the trials in Australia and one in New Zealand that conformed to New Zealand GAP (24 g ai/ha), the concentrations of residues were lower than the LOQ of 0.002 mg/kg.

In five trials in Australia and one in New Zealand that complied with the Australian maximum GAP (48 g ai/ha) and in which there were detectable residues, the ranked orders of concentrations of residues were < 0.002 (3), 0.002, 0.004 and 0.014 mg/kg for fipronil and < 0.004, 0.004, 0.0042, 0.0053, 0.0062 and 0.0215 mg/kg for the sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

The Meeting noted that the data on flowerhead brassica and head cabbage were similar and could be combined for mutual support. The combined residues were, in ranked order, for fipronil: < 0.002 (6), 0.002 (2), 0.003 (3), 0.004, 0.005, 0.006, 0.008 and 0.14 mg/kg, and for the sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone (calculated as fipronil) (median underlined): < 0.004 (3), 0.004 (2), 0.0042, 0.005 (3), 0.0053, 0.0062, 0.007, 0.008, 0.01 and 0.0215 mg/kg.

The Meeting estimated the following residue levels for flowerhead brassicas and cabbages, head: maximum residue level (fipronil), 0.02 mg/kg; STMR (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.005 mg/kg; and HR (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.0215 mg/kg.

The use of fipronil on Brussels sprouts is registered in Australia (4 x 24–48 g ai/ha; PHI, 7 days), but the results of only one trial were received. The Meeting could not recommend extrapolation of the results for cabbages, head or flowerhead brassicas to Brussels sprouts and concluded that there were insufficient data to estimate a maximum residue level.

The results of four trials on brassica leafy vegetables in Malaysia with 2 x 25 or 50 g ai/ha (GAP, 4–6 x 36 g ai/ha) were received. The Meeting could not recommend extrapolation from the results for cabbages, head or flowerhead brassicas to brassica leafy vegetables and concluded that there were insufficient data to estimate a maximum residue level.

Fipronil may be used on potato as a foliar spray e.g. in Hungary (1–2 x 20 g ai/ha; PHI, 14 days), in Spain (3 x 20–24 kg ai/ha; PHI, 14 days), the Czech Republic, Poland and Slovakia (20 g ai/ha; PHI, 14 days; number of treatments not specified) and Romania (3 x 20 g ai/ha; PHI, 30 days). Another use is soil incorporation at planting, e.g. in Italy (1 x 150 g ai/ha).

For foliar spray, the results of 29 European trials conducted according to the above GAPs were submitted. Most of the samples were analysed for fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone at an LOQ of 0.002 mg/kg for each. Two trials in Hungary were analysed with a less sensitive method (LOQ, 0.01 mg/kg), resulting in unquantifiable concentrations of residues of each compound. These trials were considered to belong to another population and were excluded from the evaluation. The concentrations of residues, in ranked order, were < 0.002 (26) and 0.003 mg/kg for fipronil and < 0.004 (23) mg/kg for the sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

For soil incorporation, six trials were carried out according to Italian GAP. The samples were analysed for fipronil, fipronil-thioether and fipronil-sulfone. One study showed high concentrations of 0.017 mg/kg fipronil and 0.009 mg/kg fipronil-sulfone. No residue was determined in the corresponding untreated sample. The Meeting considered that there was no reason to exclude this value from the evaluation. The concentrations of residues, in ranked order, were < 0.002 (4), 0.005 and 0.017 mg/kg for fipronil and < 0.004 (4), 0.007 and 0.028 mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

The combined results of the 27 trials with foliar spray and the six trials with soil incorporation were, in ranked order: < 0.002 (30), 0.003, 0.005 and 0.017 mg/kg for fipronil and ≤ 0.004 (27), 0.007 and 0.028 mg/kg for the sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

The Meeting estimated the following residue levels for potato: maximum residue level (fipronil), 0.02 mg/kg; STMR (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.004 mg/kg; highest residue (sum of fipronil, fipronil-desulfinyl, fipronil-thioetherand fipronil-sulfone), 0.028 mg/kg.

Fipronil can be used on sugar beet by soil application, e.g. in France and Italy (1 x 150–160 g ai/ha). In France, another GAP is broadcast soil incorporation before sowing at 200 g ai/ha. A further use is by foliar spray at 20–24 g ai/ha (PHI, 30 days) in Hungary and Romania.

For foliar spray, only one trial conducted in Hungary according to the GAP was reported. The concentrations of the residues of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone were lower than the LOQ of 0.01 mg/kg. The Meeting considered that one trial was inadequate to allow assessment the residue of fipronil in sugar beet after foliar spray.

Numerous trials (34) by soil treatment were carried out in France at 150–200 g ai/ha according to French or Italian GAP. Ten were analysed with a method with a LOQ of 0.01 mg/kg, but concentrations greater than the LOQ were determined in three of the trials. Hence, these trials were included in the assessment. One study showed high values of 0.16 mg/kg for fipronil and 0.015 mg/kg for fipronil-sulfone. No residue was determined in the corresponding untreated sample. The Meeting noted that there was no reason to exclude this value from the evaluation. The concentrations of residues, in ranked order, were: < 0.002 (7), 0.002, 0.003 (6), 0.005 (2), 0.007, 0.009, < 0.01 (9), 0.011, 0.013, 0.014, 0.018 (2), 0.072 and 0.16 mg/kg for fipronil and < 0.004 (7), 0.005 (2), 0.007 (3), 0.008 (2), 0.008, 0.009, 0.01, 0.011, 0.014, 0.018, < 0.02 (9), 0.021, 0.024, 0.028 (2), 0.082 and 0.17 mg/kg for the sum of fipronil, fipronil-thioether, fipronil-sulfone (calculated as fipronil).

The Meeting estimated the following residue levels for sugar beet on the basis of use by soil incorporation: maximum residue level (fipronil), 0.2 mg/kg; STMR (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.0125 mg/kg; highest residue (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.17 mg/kg.

Fipronil is registered for use as a foliar spray on cereal grains (barley, oats, rye, triticale, wheat) in many countries, but adequate data on residues have not been submitted. In France, fipronil may be used as a seed treatment at 50 g ai/100 kg in cereals. There is specific GAP for wheat in Belgium (50 g ai/100 kg seed) and in Chile (50–100 g ai/100 kg seed).

Six seed treatment trials with two- to threefold excess doses on barley were reported from France (treatment with 100 or 150 g ai/100 kg of seed). The grains were analysed for fipronil, fipronil-thioether and fipronil-sulfone. At harvest, 249–271 days after sowing, the concentrations of residues of all analytes were below the LOQ of 0.002 mg/kg of grain.

Five trials on wheat seed treatment that complied with GAP (50 g ai/100 kg seed) were carried out in France. The grains were analysed for fipronil, fipronil-thioether and fipronil-sulfone. At harvest, 128–145 days after sowing, the concentrations of residues of all analytes were below the LOQ of 0.002 mg/kg of grain. In 17 trials conducted in France and Greece trials at higher application rates (75–150 g ai/100 kg seed), the concentrations of residues in the analytes were < 0.002–0.003 mg/kg. The concentrations of residues in barley and wheat after seed treatment were < 0.002 (11) mg/kg for fipronil and < 0.004 (11) mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

The Meeting agreed to extrapolate the data on residues in barley and wheat to oats, rye and triticale for seed treatment use, and estimated the following residue levels: maximum residue level (fipronil), 0.002* mg/kg; STMR (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.004 mg/kg; and highest residue (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.004 mg/kg.

The use of fipronil as seed treatment of maize is registered in many countries: e.g. France and Italy 250 g ai/100 kg seed; Mozambique and Zimbabwe, 400 g ai/100 kg seed. Eleven trials were reported from France and Spain at 250–375 g ai/100 kg seed. The samples were analysed for fipronil, fipronil-thioether and fipronil-sulfone. Residues of all analytes were undetectable (< 0.002 mg/kg). The ranked orders were thus: < 0.002 (11) mg/kg for fipronil and < 0.004 (11) mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

Another use is by soil treatment before or at sowing (e.g. France 200, Italy 100–150, USA 112–146 g ai/ha). Fifteen southern European trials complied with French and Italian GAP and 46 North American trials with GAP in the USA. The ranked orders of residues were < 0.002 (39), 0.002, < 0.004 (16), 0.004 and < 0.01 (4) mg/kg for fipronil and < 0.004 (13), 0.004, < 0.005 (24), < 0.007 (16), 0.007, < 0.012 (2), <0.013 (3) and < 0.02 mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

Hungarian GAP allows one foliar spray at 20 g ai/ha and a 30-day PHI. The concentration of residues in maize grain in a trial that complied with GAP was < 0.01 mg/kg for fipronil and its metabolites. The Meeting considered that one trial was inadequate to allow assessment of residues of fipronil in maize after foliar spray.

The combined results of trials with seed treatment and soil incorporation for maize were, in ranked order, < 0.002 (50), 0.002, < 0.004 (16), 0.004 and < 0.01 mg/kg for fipronil and < 0.004 (24), 0.004, < 0.005 (24), < 0.007 (16), 0.007, < 0.012 (2), <0.013 (3) and < 0.02 mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

The Meeting estimated the following levels for maize: maximum residue level (fipronil), 0.01 mg/kg; STMR (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.005 mg/kg; and highest residue (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.02 mg/kg.

Sweet corn (corn-on-the-cob) was analysed in four French trials after soil treatment with fipronil before or at sowing at 200 g ai/ha. The concentrations of residues, in ranked order, were: < 0.002 (3) and 0.003 mg/kg for fipronil and < 0.004, < 0.004, 0.007 and 0.01 mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

The Meeting decided that four trials were insufficient to allow estimation of a maximum residue level for sweet corn.

Fipronil may be used on rice worldwide as a seed treatment, as soil or flooded paddy application or as a foliar spray. Numerous supervised trials with various application scenarios were reported. The following trials were conducted in accordance with GAP:

- seed treatment at 10–13 g ai/100 kg seed: one trial in Australia, five trials in France
- seed treatment at 120 g ai/100 kg seed, equal to 56 g ai/ha: 17 trials in the USA
- seed-box treatment at 0.5 g ai/nursery box: five trials in Japan
- soil incorporation before planting at 56 g ai/ha: 17 trials in the USA
- broadcast treatment on flooded paddy at 1 x 50 g ai/ha: three trials in the Philippines, one trial in Taiwan, three trials in Thailand (with no analysis for fipronil-thioether or fipronil-sulfone)
- foliar treatment: six trials in the Philippines at 1 x 50 g ai/ha and four trials in Thailand at 1 x 50 g ai/ha, with no analysis for fipronil-thioether or fipronil-sulfone; one trial in Indonesia at 1 x 25 g ai/ha, with analysis for fipronil, fipronil-thioether, fipronil-sulfone and fipronil-desulfinyl.

The concentrations of residues after seed treatment, including seed boxes and soil incorporation, in ranked order, were: < 0.001 (5), < 0.002 (6), < 0.003 (31) and < 0.01 (3) mg/kg for fipronil and < 0.002 (5), < 0.004 (6), ≤ 0.006 (31) and < 0.013 (3) mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

The concentrations after broadcast treatment on flooded paddy, were < 0.001 (7) mg/kg for fipronil and ≤ 0.001 (7) mg/kg for fipronil-desulfinyl. The sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone could not be calculated, as fipronil-thioether and fipronil-sulfone were not analysed.

The concentrations after foliar spray were < 0.001 (9), 0.002 and 0.008 mg/kg for fipronil; < 0.001 (8), 0.001 (3) and 0.005 mg/kg for fipronil-desulfinyl; and 0.016 mg/kg for the sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone (calculated as fipronil; only one sample was analysed for fipronil-thioether and fipronil-sulfone).

The Meeting decided that the data on use as a foliar spray and by broadcast onto flooded paddy were insufficient because most of the samples were not analysed according to the residue definition. It decided to derive the maximum residue levels from the data for seed or seed-box treatment and soil incorporation. The Meeting estimated the following residue levels for rice: maximum residue level (fipronil), 0.01 mg/kg; STMR (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.006 mg/kg; highest residue (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.013 mg/kg.

Use of fipronil on sorghum is registered in Australia for seed treatment (75 g ai/100 kg seed) and as a foliar spray (1.5 g ai/ha; PHI, 14 days). The results of supervised trials were available only from Australia. Three trials by foliar spray and two by seed treatment were conducted according to GAP. The samples were analysed for fipronil, fipronil-thioether, fipronil-sulfone and fipronil-desulfinyl. The concentrations of residues after seed treatment, in ranked order, were: < 0.002 (2) mg/kg for fipronil and < 0.004 (2) mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil). The concentrations of residues after foliar treatment, in ranked order, were: < 0.002 and 0.002 (2) mg/kg for fipronil and < 0.004 and 0.004 (2) mg/kg for the sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

The Meeting concluded that two trials of seed treatment and three of foliar treatment were inadequate for estimating a maximum residue level or STMR for a major crop such as sorghum.

The registered Australian use pattern on sugar cane allows application of 75 g ai/ha as one spray directed at the soil at the time of planting and/or spray to the bottom 40 cm of the stalk (PHI, 84 days). In Brazil, 200 g ai/ha are used as a soil spray in furrows at the time of planting.

Five trials of soil treatment were received from Australia, but only two complied approximately with GAP. The samples were analysed for fipronil, fipronil-thioether and fipronil-sulfone. The concentrations of residues for all analytes were lower or at the LOQ of 0.002 mg/kg at PHIs of 245 and 340 days.

Five trials in which the last treatment was spray to the bottom of the stalk were carried out in Australia. Two were approximately in accordance with Australian GAP. The concentrations of residues were lower than or at the LOQ of 0.002 mg/kg, with PHIs of 95 and 101 days for all analytes (fipronil, fipronil-thioether, fipronil-sulfone and fipronil-desulfinyl).

The Meeting concluded that there were insufficient data to estimate a maximum residue level.

Fipronil may be used worldwide for seed treatment of cotton (e.g. in Australia at 50 g ai/100 kg seed) or as foliar spray (e.g. in Mexico at 2 x 50 g ai/ha; PHI, 45 days; Brazil at 4–7 x 12–80 g ai/ha; PHI, 15 days). In the USA, GAP for foliar treatment at 28–56 g ai/ha (maximum, 224 g ai/ha per season) and a PHI of 60 days is pending approval. Numerous supervised trials of seed or soil treatment followed by foliar spray or foliar spray only were reported from Australia, Brazil, Mexico and the USA. Three from Australia and one from Brazil complied with GAP.

The concentrations of residues after foliar treatment were < 0.002, 0.003, 0.004 and < 0.01 mg/kg for fipronil and < 0.004, 0.01 (2) and < 0.02 mg/kg for the sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

Forty trials in the USA complied with the pending GAP. These trials were not evaluated as pending GAP is not considered by the Meeting.

The Meeting concluded that four trials were inadequate to allow estimation of a maximum residue level or an STMR for cotton seed, as it is a major crop.

Australian GAP permits treatment of <u>sunflower seed</u> at 75 g of fipronil per 100 kg. Data were available from four supervised trials conducted according to GAP and four trials with a twofold overdose. The samples were analysed for fipronil, fipronil-thioether and fipronil-sulfone. The concentrations of residues of all analytes were < 0.002 mg/kg.

French GAP permits seed treatment with 500 g of fipronil per 100 kg of sunflower seed. Data were available from six supervised trials conducted approximately according to GAP (375 g ai/100 kg seed) and three trials in Spain with a twofold overdose (1 kg ai/100 kg seed). The samples were analysed for fipronil, fipronil-thioether and fipronil-sulfone. The concentrations of residues were < 0.002 mg/kg (6) for fipronil and from < 0.002 to 0.004 for the other analytes.

GAP in France also allows application of 200 g ai/ha as a spray directed at the soil. Data were available from eight supervised trials in France conducted approximately according to GAP, and one trial in France and one in Italy at overdoses. The samples were analysed for fipronil, fipronil-thioether and fipronil-sulfone The concentration of residues was < 0.002 mg/kg (9) for each analyte. Eight further trials in France that complied with GAP resulted in concentrations below the LOQ of 0.01 mg/kg. As no concentration > 0.01 mg/kg was detected, the Meeting did not consider the data from France based on an LOQ of 0.01 mg/kg.

The concentrations of residues after soil and seed treatment, in ranked order, were: < 0.002 (19) mg/kg for fipronil and <u>< 0.004</u> (17), 0.007 and 0.008 mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

The Meeting estimated the following levels from trials with an LOQ of 0.002 mg/kg for sunflower seed: maximum residue level (fipronil), 0.002* mg/kg; STMR (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.004 mg/kg; and highest residue (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.008 mg/kg.

A total of 27 trials were carried out in France on soil treatment of <u>sugar beet leaves or tops</u> at 150–200 g ai/ha according to French (160 g ai/ha) or Italian GAP (150 g ai/ha). The concentrations of residues on a fresh weight basis were < 0.002 (5), < 0.01(14), 0.011, 0.012, 0.014, 0.015, 0.017, 0.018, 0.021 and 0.029 mg/kg for fipronil and < 0.004 (2), 0.004, 0.005, 0.008, <u>< 0.02</u> (13), 0.021, 0.023, 0.024, 0.025, 0.027, 0.028, 0.029, 0.033 and 0.041 mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

Allowing for the standard 23% dry matter (*FAO Manual*), the Meeting estimated the following residue levels (dry weight) in sugar beet leaves or tops for soil incorporation use: maximum residue level (fipronil), 0.2 mg/kg; STMR (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.087 mg/kg (0.02/0.23).

Numerous supervised trials were available on <u>maize forage</u> (n = 72) and <u>fodder</u> (n = 55) after seed or soil treatment. To prepare silage for feed, the whole aerial portion of the immature plant must be cut at the late dough or early dent stage. Hence, the data on residues in fodder 77–120 days after treatment were used for evaluation.

The ranked orders of concentrations of residues in forage or silage on a fresh weight basis after seed treatment and soil incorporation were: < 0.002, < 0.005 (19), < 0.007 (18), < 0.01, < 0.02 (29), 0.022 (2), 0.023 and 0.038 mg/kg for fipronil and < 0.004, < 0.01 (17), < 0.012 (11), 0.012, 0.016, < 0.02, < 0.025 (2), < 0.027 (5), < 0.04 (24), 0.041, 0.042, 0.044, 0.046, 0.048, 0.05, 0.053, 0.055 and 0.079 mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

Allowing for the standard 40% dry matter (*FAO Manual*), the Meeting estimated the following residue levels (dry weight) in maize forage: maximum residue level (fipronil), 0.1 mg/kg; and STMR (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.0675 mg/kg (0.027/0.4).

The ranked orders of residues in the corresponding dry maize fodder samples taken at harvest, on a fresh weight basis (seed treatment and soil incorporation) were: < 0.002, < 0.003 (5), < 0.005 (3), < 0.02 (41), 0.02, 0.022, 0.025 and 0.04 (2) mg/kg for fipronil and < 0.004, < 0.005, < 0.008 (2), < 0.01 (2), < 0.023 (2), < 0.025, ≤ 0.04 (23), 0.041, 0.043, 0.044 (4), 0.046, 0.05, 0.052, 0.053, 0.055, 0.057 (2), 0.061, 0.062, 0.064, 0.066 (2), 0.069, 0.072, 0.09, 0.093 and 0.14 mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

Allowing for the standard 83% dry matter in maize stover (*FAO Manual*), the Meeting estimated the following residue levels (dry weight) in maize fodder: maximum residue level (fipronil), 0.1 mg/kg, and STMR (sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone), 0.048 mg/kg (0.04/0.83).

Use of fipronil as a foliar spray on pasture grass is registered in Australia (1 x 1.3 g ai/ha; PHI, 14 days) and South Africa (1 x 7.5 g ai/ha; PHI, 21 days). Numerous supervised trials on pasture grass were carried out in Australia with one application of 1.25, 2.5, 5, 7.5, 10, 20 or 30 g ai/ha; one trial was conducted in Mauritania with 11 g ai/ha; one trial was conducted in the Russian Federation with 4 g ai/ha and three trials in South Africa, each with 7.5 g ai/ha and 15 g ai/ha, were submitted. Of the trials submitted, two from Australia and the three from South Africa were in accordance with the respective GAP. The ranked orders of concentrations of residues on a fresh weight basis were < 0.002, 0.004, < 0.05, 0.21 and 0.44 mg/kg for fipronil, and < 0.006, 0.009, 0.079, 0.51 and 0.66 mg/kg for the sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

The Meeting decided that five trials were insufficient to estimate a maximum residue level or an STMR for pasture grass.

Straw and fodder (dry) of cereal grains (barley, oats, rye, triticale, wheat): The two- to threefold overdoses used in the French trials of seed treatment for barley showed residues of fipronil-sulfone (maximum 0.038 mg/kg) in the straw. These results could not be used for evaluation because the trials were not conducted according to GAP and the results do not indicate a ‘nil residue situation’ as in the corresponding grain samples.

Five trials of treatment of wheat seed that complied with GAP (50 g ai/100 kg seed) were carried out in France. Samples were taken at harvest, 128–286 days after sowing. The ranked orders of concentrations of residues in wheat straw, on a fresh weight basis, were < 0.01 (3) and 0.011 (2) mg/kg for fipronil and < 0.02 (3), 0.021 and 0.025 mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

For foliar spray use, only one trial in Poland was received, which complied with Czech and Slovak GAP. The concentrations of residues (fresh weight) were 0.017 mg/kg for fipronil and

0.063 mg/kg for the sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

The Meeting concluded that five trials with soil treatment and one with foliar treatment were insufficient to estimate a maximum residue level and an STMR for cereal straw and fodder, dry.

Numerous supervised trials with different applications to rice straw and fodder, dry were reported. The trials conducted in accordance with GAP were:

- seed treatment at 10–13 g ai/100 kg seed: one trial in Australia, five trials in France
- seed treatment at 120 g ai/100 kg seed: 17 trials in the USA
- seed-box treatment at 0.5 g ai/box: five trials in Japan
- soil incorporation before planting (56 g ai/ha): 17 trials in the USA
- broadcast treatment on flooded paddy (1 x 50 g ai/ha): one trial in the Philippines, one in Taiwan and three in Thailand
- foliar treatment (1 x 50 g ai/ha): four trials in Thailand, one in the Philippines (at Thai GAP) and one in Taiwan (at Thai GAP)
- broadcast treatment after transplanting, followed by foliar treatment (2 x 50 g ai/ha): one trial in the Philippines.

Rice straw was the only feed item used in the calculation of the dietary burden of cattle with detectable residues of the photodegradation product fipronil-desulfinyl. As separate studies of cattle feeding were carried out with fipronil and fipronil-desulfinyl, the dietary burden must be calculated differently than for other feed items, and different STMR values are needed.

The ranked orders of concentrations of residues, on a fresh weight basis, after seed treatment, including seed box and soil incorporation, were: < 0.002 (2), < 0.003 (4), < 0.005 (4), < 0.01 (29), 0.01 (2), 0.012, 0.016 and 0.04 (2) mg/kg for fipronil and < 0.004 (2), < 0.006 (4), < 0.01 (4), < 0.013 (3), ≤ 0.02 (15), 0.02, 0.021, 0.022, 0.023 (2), 0.024 (2), 0.026 (2), 0.03 (2), 0.031, 0.033, 0.06, 0.069, 0.11 and 0.26 mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

The ranked orders of concentrations of residues, on a fresh weight basis, after foliar and broadcast onto flooded paddy were: < 0.005 (5), 0.006, 0.017, 0.02, 0.027, 0.061, 0.09 and 0.13 mg/kg for fipronil; < 0.005 (2), 0.006, 0.012, 0.021, 0.025, 0.028, 0.049, 0.075, 0.084, 0.095 and 0.2 mg/kg for fipronil-desulfinyl; < 0.01 (2), 0.011, 0.014, 0.019, 0.024, 0.038, 0.058, 0.077, 0.12, 0.19 and 0.33 mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil); and < 0.015 (2), 0.018, 0.032, 0.039, 0.047, 0.069, 0.11, 0.17, 0.2, 0.3 and 0.55 mg/kg for the sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

The Meeting noted that the data resulting from different uses (seed treatment including seed box and soil incorporation on the one hand and foliar and broadcast onto flooded paddy on the other hand)constituted one population. The combined results of the two data sets were: < 0.002 (2), < 0.003 (4), < 0.005 (9), 0.006, < 0.01 (29), 0.01 (2), 0.012, 0.016, 0.017, 0.02, 0.027, 0.04 (2), 0.061, 0.09 and 0.13 mg/kg for fipronil; < 0.005 (2), 0.006, 0.012 (2), 0.025, 0.028, 0.049, 0.075, 0.084, 0.095 and 0.2 mg/kg for fipronil-desulfinyl; < 0.004 (2), < 0.006 (4), < 0.01 (6), 0.011, < 0.013 (3), 0.014, 0.019, ≤ 0.02 (15), 0.02, 0.021, 0.022, 0.023 (2), 0.024 (3), 0.026 (2), 0.03 (2), 0.031, 0.033, 0.038, 0.058, 0.06, 0.069, 0.077, 0.11, 0.12, 0.19, 0.26 and 0.33 mg/kg for the sum of fipronil, fipronil-thioether and fipronil-sulfone (calculated as fipronil); and < 0.004 (2), < 0.006 (4), < 0.01 (4), < 0.013 (3), < 0.015 (2), 0.018, ≤ 0.02 (15), 0.02, 0.021, 0.022, 0.023 (2), 0.024 (2), 0.027 (2), 0.03, 0.031, 0.032 (2), 0.034, 0.039, 0.047, 0.06, 0.07 (2), 0.11 (2), 0.17, 0.2, 0.26, 0.3 and 0.55 mg/kg for the sum of fipronil, fipronil-desulfinyl, fipronil-thioether and fipronil-sulfone (calculated as fipronil).

Allowing for the standard 90% dry matter (*FAO Manual*), the Meeting estimated the following residue levels (dry weight) for rice straw: maximum residue level (fipronil), 0.2 mg/kg;

STMR (fipronil-desulfinyl), 0.029 mg/kg (0.0265/0.9); STMR (sum of fipronil, fipronil-thioether, fipronil-sulfone), 0.022 mg/kg (0.02/0.9).

Sorghum forage and fodder: Fipronil is registered for use as a foliar spray on sorghum at 1.5 g ai/ha (PHI, 14 days). Registration as a seed treatment at 75 g ai/100 kg seed is pending. The results of supervised trials were available only from Australia. Forage samples from trials conducted in accordance with registered or pending GAP were available from three trials with foliar spraying and two with seed treatment, but straw samples were available only from the two seed treatment trials. The samples were analysed for fipronil, fipronil-thioether, fipronil-sulfone and fipronil-desulfinyl. The concentration of residues in each analyte was < 0.002 mg/kg.

The concentrations of residues, in ranked order, in forage on a fresh weight basis after foliar spray were: < 0.002 (2) and 0.002 mg/kg for fipronil and < 0.006 (2) and 0.006 mg/kg for the sum of fipronil, fipronil-desulfinyl, fipronil-thioether, fipronil-sulfone (calculated as fipronil).

The Meeting did not consider trials conducted according to a pending GAP and concluded that three trials of foliar treatment were inadequate for estimating a maximum residue level or STMR for sorghum forage or fodder.

Cotton gin trash used as animal feed includes plant parts resulting from ginning cotton, which consist of burrs, leaves, stems, lint and seeds. Data on residues after foliar spray use were reported from one trial in Australia and 30 in the USA conducted according to the pending GAP in the USA. The Meeting does not consider trials according to a pending GAP and concluded that the data were inadequate for estimating a maximum residue level or an STMR for cottonseed.

Sunflower forage and fodder samples were taken from each of four seed treatment trials conducted in Australia at 75 or 150 g ai/100 kg seed and analysed for fipronil, fipronil-thioether, fipronil-sulfone and fipronil-desulfinyl. The concentration of residues was < 0.002 mg/kg in each analyte in all samples.

The Meeting noted that sunflower forage and fodder is not a feed item and did not recommend an MRL or STMR.

The results of analyses for residues in sugar cane leaves for forage and fodder in numerous trials conducted in Australia were submitted, but only two had been conducted according to GAP. The concentrations of all the residues (parent, fipronil-thioether, fipronil-sulfone, fipronil-desulfinyl) were < 0.002 mg/kg (fresh weight).

The Meeting considered that two trials were insufficient to allow estimation of a maximum residue level or an STMR.

Fate of residues during processing

The effect of processing on the concentrations of residues of fipronil has been studied in potatoes, maize, cotton seed, sunflower seed and sugar cane. No study on the effects of processing on the nature of the residue was received. As cauliflower, broccoli, cabbages and potatoes are usually eaten cooked, studies of the effects of processing would be desirable, as the proportions of parent compound and the relevant metabolites might be changed by cooking.

In one trial on potato treated by foliar spray in the USA, 0.0035 mg/kg of fipronil and 0.003 mg/kg of fipronil-desulfinyl were detected in the tubers. The concentrations of residues of fipronil-thioether and fipronil-sulfone were lower than the LOQ/MLD of 0.003/0.001 mg/kg. When the tubers were processed to chips, flakes and wet peel, the following processing factors were calculated:

Commodity	Fipronil + fipronil-desulfinyl (mg/kg), calculated as fipronil	Processing factor
Tuber	0.0068	
Chips	< 0.004	< 0.59
Flakes	< 0.004	< 0.59
Wet peel	0.056	8.2

In a second processing study in the USA, potatoes were treated in the furrow and then by spraying of foliage. Residues of fipronil, fipronil-thioether, fipronil-sulfone and fipronil-desulfinyl were detected in the tubers, which were processed to chips, flakes and wet peel. The following processing factors were calculated:

Commodity	Fipronil + fipronil-thioether + fipronil-sulfone + fipronil-desulfinyl (mg/kg), calculated as fipronil	Processing factor
Tuber	0.057	
Chips	< 0.0126	< 0.221
Flakes	0.0156	0.274
Wet peel	0.394	6.91

As the concentrations of residues in raw agricultural commodities in the first study were near the LOQ, the Meeting applied only the processing factors from the second study to the STMR value of 0.004 mg/kg for whole potatoes in order to calculate the following STMR-P values: chips, 0.0009 mg/kg; flakes, 0.0011 mg/kg; wet peel, 0.0276 mg/kg.

A study of <u>maize</u> processing was carried out in the USA after a single in-furrow application at planting at an exaggerated application rate. No detectable residues were reported in raw or processed commodities. No STMR-P value could be estimated.

The results of two processing studies carried out in the USA on <u>cotton seed</u> were submitted. The processed fractions were meal, hulls, crude and refined (edible) oil. Two further, limited studies carried out in Mexico show no fipronil or its metabolites in oil. The processing factors were based on the studies in the USA and the second Mexican study and are shown below:

Commodity	Fipronil + fipronil-thioether + fipronil-sulfone + fipronil-desulfinyl (mg/kg), calculated as fipronil	Processing factor
Seed	0.337	
Meal	< 0.008	< 0.025
Hulls	0.042	0.12
Crude oil	0.111	0.33
Refined oil	0.074	0.22
Seed	0.289	
Meal	< 0.0126	< 0.044
Hulls	0.062	0.21
Crude oil	0.109	0.38
Refined oil	0.097	0.34
Seed	< 0.031	
Crude oil	<0.024	< 0.77
Seed	0.148	
Crude oil	< 0.031	0.21

The mean processing factors were 0.035 for cotton meal, 0.165 for cotton hulls, 0.307 for cotton crude oil and 0.28 for cotton refined oil. As no STMR value was derived for cotton seed, no STMR-P values were estimated.

In numerous supervised trials conducted on sunflower seed in southern Europe, residues in sunflower oil extracted from the seeds and the cake solid were also measured. As neither fipronil nor its metabolites were detected in raw agricultural commodities, no STMR-P value could be estimated.

Nine studies on processing of sugar cane were submitted, but only two (each with two independent trials) showed residues in raw agricultural commodities and could be used to estimate processing factors:

Commodity	Fipronil + fipronil-thioether + fipronil-sulfone + fipronil-desulfinyl (mg/kg), calculated as fipronil	Processing factor
	1 x 100 g ai/ha soil treatment + 2 x 50 g ai/ha foliar treatment	
Cane	0.0198	
Bagasse	0.0123	0.621
Juice	< 0.007	< 0.35
Cane	0.0198	
Bagasse	0.0154	0.778
Juice	0.007	0.35
	2 x 100 g ai/ha foliar treatment	
Cane	0.0363	
Bagasse	0.0213	0.587
Juice	< 0.008	< 0.22
Cane	0.0303	
Bagasse	0.0283	0.934
Juice	< 0.009	< 0.297

A mean processing factor of 0.73 was calculated for bagasse, the material left over after pressing out the juice; the juice is made into molasses and sugar. The mean processing factor was < 0.3 for sugar juice. As no STMR value was derived for sugar cane, no STMR-P values were estimated.

Residues in animal commodities

Dietary burden in animals

The Meeting estimated the dietary burden of fipronil residues and its toxicologically significant metabolites in farm animals on the basis of the diets listed in Appendix IX of the *FAO Manual*.

Separate feeding studies were carried out in cattle to determine the residues of fipronil and of its photodegradation product fipronil-desulfinyl. Detectable amounts of fipronil-desulfinyl were found only in rice straw among all the feed items considered for cows. The dietary burden was therefore calculated separately for comparison with the results of:

- the feeding study of fipronil by summing the concentrations of residues of fipronil, fipronil-thioether and fipronil-sulfone, calculated as fipronil (all animal feed)
- the feeding study of fipronil-desulfinyl by summing the concentrations of residues of fipronil-desulfinyl (rice straw only).
-

As the plateau concentration of fipronil in milk in the feeding study in dairy cows was reached slowly (> 2 weeks), the STMR and STMR-P values of the feed item were used to calculate the dietary burden for estimation of MRLs, STMR values and HR values for animal commodities, as follows:

Dietary burden of sum of fipronil, fipronil-thioether and fipronil-sulfone, calculated as fipronil

Commodity	Group	STMR or STMR-P (mg/kg)	Dry matter (%)	Residue, dry weight (mg/kg)	Choose diets (%)			Residue contribution (mg/kg)		
					Beef cattle	Dairy cows	Poultry	Beef cattle	Dairy cows	Poultry
Barley grain	GC	0.004	88	0.0045						
Maize	GC	0.005	88	0.0057			80			0.00456
Maize forage	AF	0.0675	100	0.0675	5	50		0.0034	0.0338	
Maize fodder	AS	0.048	100	0.048						
Oats	GC	0.004	89	0.0045						
Potato, wet peel		0.0276	15	0.184	75	40		0.138	0.0736	
Rice	GC	0.006	88	0.0068			20			0.00136
Rice straw and fodder	AS	0.022	100	0.022						
Rye	GC	0.004	88	0.0045						
Sugar beet leaves or tops		0.087	100	0.087	20	10	–	0.0174	0.0087	
Wheat	GC	0.004	89	0.0045						
		Total			100	100	100	0.159	0.116	0.006

Dietary burden of fipronil-desulfinyl

Commodity	Group	STMR (mg/kg)	Dry matter (%)	Residue, dry weight (mg/kg)	Choose diets (%)			Residue contribution (mg/kg)		
					Beef cattle	Dairy cows	Poultry	Beef cattle	Dairy cows	Poultry
Rice straw and fodder	AS	0.029	100	0.029	10	10	–	0.0029	0.0029	–

Feeding studies

Cows, fipronil: Groups of three lactating cows were given fipronil in bolus doses equivalent to 0.04, 0.13 or 0.43 ppm in the diet, daily for 35 days. Milk was analysed for fipronil and for fipronil-thioether and fipronil-sulfone. A plateau concentration of fipronil-derived residues was observed in milk after 25 days at the high dose. The residue in milk consisted almost entirely of fipronil-sulfone. Fipronil-thioether was detected in a single sample of milk; trace amounts of fipronil (< 0.01 mg/kg) were detected at the high dose. After 35 days of dosing, the cows were killed, and liver, kidney, fat and muscle were collected for analysis from each animal. Of the four tissues examined, fat contained the highest concentration of fipronil residues. Most of the residues consisted of fipronil-sulfone; fipronil was detected in fat of cows at the high dose at a concentration slightly above the LOQ.

The residue for compliance with the MRL is defined as the sum of fipronil and fipronil-sulfone, and that for the STMR as the sum of fipronil, fipronil-sulfone, fipronil-thioether and fipronil-desulfinyl. Fipronil-desulfinyl was not determined. As no concentrations of fipronil-thioether or fipronil above the LOQ were determined in milk, muscle, kidney or liver at the highest dose, the Meeting decided to calculate both the maximum residue levels and the STMR values on the basis of the fipronil-sulfone residues. Both fipronil and fipronil-sulfone were found in fat samples from cows at the highest dose. If all concentrations are below the LOQ, it is reasonable to assume that the concentration of combined residues is lower than the LOQ for fipronil-sulfone in milk, muscle, kidney and liver and lower than the combined LOQs for fipronil and fipronil-sulfone in fat. When one component is above and the other below the LOQ, the concentration of combined residue is assumed to be below or close to that of the measurable component plus the LOQ of the other.

The dietary burden was calculated as follows: 0.159 mg/kg (typical residue value in beef cattle) and 0.116 mg/kg (typical residue value in dairy cows). The table below shows the actual and interpolated concentrations of residues used to estimate dietary intake as the sum of fipronil and fipronil-sulfone (calculated as fipronil), on the basis of actual concentrations in cows given the intermediate dose (0.13 ppm):

Feed level (ppm) *Interpolated* / actual	Fipronil and fipronil-sulfone residues (mg/kg), calculated as fipronil								
	Milk (mean)	Muscle		Liver		Kidney		Fat	
		Highest	Mean	Highest	Mean	Highest	Mean	Highest	Mean
MRL beef cattle *0.159* / 0.13		*0.0183* / 0.015		*0.0746* / 0.061		*0.0171* / 0.014		*0.279* / 0.228	
MRL dairy cows *0.116* / 0.13	*0.0107* / 0.012								
STMR beef cattle *0.159* / 0.13			*0.0143* / 0.0117		*0.0596* / 0.0487		*0.0134* / 0.011		*0.215* / 0.176
STMR dairy cows *0.116* / 0.13	*0.0107* / 0.012								

Cows, fipronil-desulfinyl: The concentrations of fipronil-desulfinyl residues were determined in animal commodities after repeated dosing of groups of three lactating given bolus doses of fipronil-desulfinyl equivalent to 0.025, 0.075, 0.3 or 1 ppm in the diet, daily for 35 consecutive days. At the plateau (after 15–20 days), the concentration of the analyte in milk paralleled the administered dose in all but the high-dose group. Fipronil-desulfinyl residues were associated more with milk fat rather than skim milk; the milk fat concentration factor was determined to be approximately 16.

The dietary burden was calculated as 0.0029 mg/kg (STMR for beef and dairy cattle). The following table shows the highest and the mean actual and interpolated concentrations of residues of fipronil-desulfinyl, on the basis of the actual concentrations in the group given the lowest dose (0.025 ppm):

Feeding level (ppm) *Interpolated* / actual	Fipronil-desulfinyl residues (mg/kg), calculated as fipronil								
	Milk (mean)	Muscle		Liver		Kidney		Fat	
		Highest	Mean	Highest	Mean	Highest	Mean	Highest	Mean
MRL, beef and dairy cattle *0.0029* / 0.025	*0.0004* / 0.0033	*0.0004* / 0.0033		*0.0048* / 0.0418		*0.0008* / 0.0066		*0.0055* / 0.0473	
STMR, beef and dairy cattle *0.0029* / 0.025	*0.0004* / 0.0033		*0.0003* / < 0.0022		*0.0046* / 0.0396		*0.0006* / 0.0055		*0.0051* / 0.044

The following tables show the combined data from the two feeding studies and the values selected for estimation of MRLs, STMR values and HR values for animal commodities. The residue concentrations are calculated as fipronil.

MRLs for animal commodities:

Commodity	Sum of fipronil and fipronil-sulfone (mg/kg)	Proposed MRL (mg/kg)
Milk	0.0107	0.02
Liver	0.0746	0.1
Kidney	0.0171	0.02
Meat (fat)	0.279	0.5 (fat)

Highest residues for animal commodities:

Commodity	Sum of fipronil and fipronil-sulfone (mg/kg)	Fipronil-desulfinyl (mg/kg)	Sum of fipronil, fipronil-sulfone and fipronil-desulfinyl (mg/kg)	Proposed HR (mg/kg)
Liver	0.0746	0.0048	0.0794	0.079
Kidney	0.0171	0.0008	0.0179	0.018
Meat (muscle)	0.0183	0.0004	0.0187	0.019

STMR values for animal commodities:

Commodity	Sum of fipronil and fipronil-sulfone (mg/kg)	Fipronil-desulfinyl (mg/kg)	Sum of fipronil, fipronil-sulfone and fipronil-desulfinyl (mg/kg)	Proposed STMR (mg/kg)
Milk	0.0107	0.0004	0.0111	0.011
Liver	0.0596	0.0046	0.0642	0.064
Kidney	0.0134	0.0006	0.014	0.014
Meat (muscle)	0.0143	0.0003	0.0146	0.015

The Meeting estimated maximum residue levels of 0.02 mg/kg for cattle milk, 0.1 mg/kg for cattle liver, 0.02 mg/kg for cattle kidney and 0.5 mg/kg (fat) for cattle meat. It recommended that the HR values be 0.079 mg/kg for cattle liver, 0.018 mg/kg for cattle kidney and 0.019 mg/kg for cattle meat. The estimated STMR values are 0.011 mg/kg for cattle milk, 0.064 mg/kg for cattle liver, 0.014 mg/kg for cattle kidney and 0.015 mg/kg for cattle meat.

Hens, fipronil: The concentrations of fipronil-derived residues in poultry commodities were determined after repeated dosing of groups of 10 laying hens given bolus doses equivalent to 0.01, 0.031 or 0.103 ppm, daily for 42 consecutive days. Eggs were collected and analysed during this period. A plateau concentration of fipronil-derived residues was observed in eggs after about 15 days of dosing. The hens were killed 42 days after dosing was initiated, and liver, skin with adhering fat and muscle were collected for analysis; eggs and tissues were analysed for fipronil, fipronil-sulfone and fipronil-thioether.

The average concentration of fipronil-sulfone equivalents in eggs from hens at the lowest dose reached a plateau by day 12 of treatment, when there were only trace amounts (< 0.01 mg/kg) of fipronil-sulfone equivalents. In hens given the intermediate and highest doses, plateau concentrations were reached after about 28 days. No fipronil-thioether was observed in eggs at any dose, and only trace amounts of fipronil (< 0.01 mg/kg) were observed at the high dose.

The concentrations of residues were < 0.01 mg/kg in muscle and liver in hens on the low-dose regime and 0.013 mg/kg in skin with adhering fat. At all doses, fipronil-sulfone was found at much higher concentrations in skin with adhering fat than in all other tissues. The total residue in fat comprised almost entirely fipronil-sulfone, fipronil constituting < 10% in the high-dose group.

The dietary burden was calculated as 0.006 mg/kg (STMR). The following table shows the highest and the mean actual and interpolated concentrations of the sum of fipronil and fipronil-sulfone, based on the actual concentrations found in the group given the low dose (0.01 ppm):

Feed level (ppm) *Interpolated* / actual	Fipronil and fipronil-sulfone residues (mg/kg), calculated as fipronil							
	Eggs		Muscle		Liver		Skin with fat	
	Highest	Mean	Highest	Mean	Highest	Mean	Highest	Mean
MRL *0.006* / 0.01	*0.0078* / 0.013		< *0.006* / < 0.01		< *0.006* / < 0.01		*0.0084* / 0.014	
STMR *0.006* / 0.01		*0.006* / 0.01		< *0.006* / < 0.01		*0.006* / < 0.01		*0.008* / 0.0133

Hens, fipronil-desulfinyl: A separate feeding study was not provided, but in a study of metabolism in hens given [^{14}C]fipronil-desulfinyl 50–70% of the dose was recovered in the excreta. The edible tissues and eggs contained < 6% of the total applied dose, with 1–2% in egg white, 3–5% in yolk and 4–6% in tissues. The only poultry feed item that contained detectable residues of fipronil-desulfinyl was rice grain. The concentrations of residues in 29 samples of rice treated by foliar spray or in flooded paddies were < 0.001 (27), 0.002 and 0.005 mg/kg. The Meeting concluded that quantifiable residues of fipronil-desulfinyl are unlikely to occur in eggs or edible poultry tissues.

On the basis of the results of the feeding study with 0.01 ppm fipronil, the Meeting estimated maximum residue levels of 0.02 mg/kg for eggs, 0.02 mg/kg for poultry, edible offal and 0.0* for poultry meat. It recommended HR values of 0.0078 mg/kg for eggs, 0.0084 mg/kg for poultry, edible offal and 0.006 mg/kg for meat. The estimated STMRs were 0.006 mg/kg for eggs, 0.008 mg/kg for poultry, edible offal and 0.006 mg/kg for meat.

Recommendations

The Meeting estimated the following maximum residue levels and STMR values and recommended them for use as MRLs, STMR values and HR values:

Definition of the residue:

- for compliance with MRLs for plant commodities: fipronil
- for compliance with MRLs for animal commodities: sum of fipronil and 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulfonyl pyrazole (fipronil sulfone), expressed as fipronil.
- for estimation of dietary intake of plant and animal commodities: sum of fipronil, 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylsulfonyl-pyrazole (fipronil-sulfone), 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoro-methylthio-

pyrazole (fipronil thioether) and 5-amino-3-cyano-1-(2,6-dichloro-4-trifluoromethylphenyl)-4-trifluoromethylpyrazole (fipronil-desulfinyl), expressed as fipronil

The residue is fat-soluble.

Commodity		Recommendation			
CCN	Name	MRL (mg/kg)		STMR, STMR-P (mg/kg)	HR (mg/kg)
		New	Previous		
FI 0327	Banana	0.005	–	0.004	0.005
GC 0640	Barley	0.002*	–	0.004	0.004
VB 0041	Cabbages, Head	0.02	–	0.005	0.0215
MO 1280	Cattle, kidney	0.02	–	0.014	0.018
MO 1281	Cattle, liver	0.1	–	0.064	0.079
MM 0812	Cattle meat	0.5 (fat)	–	0.015	0.019
ML 0812	Cattle milk	0.02	–	0.011	
PE 0112	Eggs	0.02	–	0.006	0.0078
VB 0042	Flowerhead brassicas	0.02	–	0.005	0.0215
GC 0645	Maize	0.01	–	0.005	0.02
AF 0645	Maize forage	0.1[1]	–		
AS 0645	Maize fodder	0.1[1]	–		
GC 0647	Oats	0.002*	–	0.004	0.004
VR 0589	Potato	0.02	–	0.004	0.028
	Potato chips			0.0009	
	Potato flakes			0.0011	
PO 0110	Poultry, edible offal of	0.02	–	0.008	0.0084
PM 0110	Poultry meat	0.01*	–	0.006	0.006
GC 0649	Rice	0.01	–	0.006	0.013
AS 0649	Rice straw and fodder, dry	0.2[1]	–		
GC 0650	Rye	0.002*	–	0.004	0.004
VR 0596	Sugar beet	0.2	–	0.0125	0.17
AV 0596	Sugar beet leaves or tops	0.2[1]	–		
SO 0702	Sunflower seed	0.002*	–	0.004	0.008
GC 0653	Triticale	0.002*	–	0.004	0.004
GC 0654	Wheat	0.002*	–	0.004	0.004

[1] Expressed on dry weight basis

Further work or information

Desirable

1. Study of hydrolysis to determine the nature of residues after processing
2. Studies of processing of cabbages (cooking, sauerkraut preparation)
3. Studies of processing of potatoes (cooking, oven baking, microwaving)

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Dietary risk assessment

Long-term intake

The Meeting estimated 22 STMR values for fipronil, which were used to calculate dietary intake. The results are shown in Annex 3 (Report 2001).

The IEDIs for the five GEMS/Food regional diets, based on estimated STMR values, were 20–60% of the ADI. The Meeting concluded that dietary intake of fipronil residues is unlikely to present a public health concern.

Short-term intake

The IESTI of fipronil was calculated for the food commodities (and their processing fractions) for which MRLs, STMR values and/or HR values were established and for which data on consumption were available. The results are shown in Annex 4 (Report 2001).

The calculated short-term intakes were less than 100% of the acute RfDs for children and for the general population. The Meeting concluded that short-term intake of residues of fipronil, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

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