

FENAMIPHOS (85)

EXPLANATION

Fenamiphos was first reviewed by the JMPR in 1974, with subsequent residue evaluations published in 1977, 1978 and 1980. The compound was scheduled for periodic review at the 27th (1995) Session of the CCPR (ALINORM 95/24A Appendix IV). The 30th Session noted that the TMDI based on existing CXLs slightly exceeded the new ADI of 0.0008 mg/kg bw allocated by the 1997 JMPR.

The manufacturer submitted a comprehensive data package in support of the existing CXLs for banana, Brussels sprouts, cabbages, coffee beans, cotton seed, grapes, melons, oranges, peanut, pineapple and tomato. Additional data have been provided to support new residue limits for apple, cherries, lemons, limes, grapefruit, onions, peaches and peppers.

IDENTITY

ISO Common name: fenamiphos

Chemical name

IUPAC: ethyl 4-methylthio-*m*-tolyl isopropylphosphoramidate

CA: ethyl 3-methyl-4-(methylthio)phenyl (1-methylethyl)phosphoramidate

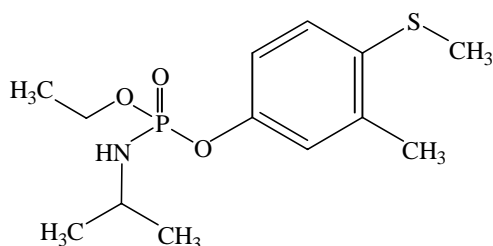
CAS No.: 22224-92-6

Synonyms: Nemacur, Bayer 68138

Molecular formula: C₁₃H₂₂NO₃PS

Molecular weight: 303.40 g/mole

Structural formula:



Physical and chemical properties

Pure active ingredient

Vapour pressure: 0.12 mPa (1.2×10^{-6} mbar) at 20°C; 0.23 mPa (2.3×10^{-6} mbar) at 25°C (Weber, 1984)

Melting point: 49°C (Klusacek *et al.*, 1986)

Purity:	99.6 molar percent (DTA); 99.5% elemental assay
Octanol/water partition coefficient:	2000 ± 370, log P _{ow} 3.30, at 20°C by HPLC (Krohn, 1984)
Solubility at 20°C:	water 0.4 g/l, 0.558 g/l (Battor <i>et al.</i> , 1984); hexane 10-20 g/l; dichloromethane, toluene and 2-propanol >200 g/l.
Specific gravity:	1.191 g cm ⁻³ (Weber, 1987)
Hydrolysis at 25°C:	(sterile solution in dark), half-life 245 days at pH 5, 301 days at pH 7, 235 days at pH 9 ¹ (Mulford, 1987)
Photolysis:	half-life 3.6 hours at pH 7 (Dime <i>et al.</i> , 1983)
Dissociation constant:	does not show basic or acidic properties in water; not possible to specify pKa value in aqueous system (Stupp, 1992)
Thermal stability:	stable at room temperature (Klusacek <i>et al.</i> , 1986)
Henry's Law Constant:	1.2 × 10 ⁻⁴ Pa.m ³ .mole ⁻¹ at 20°C (Krohn, 1995)

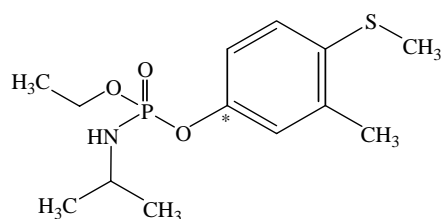
Technical material

Purity: >92%; impurities total <10.5%

METABOLISM AND ENVIRONMENTAL FATE

Animal Metabolism

Rats. In a series of experiments by Ecker *et al.* (1989) male and female rats were dosed with [1-phenyl-^{13,14}C]fenamiphos either intravenously or orally. The position of the label is shown below.



The dose groups were as follows.

0.3 mg/kg bw, single intravenous low dose

0.3 mg/kg bw single oral low dose

One oral low dose of unlabelled compound/day for 14 days followed by a single oral low dose of the radiolabelled compound

3 mg/kg bw, single oral high dose

Urine from each animal was collected in the periods 0-4, 4-8, 8-24, 24-32 and 32-48 hours after administration. Faeces were collected in the periods 0-24 and 24-48 hours, and CO₂ was trapped

¹ K_d pH 5 = 2.81 × 10⁻³; pH 7 2.3 × 10⁻³; pH 9 2.95 × 10⁻³.

in ethanolamine during 0-8, 8-24, 24-32 and 32-48 hours after administration. The following samples were taken *post mortem*: brain, heart, muscle, kidney, liver, skin, carcass, GI tract, lung, spleen, ovaries, uterus, testes and bone. Radioactivity was measured in all the samples by liquid scintillation counting and metabolites were quantified and identified by HPLC, GC-MS and/or NMR in all samples.

Urine was the major route of elimination in both male and female rats, with 93 to 100% of the administered radioactivity eliminated within 48 hours after dosing in all groups. The faeces contained 1.5 to 3.8% of the administered dose.

Low radioactive residues in the range 0.045 to 0.23% of the administered dose were found in the tissues and organs (excluding the GI tract). In all groups, the levels were below the limit of determination and the samples were not examined further.

The identified metabolites in the excreta accounted for more than 93% of the total recovered radioactivity, as shown in Table 1. The major metabolites were fenamiphos sulfoxide phenol (13-22% except in repeatedly dosed males where it accounted for only 4%) and its sulfate conjugate (40-54%), indicating that an important transformation pathway is oxidation of the methylthio group followed by cleavage of the isopropyl-nitrogen and aryl ester bonds.

Table 1. Metabolite distribution in the excreta of rats dosed with fenamiphos (Ecker *et al.*, 1989).

Compound	¹⁴ C, % of dose							
	0.3 mg/kg bw i.v.		0.3 mg/kg bw oral		14 × 0.3 mg/kg bw unlabelled + 1 × 0.3 mg/kg labelled		3 mg/kg bw oral	
	M	F	M	F	M	F	M	F
FSO	2.3	6.6		11.6	2.9		0.3	1.3
DIFSO			0.2	0.1		1.7	0.4	0.7
FP	8.4	3.5	9.6	0.8	5.3	9.8	4.6	4.0
FSOP	11.8	19.3	11.8	18.5	4.0	21.8	21.5	12.7
FSO ₂ P	4.5	2.6	3.8	3.0	1.9	4.9	10.8	6.5
FP-sulfate	19.3	15.8	6.9	8.2	5.3	4.9	6.1	5.7
FSOP-sulfate	40.2	44.0	53.7	42.5	48.4	45.3	43.4	40.3
FSO ₂ P-sulfate	7.8	4.2	7.9	7.9	15.1	7.5	10.0	11.5
OH-FSO ₂ P-sulfate	0.6	0.1		1.0	10.0			11.3
Identified	94.9	96.1	93.9	93.6	92.9	95.9	97.1	94.0
Unidentified	4.7	3.4	5.2	5.6	6.4	3.4	2.4	5.6
Solids	0.2	0.3	0.8	0.7	0.5	0.5	0.4	0.3
Body	0.2	0.2	0.1	0.2	0.2	0.2	0.1	0.1
Total	100	100	100	100	100	100	100	100

FP	fenamiphos phenol	FP-sulfate	fenamiphos phenol sulfate
FSO	fenamiphos sulfoxide	FSOP	fenamiphos sulfoxide phenol
FSOP-sulfate	fenamiphos sulfoxide phenol sulfate	FSO ₂ P	fenamiphos sulfone phenol
FSO ₂ P-sulfate	fenamiphos sulfone phenol sulfate	DIFSO	desisopropyl-fenamiphos sulfoxide
OH-FSO ₂ P	hydroxy-fenamiphos sulfone phenol sulfate		

The metabolite OH-FSO₂P-sulfate accounted for about 10% of the ¹⁴C in the repeatedly dosed male and the high-dose female groups, but for ≤1% in the other groups. This metabolite is hydroxylated in either the phenyl ring or at the 3-methyl group. It is probably formed by direct hydroxylation of FSO₂P-sulfate. The proposed metabolic pathways of fenamiphos in animals are shown in Figure 1.

In an earlier series of experiments (Gronberg, 1969), groups of male and female rats received single oral doses of 2 or 2.8 mg/kg bw of labelled fenamiphos ([¹⁴C]ethyl, [³H]methylthio or [¹⁴C]isopropyl) or 2 mg/kg bw [*ethyl*-¹⁴C]fenamiphos sulfoxide or sulfone. The animals were monitored for 48 hours during which period urine, faeces, CO₂ and water vapour were collected and

fenamiphos sulfoxide phenol and fenamiphos sulfone phenol, accounting for 19-31% and 6-8.5% of the administered label respectively, but 6 to 44% remained uncharacterized. The total radioactivity in the liver, kidneys and fat varied with time (Table 2).

Table 2. Total radioactive residues in the tissues and organs of rats dosed with [*ethyl*-¹⁴C]fenamiphos at 2.8 mg/kg bw.

Sample	¹⁴ C, mg/kg fenamiphos equivalents				
	0.5 hours	1 hour	2 hours	4 hours	9 days
Brain					0.3
Heart					0.3
Liver	4.1	17.7	6.7	11.1	1.4
Kidney	0.6	4.6	1.5	3.1	0.6
Fat	0.1	0.3	0.1	0.4	0.6
Muscle					0.2
GI tract					0.5

The distribution of radioactivity was also investigated by whole body autoradiography (Weber, 1988). Rats were dosed orally at 3 mg/kg bw and autoradiograms taken when they were killed after 0.5, 2, 8, 24 or 48 hours. After 0.5 hours radioactivity was detectable in almost all tissues and organs, with very high concentrations localised in the stomach and small intestine, bladder and kidneys. The distribution pattern was similar after 2 hours, but by 8 hours after administration the radioactive concentrations had decreased in all the tissues and organs.

The results support those of other studies, showing that fenamiphos is largely excreted from the body within a 12-hour period, with very low levels remaining in the tissues and organs after 9 days.

Dairy cow. Fenamiphos sulfoxide is a major plant metabolite and soil degradation product of fenamiphos. [*U-phenyl*-¹⁴C]fenamiphos sulfoxide was orally administered to a dairy cow in a single dose of 0.8 mg/kg bw¹ (Gronberg *et al.*, 1974). Blood, urine and milk samples were taken at hourly intervals. Faeces were collected as eliminated. The cow was killed 4 hours after administration and samples of brain, heart, liver, kidney, muscle (round, loin, flank and shoulder), fat (omental, renal and subcutaneous) and gastrointestinal contents were taken for analysis. All samples were analysed for radioactivity by scintillation counting and chromatography (GPC, TLC and GLC) following appropriate work-up.

Approximately 88% of the administered dose was recovered from the urine, faeces, milk, tissues and rumen contents. Of the recovered dose, approximately 47% was in the rumen contents, 39% was eliminated in the urine and 1.4% found in the tissues. The distribution of the radioactivity in the tissues is shown in Table 3.

Metabolites were characterized in specific tissue samples. A large proportion of the radioactivity remained unidentified. Of the identified metabolites, unchanged fenamiphos sulfoxide and fenamiphos sulfoxide phenol were the major components of the radioactivity. Some fenamiphos was also detected in fat, liver, kidney and heart.

¹ Assuming daily feed consumption of 3% bodyweight, the equivalent dose is 26.7 ppm in the feed; animal weight 418 kg.

Peak plasma concentrations of ^{14}C were 0.60 $\mu\text{g/ml}$ as fenamiphos 0.25 hours after the first dose and decreased to 0.12 $\mu\text{g/ml}$ at 6 hours. A calculated half-life of 4.5 hours was reported for elimination from plasma over the 6-hour period that was monitored. A large proportion of the administered radioactivity was eliminated, as shown in Table 4.

Table 4. Elimination of radioactivity from a lactating goat (Abbink *et al.*, 1988a; Weber and Ecker, 1990).

Route of elimination	Dose no.	Time after first dose (h)	% of total administered dose	Total, %
Urine	1	0		61.47
		8	23.83	
	2	24	8.94	
		32	19.16	
Faeces	1	0		3.63
		8	0.38	
	2	24	0.31	
		32	1.81	
Milk	1	0		0.065
		8	0.021	
	2	24	0.005	
		32	0.030	
	3	48	0.009	65.17

Urine was the predominant route of elimination accounting for 61.5% of the total administered dose. Elimination via the faeces and milk accounted for 3.63% and 0.065% of the total dose respectively. The total radioactivity in the edible tissues and organs was reported as 0.3% of the administered dose; the total recovered radioactivity was 65.5%.

The distribution of the radioactivity in the tissues is shown in Table 5. The highest levels of radioactivity were found in the liver and kidneys, with very low levels in muscle and fat.

Table 5. Distribution of radioactivity in the tissues of a lactating goat.

Sample	$\mu\text{g/g } ^{14}\text{C}$ as fenamiphos	% of TRR
Liver	0.129	0.0943
Kidney	0.041	0.044
Muscle		0.05 (total*)
Round	0.005	
Flank	0.004	
Loin	0.006	
Fat		0.16 (total*)
Perirenal	0.001	
Subcutaneous	<0.001	
Omental	<0.001	

*Calculated assuming 30% and 12% of bw for total body muscle and total body fat respectively.

The radioactive compounds were characterized further in milk, liver and kidney. Samples of milk taken 8 hours after the first and second doses were pooled, extracted and analysed. Samples of all three substrates were analysed 8 and 24 months after storage (2nd and 3rd analyses).

Milk was mixed with methanol, the sediment was filtered and washed again with methanol. The remaining extract was evaporated and the radioactive compounds separated from by preparative

HPLC. Separated radioactive fractions were compared with reference compounds by contaminants co-injection; purified extracts were identified by direct peak matching.

The radioactive compounds identified in milk, liver and kidney are shown in Table 6. Fenamiphos phenol derivatives and their sulfate conjugates, fenamiphos phenol sulphate, fenamiphos phenol sulfoxide sulphate and fenamiphos phenol sulfone sulphate, were the major metabolites found in milk; fenamiphos was not present. The analyses of milk and liver 8 and 24 months after the samples were initially analysed (Weber and Ecker, 1990) showed a different metabolic profile in the third analysis (24 months) from that in the first and second analyses, as shown in Table 6.

Table 6. Radioactive compounds in the milk, liver and kidneys of a lactating goat determined after 0, 8 and 24 months storage (2nd and 3rd analyses) (Weber and Ecker, 1990).

Compound	% of ¹⁴ C in sample, 3 determinations								
	Milk			Liver			Kidneys		
	1st	2nd	3rd	1st	2nd	3rd	1st	2nd	3rd
Fenamiphos (F)				6.42		38.79			
FSOP	5.52	4.17	52.75	13.23	15.58	14.42		5.76	11.34
FSOP-sulfate	36.42	39.94		5.79	14.08	6.60	21.96	30.97	34.66
FP			8.36			1.82			
FP-sulfate	18.48	18.27				16.20	14.12		
FSO ₂ P			30.36			1.49			
FSO ₂ P-sulfate	29.98	27.69					14.62		
FSO	2.84	3.09	2.14	31.58	51.14	16.36		16.05	15.11
DIFSO	2.00	2.08							
Total identified	95.24	95.24	93.61	51.02	80.80	95.68	50.70	52.78	61.11
Unidentified	1.53	1.13	3.61	2.07	7.78		4.61	8.26	9.23
Solids	1.66	1.32	0.77	9.61	3.05	3.25	2.11	2.51	3.82
Heptane phase			0.75		2.87	0.85	24.73	22.19	
Minor fraction	1.58	2.31	1.27	11.25	5.50	0.23		14.26	25.85
Losses				20.05			17.85		
Total	100.01	100	100.01	94	100	100.01	100	100	100.01

FP	fenamiphos phenol	FP-sulfate	fenamiphos phenol sulfate
FSOP	fenamiphos sulfoxide phenol	FSOP-sulfate	fenamiphos sulfoxide phenol sulfate
FSO ₂ P	fenamiphos sulfone phenol	FSO ₂ P-sulfate	fenamiphos sulfone phenol sulfate
DIFSO	desisopropyl-fenamiphos sulfoxide	FSO	fenamiphos sulfoxide

The major radioactive compounds in the liver were fenamiphos sulfoxide, fenamiphos sulfoxide phenol and its sulfate conjugate in the first and second analyses. In the third analysis, fenamiphos, fenamiphos sulfoxide phenol and fenamiphos phenol sulfate were the main compounds. In the first liver analysis, 20% of the radioactivity was lost and 11% was present in a minor fraction which was not further characterized.

In the kidneys fenamiphos sulfoxide phenol sulphate was the main residue in all three analyses, with fenamiphos sulfoxide and fenamiphos sulfoxide phenol also prominent in the second and third analyses and fenamiphos phenol sulfate and fenamiphos sulfone phenol sulfate prominent in the first. The third analysis did not differ significantly from the second, as it had in the liver. The total characterized radioactivity was low in kidney, as 22% and 25% of the ¹⁴C was in the heptane phase from the 1st and 2nd analyses respectively and 26% was in the minor fraction in the 3rd analysis.

The radioactive compounds found in the muscle and urine are shown in Table 7. The metabolite composition in the flank muscle differed from that in loin and round muscle.

Table 7. Distribution of radioactive compounds in muscle and urine (Weber and Ecker, 1990).

Compound	% of total ¹⁴ C in			
	Muscle			Urine
	Round	Loin	Flank	
FSOP			33.7	0.3
FSOP-sulfate	25.0	25.7		27.7
FP-sulfate				13.8
DIFSO ₂			28.2	
FSO ₂ P-sulfate	35.7	13.1	38.1	5.2
FSO	39.3	61.2		
Total identified	100	100	100	47

In urine, only 47% of the radioactivity was identified; 59% was characterized as an unidentified metabolite M1 and approximately 4% as unidentified metabolites M2 and M3.

Laying hens. Two groups of five hens were dosed orally with [1-*phenyl*-^{13,14}C]fenamiphos at a level of 1 mg/kg bw (equivalent to 10 ppm in the feed assuming a total feed intake of 10% bw/day as a maximum for hens) for three days (Abbink *et al.*, 1988b). Samples of blood were collected 0.25, 0.5, 1, 2, 4, 6 and 24 hours after the third dose. Excreta were collected at 24 hour intervals immediately before the next dose. Eggs were collected twice daily, in the morning before the dose and 8 hours after dosing. Eggs were also removed from the oviduct at slaughter.

The birds were killed 0.5 hours after the third dose and samples of kidney, liver, heart, gizzard (without lining and contents), skin (without subcutaneous fat), muscle (breast and thigh) and subcutaneous fat were taken for radioanalysis. All tissue samples were freeze-dried before combustion and liquid scintillation counting. HPLC was used for isolation and characterization of the radioactive compounds.

Radioactivity in the blood plasma reached a maximum of 0.44 µg/ml as fenamiphos (average 0.3 µg/ml) 0.5 hours after the third dose which decreased to 0.029 µg/ml (average 0.028 µg/ml) at 24 hours¹. The dose was eliminated with a half-life of 4.3 hours over the 24 hour monitoring period.

The total recovered radioactivity in individual birds ranged from 64 to 73% of the dose with elimination in the excreta ranging from 60 to 70% of the administered radioactivity. Eggs contained a maximum of 0.03% of the radioactivity. The TRR in the tissues ranged from 1.74 to 4.85%. The distribution of radioactivity in the tissues and eggs is shown in Tables 8 and 9.

Table 8. Total radioactive residues in hen tissues and eggs.

Sample	¹⁴ C, µg/g as fenamiphos
Liver	0.613
Kidney	2.267
Heart	0.230
Muscle	
Thigh	0.104
Breast	0.062
Skin	0.138
Subcutaneous fat	0.092
Eggs	0.010, 0.012
Gizzard	0.251

¹ Reported in a subsequent study.

Table 9. Radioactive residues in eggs and tissues expressed as a percentage of total recovered radioactivity (Abbink *et al.*, 1988b).

Compound	% of total radioactivity in								
	Liver	Kidney	Heart	Skin	Fat	Thigh	Breast	Gizzard	Eggs*
Fenamiphos (F)	0.4	0.4	7.7	10.4	16.5	10.4	10.8	30.4	12; 14.1
FSOP	3.4	27.2	6.7	11.1	13.2	16.4	15.4	6.8	10.4; 3.4
FSO ₂ P	8.7	30.4	19.3	13.0	10.5	9.6	8.0	16.0	10.8; 8.7
FP	14.1	0.9			31.1	2.0	1.6	5.4	6.8; 14.1
DIFSO								23.0	
FSO						15.9	17.1	2.7	
FSO ₂							10.7		
FP-sulfate	10.3	9.9	7.5	12.6	4.0	2.4	3.5	2.4	12.8; 10.3
FSOP-sulfate	3.8	9.6	8.5	16.7		4.7	4.0	3.0	10.6; 3.8
FSO ₂ P-sulfate	17.2	4.1	7.0	10.7		8.2	5.5	0.9	12.4; 17.2
Total identified	57.9	82.5	56.7	74.5	75.3	69.6	76.6	90.6	75.8; 71.6
Unidentified	14.6	4.2	11.2		8.7	7.2	1.9		12.5; 14.6
Total recovered	72.5	86.7	67.9	74.5	84.0	76.8	78.5	90.6	88.3; 86.2

FP	fenamiphos phenol	FP-sulfate	fenamiphos phenol sulfate
FSO	fenamiphos sulfoxide	FSO ₂	fenamiphos sulfoxide sulfone
FSOP	fenamiphos sulfoxide phenol	FSOP-sulfate	fenamiphos sulfoxide phenol sulfate
FSO ₂ P	fenamiphos sulfone phenol	FSO ₂ P-sulfate	fenamiphos sulfone phenol sulfate
DIFSO	desisopropyl-fenamiphos sulfoxide		

*First figures 24 hours and second figures 0.5 hours after third dose.

The main radioactive compounds in the tissues and eggs were generally fenamiphos sulfone phenol, fenamiphos sulfoxide phenol, fenamiphos phenol and their sulfate conjugates. Fenamiphos was also present in all the samples, and prominent except in liver and kidney.

In a subsequent study (Ecker and Weber, 1990) the radioactive residues in the eggs, liver and muscle were determined 8 and 24 months after the initial analyses. The results are shown in Table 10.

Table 10. Percentage distribution of radioactive compounds in the liver, muscle and eggs analysed initially and after 8 and 24 months storage (1st, 2nd and 3rd analyses; Ecker and Weber, 1990).

Compound	Liver			Muscle (breast)			Eggs		
	1st	2nd	3rd	1st	2nd	3rd	1st	2nd	3rd
Fenamiphos (F)	0.24	2.67	1.47	10.02	12.58	9.58	5.59; 9.67	5.63	13.35
FSOP	11.68	20.66	29.97	14.31	12.97	23.69	4.4; 2.05	16.26	23.14
FSOP-sulfate	9.34	3.43	6.23	3.70	9.14	5.61	4.74; 2.8	13.87	13.33
FP	2.98	1.68	1.47	1.42	2.44	2.59	3.25; 9.84		
FP-sulfate	6.24	4.66	3.98	3.3		3.73	5.95; 7.1	5.37	7.18
FSO ₂ P	22.22	25.20	25.34	7.42	8.13	18.88	5.64; 5.99	9.79	
FSO ₂ P-sulfate	3.93		1.73	5.12	7.29				
FSO		8.16	4.35	16.14	16.99	16.64	5.42 (0.5)	16.59	22.23
DIFSO	14.77								
DIFSO ₂				9.98	10.23	0.86	4.23; 6.67	6.00	
Total identified	71.40	66.46	74.54	71.41	79.77	81.58	39.22; 44.62	73.51	79.23
Unidentified	8.33	12.37	8.97	16.80	6.36	2.76	18.23; 12.60	20.10	9.27
Solids	1.63	1.68	4.30	5.99	8.83	11.22	7.79; 6.56	4.93	8.10
Heptane phase	3.17	0.48	0.27	0.54	0.25	1.18	2.74; 4.91	6.58	3.49
Losses	18.63	19.44	12.19	5.79	5.04	4.43	35.39; 25.32	0.59	

Compound	Liver			Muscle (breast)			Eggs		
	1st	2nd	3rd	1st	2nd	3rd	1st	2nd	3rd
Total	103.16	100.43	100.27	100.53	100.25	101.17	103.37; 94.01	105.71	100.09

FP	fenamiphos phenol	FP-sulfate	fenamiphos phenol sulfate
FSO	fenamiphos sulfoxide	FSOP-sulfate	fenamiphos sulfoxide phenol sulfate
FSOP	fenamiphos sulfoxide phenol	FSO ₂ P-sulfate	fenamiphos sulfone phenol sulfate
FSO ₂ P	fenamiphos sulfone phenol	DIFSO ₂	desisopropyl-fenamiphos sulfone
DIFSO	desisopropyl-fenamiphos sulfoxide		

Storage of liver and muscle for 8 and 24 months did not consistently affect the residue levels except those of fenamiphos sulfoxide phenol (FSOP) which increased. In muscle fenamiphos sulfone phenol increased also. In eggs, fenamiphos sulfoxide and its phenol increased.

The transformation pathways in rats, goats, dairy cows and hens show rapid oxidation of fenamiphos at the methylthio group to fenamiphos sulfoxide and fenamiphos sulfone. Both sulfoxide and sulfone are eventually eliminated after cleavage of the phosphate ester bond.

Plant metabolism

Beans. Mixtures of [*ethyl*-¹⁴C]fenamiphos and [*methylthio*-³H]fenamiphos were applied by stem injection or soil treatment to growing snap beans (Khasawinah, 1972a). For the stem injection 1 mg of the mixture in a ratio of ³H:¹⁴C = 9 was applied to a site 5 cm above the ground on the main stem. The soil treatment was at a rate equivalent to 6.7 kg ai/ha with a ratio of ³H:¹⁴C = 9.6. Each plant was placed in a glass growth chamber connected to acid and base traps and attached to an air pump. After 4 weeks the plants were extracted and the radioactivity in the extracts, the treated soil and the liquid in the traps was quantified by liquid scintillation spectrometry. TLC was used to separate the extracted mixtures, and was followed by enzymic and/or acid hydrolysis and gel permeation chromatography.

The distribution of the radioactivity from each treatment is shown in Table 11.

Table 11. Distribution of radioactivity in snap beans following stem injection or soil treatment.

Sample	% of applied ¹⁴ C	% of applied ³ H
Stem injection		
Extracted plant material	11	32
Solids	29.1	23.8
Water ¹	2.1	15.2
Acid trap		7.4
Base trap	38.2	
Total	80.4	78.6
Soil treatment		
Plant		
Extracted material	0.86	0.98
Solids	2.78	0.58
Water ¹	0.25	0.38
Traps		
Acid		1.26
Base	12.34	
Soil		
Extracted soil	66.22	68.13
Solids	24.06	24.06
Water		0.43
Drainage water	0.12	4.90
Total	107	101

¹ Aqueous phase remaining from the plant extraction

The results show that much of the radioactivity from the stem injection remained unextracted and was present in solids and volatiles caught in the base trap. About 90% of the radioactivity from the soil treatment remained in the soil and 24% was present in the solids after extraction; very little was present in the extracted plant material.

The distribution of the metabolites in the extracted plant material following the two treatments is compared in Table 12.

Table 12. Metabolites in snap bean extracts after treatment with ^{14}C - and ^3H -labelled fenamiphos (Khasawinah, 1972a).

Compound	% of total ^{14}C - or ^3H in sample			
	Stem injection		Soil treatment	
	^{14}C	^3H	^{14}C	^3H
FSO	30.8	10.8	39.5	34.5
FSO ₂	23.7	9.7	24.8	29.6
FSOP		21.9		33.5
FSO ₂ P		40.8		
Non-polar	26.2	1.0	27.8	

The results show that the same metabolites were formed from both treatments apart from the high proportion of the sulfone phenol from the stem injection. Much of the radioactivity from the ^{14}C label remained in the non-polar fraction; further analysis indicated that it was probably incorporated into natural plant products.

In a subsequent study (Khasawinah, 1973a), 1 mg of ^{14}C -ring- and ^3H -methylthio-labelled fenamiphos in a ratio of $^3\text{H}:^{14}\text{C}$ of 4.1 was applied as a stem injection. The plant was placed in a glass chamber for 4 weeks. The experimental procedure was as before. The distribution of the radioactivity is shown in Table 13 and the identified metabolites in Table 14.

Table 13. Distribution of radioactivity in stem-injected snap beans (Khasawinah, 1973a).

Sample	% of applied ^{14}C	% of applied ^3H
Extracted plant material	35.4	35.4
Solids	24.8	24.8
Water	42.0	42.0
Acid trap		1.0
Base trap	1.0	
Total	103.2	103.2

The amount of radioactivity in the extracted fraction and the aqueous phase was higher than in the previous study, and less volatile material was produced. Enzymic hydrolysis of the aqueous phase revealed that the activity was predominantly due to glucose conjugates of fenamiphos sulfoxide and fenamiphos sulfone phenols.

Table 14. Metabolites in beans after stem injection with ^{14}C - or ^3H -labelled fenamiphos.

Compound	% of applied ^{14}C or ^3H
FSO	41.9
FSO ₂	23.6
FSOP	15.7
FSO ₂ P	11.9
TLC origin	6.9

The $^{14}\text{C}:^3\text{H}$ ratio remained identical to that in the administered mixture. Overall, the metabolite composition has not changed much from that found in the previous study, although 100% of the applied radioactivity was recovered.

Bean plants were treated by stem injection or uptake from solution with 1 mg/plant of labelled fenamiphos (Pither and Gronberg, 1977). The following label combinations were used.

$[^3\text{H}]$ ethyl $[^{14}\text{C}]$ phenyl; ratio $^3\text{H}:^{14}\text{C} = 10$
 $[^3\text{H}]$ isopropyl $[^{14}\text{C}]$ phenyl; ratio $^3\text{H}:^{14}\text{C} = 10$
 $[^3\text{H}]$ methylthio $[^{14}\text{C}]$ isopropyl; ratio $^3\text{H}:^{14}\text{C} = 10$
 $[^3\text{H}]$ methylthio $[^{14}\text{C}]$ isopropyl; ratio $^3\text{H}:^{14}\text{C} = 5$

The plants were harvested at intervals after treatment, extracted with methanol, and the extract partitioned with water into methyl chloride. The organic and aqueous fractions were radioassayed and the remaining solids were combusted. The extracted radioactive compounds were characterized by GLC, HPLC and/or GC-MS. The distribution of the radioactivity in various fractions is shown in Table 15.

Table 15. Distribution of radioactivity in extracts of treated bean plants (Pither and Gronberg, 1977).

Label	Treatment	DAT ¹	% of radioactivity in sample		
			Aqueous	Organic	Insoluble
$[^3\text{H}]$ ethyl + $[^{14}\text{C}]$ phenyl	Stem injection (ethanol)	4	10	80.1	9.9
		7	20	56.6	22.8
$[^3\text{H}]$ isopropyl + $[^{14}\text{C}]$ phenyl	Stem injection (ethanol)	4	7.2	76.8	16.0
		7	28.8	48.6	22.6
$[^3\text{H}]$ ethyl + $[^{14}\text{C}]$ phenyl	Stem injection (glycerol)	7	41.7	49.5	8.8
		14	54.3	35.9	9.8
$[^3\text{H}]$ isopropyl + $[^{14}\text{C}]$ phenyl	Stem injection (glycerol)	7	33.5	45.1	21.4
		14	63.0	25.7	11.3
$[^3\text{H}]$ methylthio + $[^{14}\text{C}]$ isopropyl	Stem injection (glycerol)	7	24.0	68.7	7.3
		7	21.0	71.0	8.0
$[^3\text{H}]$ ethyl + $[^{14}\text{C}]$ phenyl	Solution uptake	7	9.8	81.9	8.3
		14	5.2	72.3	22.6
		21	25.9	56.9	17.3
$[^3\text{H}]$ isopropyl + $[^{14}\text{C}]$ phenyl	Solution uptake	7	16.6	76.2	7.2
		14	7.3	80.9	11.8
		21	15.9	70.1	14.0

¹Days after treatment

Most of the radioactivity was generally in the organic extracts regardless of the treatment and the identity of the radiolabel. Stem injection with glycerol¹ transferred more radioactivity into the aqueous phase than the corresponding treatment with ethanol. The distribution of the metabolites in the organosoluble fraction is shown in Table 16.

Table 16. Percentage distribution of radioactivity in organosoluble residues of fenamiphos in treated bean plants.

Treatment and label	DAT	% of radioactivity in sample					
		F	FSO	FSOP	FSO ₂	FSO ₂ P	DIFSO
Stem injection							
$[^3\text{H}]$ ethyl + $[^{14}\text{C}]$ phenyl	4	17.4	31.4	4.9	13.3	6.4	3.6
$[^3\text{H}]$ isopropyl + $[^{14}\text{C}]$ phenyl	4	10.9	30.8	6.8	12.5	11.1	3.6
$[^3\text{H}]$ ethyl + $[^{14}\text{C}]$ phenyl	7	14.2	19.8		8.6		3.8
	14	9.1	13.4		4.3	1.5	2.7
$[^3\text{H}]$ isopropyl + $[^{14}\text{C}]$ phenyl	7	6.6	17.5	4.3	7.7		3.0

¹ Glycerol stem injection 9:1 glycerol/H₂O; ethanol stem injection 1:1 ethanol/H₂O.

	14	2.3	11.1	1.8	4.5		2.5
[³ H]methylthio + [¹⁴ C]isopropyl	7	12.3	41.6		13.5		0.4
Solution uptake							
[³ H]ethyl + [¹⁴ C]phenyl	7	62.6	15.4				1.7
	14	31.8	22.8	10.9	5.2		0.9
	21	15.7	21.5	6.5	5.2	3.2	1.3
[³ H]isopropyl + [¹⁴ C]phenyl	7	46.5	18.8		7.6		
	14	51.5	18.0	5.6	4.5		0.6
	21	26.0	26.9	7.5	5.5		0.6

The results are in broad agreement with those from the previous study, with fenamiphos sulfoxide and fenamiphos sulfone generally the main metabolites after both injection and solution uptake. Fenamiphos was the predominant compound from solution uptake after 7 to 14 days.

Tomatoes. In two experiments, labelled fenamiphos was applied to the soil round tomato plants at a rate equivalent to 6.72 kg ai/ha 20 to 30 days before ripening of the fruit (Khasawinah, 1973b). In one experiment [¹⁴C]ethyl- and [³H]methylthio-labelled fenamiphos (ratio ³H:¹⁴C = 9.6) was applied to plants growing outdoors, and in the other the labels were [U-¹⁴C]phenyl and [³H]methylthio (³H:¹⁴C ratio = 4.1) and the fenamiphos was applied to glasshouse plants. Tomatoes were harvested for analysis as they ripened and foliage samples were also taken. Two varieties were used in the outdoor experiment. The results are shown in Tables 17 and 18.

Table 17. Distribution of radioactivity in extracts of treated tomatoes (Khasawinah, 1973b).

Treatment	Variety	DAT	% of radioactivity in sample		
			Aqueous	Organic	Insoluble
[¹⁴ C]ethyl + [³ H]methylthio (Outdoors)	Roma	22	32.5	46.5	20.9
		30	31.1	42.0	26.9
		37	27.5	38.5	33.9
		42	31.8	33.6	34.5
	Valiant	23	27.9	51.4	20.8
		37	30.9	40.8	28.3
		44	34.5	29.5	36.0
		50	41.9	24.5	33.6
		50*	17.1	50.4	32.5
[U- ¹⁴ C]phenyl + [³ H]methylthio (Glasshouse)		10	16.5	82.4	1.1
		34	50.5	42.4	1.3
		46	52.7	45.6	1.7
		66	64.4	33.6	2.0
		74	64.8	32.4	2.8

* foliage

The distribution of the radioactivity from ethyl- and ring-labelled fenamiphos differed, particularly in the percentage of radioactivity found in the insoluble fractions and in the increase of the ring label in the aqueous fractions with time.

Table 18. Distribution of radioactivity in organic extracts of tomato fruit and foliage

Treatment	DAT	% of radioactivity in sample				
		F	FSO	FSOP	FSO ₂	FSO ₂ P
[¹⁴ C]ethyl + [³ H]methylthio (Outdoors)	22	8.33	76.7		13.3	
	30		90		5.4	
Roma variety	37	7.5	69		8	
	42	6.3	56.7		11.7	
Valiant variety	23		69.2		19.2	
	37	6.9	65.9		11.1	
	44		54.9		19.5	

	50		50		22.6	
	50*		50		9.1	
					40.0	
[U- ¹⁴ C]phenyl+ [³ H]methylthio	10	5.3	81.3		10.0	
(Glasshouse)	34	8.9	57.8	5.2	15.9	7.0
	46	7.9	58.9	3.2	12.1	5.8
	66		66.7	3.3	18.3	5.0
	74		75	3.7	10.0	6.2

* foliage

Fenamiphos sulfoxide and fenamiphos sulfone were the main compounds found in the organic extracts. The percentages found do not differ greatly between the ethyl- and ring-labelled compounds or between the two varieties. The phenol derivatives were identified in the glasshouse crops at levels below 10% of the extracted ³H. The glasshouse and outdoor experiments were monitored for 74 and 50 days respectively. The data confirm that oxidation at the methylthio group followed by cleavage of the phosphate bond is the primary metabolic pathway in tomatoes.

Carrots. Carrots were transplanted into soil treated with a mixture of [¹⁴C]ethyl- and [³H]methylthio-labelled fenamiphos at a rate equivalent to 10.08 kg ai/ha (Khasawinah, 1973c). The plants were grown under field conditions and harvested 53, 67 and 86 days after treatment. The carrots were separated from the foliage before extraction and analysis. The radioactivity was distributed among the aqueous and organic phases and remaining solids as shown in Table 19.

Table 19. Distribution of radioactivity in extracts of carrots and foliage (Khasawinah, 1973c).

Sample	DAT	% of radioactivity in sample		
		Aqueous	Organic	Insoluble
Carrots	53	25	40.1	34.1
	67	18.5	26.2	55.4
	86	22.4	29.8	47.8
Foliage	53	25.2	38.6	36.2
	67	19.6	22.2	58.2
	86	19.2	15.2	65.7

Much of the radioactive residue was insoluble. Hydrolysis of the aqueous fraction with β -glucosidase showed that 49-69% and 44-76% of the water-soluble residue in carrots and foliage respectively was mainly composed of phenol sulfoxide and sulfone phenol conjugates. No unchanged fenamiphos was found.

Cabbage. In two experiments (Khasawinah, 1973d) cabbage seedlings were transplanted into soil treated either with fenamiphos labelled with 1-ethyl-¹⁴C and methylthio-³H at a rate equivalent to 13.44 kg ai/ha (I) or [U-phenyl-¹⁴C] and [methylthio-³H]fenamiphos at a rate equivalent to 33.59 kg ai/ha (II). The C:H ratios were 9.6 and 4.4 respectively. Cabbage heads were harvested at intervals for analysis. The distribution of radioactivity among the extract fractions is shown in Table 20, and among the metabolites in Table 21.

Table 20. Distribution of radioactivity in treated cabbage (Khasawinah, 1973).

Label and sample	DAT	% of radioactivity in sample		
		Aqueous	Organic	Insoluble
[¹⁴ C]ethyl + [³ H]methylthio (I) Outer leaves	36	2.4	67.9	29.8
	71	26.1	27.5	46.3
Inner leaves	36	3.1	66.3	30.5
	71	35.3	17.6	47.1
Whole head	61	29.5	34.6	35.9

$[^{14}\text{C}]$ phenyl + $[^3\text{H}]$ methylthio (II)				
Whole head	50	47.0	51.5	1.8
Inner leaves	90	48.1	45.7	6.2

The results of experiment I clearly show that as the cabbage matures the percentages of water-soluble and insoluble radioactivity increase while the proportion of organosoluble activity decreases. In experiment II, as a result of the higher rate of application the pattern of distribution is similar after 50 and 90 days.

Table 21. Radioactive metabolites in the organic phase from extracted cabbage.

Label and sample	DAT	Radioactivity, mg/kg as fenamiphos					
		FSO	FSOP	FSO ₂	FSO ₂ P	Origin	Rf >0.6
$[^{14}\text{C}]$ ethyl + $[^3\text{H}]$ methylthio (I)							
Outer leaves	36	1.35		0.23		1.05	0.23
	71	0.19	0.62	0.25	0.78	0.11	0.23
Inner leaves	36	0.28		0.09		0.42	0.08
	71	0.02		0.01		0.01	0.03
Whole head	61	0.09	0.09	0.04	0.09	0.06	0.08
$[U-^{14}\text{C}]$ phenyl+ $[^3\text{H}]$ methylthio (II)							
Whole head	50	6.08	0.44	1.10	0.39	3.17	ND
Inner leaves	90	0.32	0.01	0.03	0.01	0.04	ND

The main identified components of the residue were fenamiphos sulfoxide and fenamiphos sulfone or the corresponding phenols, but comparable levels of radioactivity were found at the TLC origin.

Enzymatic hydrolysis of the aqueous fraction indicated that the water-soluble metabolites were predominantly glucoside conjugates of the phenol derivatives. Radioactivity in the insoluble fraction was released by acid digestion and found to be associated with the metabolites found in the organic fraction.

The transformation pathways in cabbages are similar to those found in tomatoes, beans and carrots, namely oxidation at the methylthio group and cleavage of the phosphate bond to give phenol derivatives of the sulfoxide and the sulfone and their water-soluble conjugates.

Pineapples. Pineapple plants were treated with labelled fenamiphos by stem injection, soil drench or spray according to the regimes shown in Table 22 (Flint, 1973).

Table 22. Treatment regimes in pineapple metabolism experiments (Flint, 1973).

Experiment/Label	Treatment	Rate	Sampling (DAT)
I $[^{14}\text{C}]$ ethyl + $[^3\text{H}]$ methylthio-	Stem injection	10.02 mg/plant	1, 5, 10 and 18
II U-phenyl- ^{14}C + $[^3\text{H}]$ methylthio-	Stem injection	10.02 mg/plant	1, 5, 10 and 16
III U-phenyl- ^{14}C -	Soil application	22.4 kg ai/ha	15, 30, 60 and 90
IV U-phenyl- ^{14}C -	Spray	0.89-1.1 kg ai/ha	1, 5, 10 and 30
V $[^{14}\text{C}]$ ethyl + $[^3\text{H}]$ methylthio-	Spray	0.89-1.1 kg ai/ha	15 and 30

The stem injection was applied to the centre of the fruit stalk and the soil application was poured around the base of each plant. Whole pineapple plants were collected at sampling and stalk, fruit and foliage were extracted for analysis. Soil samples from experiment III were also analysed. The results are shown in Tables 23-25.

Table 23. Distribution of radioactivity in soil and foliage after treatment of pineapples.

Treatment	Rate	DAT	Radioactivity, mg/kg as fenamiphos	
			Soil	Foliage
Soil application III	22.4 kg ai/ha	15	24.10	0.11
		30	2.10	0.27
		60	5.20	0.12
		90	4.90	0.32
Spray IV	≈1.1 kg ai/ha	1	0.09	14.46
		5	0.05	3.68
		10	0.82	7.26
		30	0.48	3.53
Spray V	0.89 kg ai/ha	15	0.09	4.82
		30	1.77	1.57
Stem injection I	10.02 mg/plant	18	0	1.14
Stem injection II	10.02 mg/plant	16	0.005	0.23

Table 24. Distribution of radioactivity in extracts of pineapple fruit.

Treatment	DAT	% of radioactivity in sample		
		Aqueous	Organic	Insoluble
<u>Stem Injection I</u>	1	7.7	76.9	15.38
	5	20.0	76.0	4.0
	10	14.8	62.9	22.2
	18	57.5	31.2	11.2
<u>Stem Injection II</u>	1	12.5	83.3	4.2
	5	2.8	11.3	1.4
	10	46.1	46.1	7.7
	16	35.0	50.0	15.0
<u>Soil Application III</u>	15	15.0	47.5	37.5
	30	25.6	60.0	14.4
	60	55.3	10.9	34.0
	90	58.6	8.1	33.3
<u>Spray IV</u>	1	3.5	92.9	3.5
	5	9.8	89.1	1.0
	10	15.1	73.6	11.3
	30	29.2	62.5	8.3
<u>Spray V</u>	15	14.8	78.8	6.3
	30	17.5	76.3	11.4

For all treatments the results show a general increase in water-soluble radioactivity with an associated decrease in organosoluble radioactivity with time. A high proportion of the radioactive material in the fruit was organosoluble. Its constituents in experiments III and IV are shown in Table 25.

Table 25. Organosoluble radioactive residues in treated pineapple plants.

Treatment and sample	DAT	¹⁴ C, µg/kg (ppb) as fenamiphos for F, FSO and FSO ₂ ; as fenamiphos phenol for FSOP and FSO ₂ P					
		F	FSO	FSOP	FSO ₂	FSO ₂ P	Unknown
Soil application III	15		0.0017	0	0.1	0	0.0001
	30		0.009	0.0001	0.0002	0.0001	0
	60		0.0035	0.0002	0.0004	0.0002	0.0001
	90		0.0044	0.0001	0.0006	0.0004	0
Spray IV	surface	1	0.94	1.32	0.01	0.02	0.01
	pulp	1	0.05	0.05	0.001	0.001	0.001
	surface	5	0.85	2.41	0.04	0.11	0.02
	pulp	5	0.05	0.28	0.002	0.02	
	surface	10	0.15	1.27	0.03	0.12	0.02
	pulp	10	0.02	0.25	0.01	0.03	0.002
	surface	30	0.05	1.16	0.05	0.17	0.03
	pulp	30	0.001	0.02	0.001	0.003	0.001

The metabolites identified in fruit from the stem injection treatments were predominantly fenamiphos sulfoxide and sulfone and their phenols. In experiment II fenamiphos sulfoxide, fenamiphos sulfone and their phenol derivatives accounted for 48.5, 1.8, 18 and 4% of the radioactivity in the fruit respectively.

In enzymatic hydrolysates of the aqueous fractions from extracts of fruit treated by stem injection and spraying, 14 to 34% and 6 to 14% of the TRR was identified as fenamiphos sulfoxide phenol and fenamiphos sulfone phenol respectively.

In summary the data show that the metabolites in pineapple fruit are similar to those found in other plants, namely fenamiphos sulfoxide and sulfone and their phenol derivatives, irrespective of the application method. The main transformation pathway is stepwise oxidation at the methylthio group followed at each stage by cleavage of the phosphate group to leave the corresponding phenolic compounds.

Waggoner (1972) treated tomatoes, potatoes, beans and peanuts with labelled fenamiphos and identified the metabolites fenamiphos sulfoxide and fenamiphos sulfone by IR and mass spectrometry.

Tobacco seedlings were treated with [*ethyl*-¹⁴C]- and [*methylthio*-³H]fenamiphos at a rate equivalent to 11.2 kg ai/ha either a week before or a week after transplanting and grown indoors or outdoors (Khasawinah, 1971). Leaves collected 7 to 70 days after treatment were analysed. Radioactivity was also determined in cured leaves.

The radioactive residue was mainly composed of fenamiphos sulfoxide and fenamiphos sulfone, at levels ranging from 60 to 92% and 8 to 40% of the applied radioactivity respectively in plants after transplanting, and from 55 to 95% and 5 to 45% when treatment was before transplanting. Curing the leaves for 50 to 100 days resulted in some loss of radioactivity.

Rotational crops

Soil was treated with [¹⁴C]ethyl- and [³H]methylthio-labelled fenamiphos at a rate equivalent to 11.2 kg ai/ha and tobacco plants were grown in the soil for 70 days (Khasawinah, 1972b). The soil was maintained in the laboratory for 3 months, then stored in a freezer for 10 months. The TRR in the soil was then 1.7 mg/kg fenamiphos equivalents. Dilution with untreated soil resulted in residues as fenamiphos of 0.65 mg/kg fenamiphos sulfoxide, 0.07 mg/kg fenamiphos sulfone and 0.18 mg/kg unextractable. Soya bean plants were grown in the treated soil for 150 days and seedlings were removed 19 days after planting. Seedlings and samples of leaves, stems, shells and seeds from mature plants were extracted (Table 26).

Table 26. Distribution of radioactivity in extracts of soya bean plants grown as a rotational crop.

Sample	Radioactivity as mg/kg		
	Aqueous (as FSO ₂ P)	Organic (as FSO + FSO ₂)	Solids (as fenamiphos)
Seedlings	4.8 (FSO + FSO ₂ P)	16.9	1.7
Leaves	8.7	3.5	2.7
Stems	0.6	0.35	0.7
Shells	0.2	0.11	0.3
Beans	0.05	0.19	0.5

Further analysis showed that the 16.9 mg/kg sum of fenamiphos sulfoxide and sulfone concentrated by the seedlings was in a ratio of 73:17. In mature plants, the sum of the two metabolites was 3.5 mg/kg in dry leaves and 0.19 mg/kg in dry beans.

Enzymatic hydrolysis of the water-soluble fractions from seedlings and mature plants released fenamiphos sulfoxide phenol equivalent to 60% and 10% of the recovered radioactivity respectively and fenamiphos sulfone phenol equivalent to 40% and 90%.

In a subsequent crop rotation study on soya beans (Hanna and Schermoly, 1980), ring-labelled [^{13,14}C]fenamiphos was applied to sandy loam soil at a rate equivalent to 6.72 kg ai/ha. Soya beans were planted immediately after treatment and harvested after 30 days (emergency re-plant) and 120 days (immediate rotational crop). Sugar beet, wheat and mustard were planted 31 days after treatment and sugar beet, oats and mustard 115 days after treatment. Soil was analysed at intervals at and after the 31- and 115-day plantings. Crop samples were separated into tops, roots, foliage, stalks, greens and chaff before analysis. The results are shown in Tables 27-29.

Table 27. Total radioactive residues in soil and in crops planted 31 and/or 115 days after soil treatment.

DAT	TRR, mg/kg as fenamiphos, in soil and rotational crops planted after 31 or 115 days									
	Soil		Sugar beet (31)		Sugar beet (115)		Mustard (31)	Mustard (115)	Wheat forage (31)	Oat forage (115)
	30 ¹	120 ¹	Root	Tops	Root	Tops				
0	3.07	2.98								
31	2.29	2.66								
45		1.99								
59	2.98		13.0				8.53		25.7	
76		1.37								
90		1.70								
115	1.68	1.80	3.60				1.35		3.75	
143	1.62		2.81				0.60		2.56	
185	1.57	1.91	1.12	1.28	0.77		0.42	0.72	1.12	0.91
213		1.43			0.66			0.49		1.09
276		1.90	0.73	0.30	0.47	0.44	0.25	0.36	0.81	0.94
308	1.54	1.88	0.61	0.30	0.44	0.41		0.37		1.67
328	1.64	1.28			0.69	0.19		0.31		
350		1.12								
365 Harvest									3.50 straw	1.91 straw
									0.43 grain	0.63 grain
									2.48 chaff	3.06 chaff

¹DAT for soya bean harvest

The data show little difference between the TRR in crops planted 30 days and 120 days after treatment. Sugar beet roots differ from tops and from the other plants in that the TRR in the roots is higher in the 31-day than in the 115-day planting at the same intervals after treatment. The data suggest that fenamiphos did not break down rapidly in these conditions.

Table 28. Distribution of radioactivity in extracted fractions of rotational crops.

Crop	Planting, DAT	¹⁴ C, % of TRR and mg/kg as fenamiphos					
		Organic		Aqueous		Insoluble	
		% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Sugar beet	31	13	0.04	44	0.13	43	0.13
Sugar beet tops	31	13	0.08	78	0.48	9	0.05
Sugar beet	115	16	0.03	50	0.10	34	0.06
Sugar beet tops	115	15	0.10	70	0.48	15	0.11
Mustard	31	23	0.06	68	0.17	9	0.02
	115	25	0.08	65	0.20	10	0.03
Wheat straw	31	30	1.38	31	1.43	39	1.79
Oat straw	115	23	0.86	31	1.16	46	1.72
Oat grain	115	10	0.06	20	0.12	70	0.45

The results are in agreement with those found in plant metabolism studies conducted over prolonged periods: most of the radioactivity is extracted into the aqueous fractions owing to conjugation of the metabolites.

Table 29. Metabolites in organosoluble fractions of extracts of rotational crops.

Crop	Planting, DAT	% of TRR in samples as				FSO + FSO ₂ , as mg/kg as F
		FSO	FSO ₂	Phenols	Total (FSO + FSO ₂)	
Sugar beet	31	3	4	1	7	0.02
Sugar beet tops	31	2	4	2	6	0.04
Sugar beet	115	5	5	1	10	0.02
Sugar beet tops	115	2	3	4	5	0.03
Mustard	31	4	5	2	9	0.02
	115	5	8	1	13	0.04
Wheat straw	31	4	5	16	9	0.41
Oat straw	115	1	1	6	2	0.07
Oat grain	115	1	1	4	2	0.01

Fenamiphos sulfoxide, fenamiphos sulfone and their phenols were identified as the major radioactive components in the organic fraction.

[1-*phenyl*-^{13,14}C]fenamiphos was applied to sandy loam soil at a rate of 7.6 kg ai/ha (Linke-Ritzer and Brauner, 1990). Rotational crops of silver beet (Swiss chard), red beets and wheat were planted 30, 120 and 269 days after application (three crop rotations). Soya beans were planted in the treated soil as a cover crop and removed 30, 120 and 269 days after treatment to plant the rotational crop. The crops were harvested at maturity, and samples of immature wheat forage were taken in addition to the mature crop. Soil samples were collected 30, 120, 269 and 392 days after application. The results are shown in Tables 30-32.

Table 30. Total radioactivity and identified compounds in soil at intervals after treatment.

Compound or fraction	Residues in soil, % of applied radioactivity at DAT			
	30	120	269	392
Fenamiphos	0.22	0.06	0.01	–
FSO ₂	0.43	0.48	0.16	0.14
FSO ₂ P	0.28	0.46	0.01	0.02
FSOP	0.35	0.14	0.02	0.01
FSO	2.64	1.29	0.24	0.20
Origin	0.05	0.07	0.03	0.02
Bound	0.54	1.26	1.32	1.18
Total	4.51	3.80	1.80	1.60
TRR*	75	63	30	27

* Assuming TRR at day 0 =100%.

The figures clearly show that the total radioactivity in soil decreases with time after application. Similarly, the residues found in the crops show that the radioactivity decreased with each rotation (Table 31).

Table 31. Total radioactive residues in rotational crops.

Crop	TRR, mg/kg fenamiphos equivalents at each rotation		
	Rotation 1 (30 days)	Rotation 2 (120 days)	Rotation 3 (269 days)
Swiss chard	8.71	1.25	0.57
Red beets			
Roots	4.62	0.48	0.10
Tops	7.31	2.83	0.36

Wheat			
Forage	17.30	15.17	2.36
Straw	46.43	19.79	4.78
Kernels	0.98	0.73	0.20

The distribution of radioactivity in the aqueous and organic phases of the crop extracts (Table 32) indicates that in the first rotation the radioactivity is predominantly extracted into the organic phase and at the second and third rotations there is an increase in the extraction into the aqueous phase and in the bound residues.

Table 32. Distribution of radioactivity in extracts of rotational crops (Linke-Ritzer and Brauner, 1990).

Fraction	% of radioactivity in sample					
	Swiss chard	Red Beets		Wheat		
		Roots	Tops	Forage	Straw	Grain
Rotation 1						
Organic	54	29	36	67	34	17
Aqueous	45	51	62	28	56	59
Bound	0	20	3	6	10	24
Rotation 2						
Organic	9	10	17	68	21	7
Aqueous	81	61	73	29	67	25
Bound	10	30	11	3	12	68
Rotation 3						
Organic	15	5	13	75	17	4
Aqueous	74	49	71	16	62	17
Bound	11	46	17	9	21	79

The results are consistent with the formation of phenol conjugates and bound residues as fenamiphos is progressively metabolized.

Table 33. Identification of radioactive residues at each rotation (Linke-Ritzer and Brauner, 1990).

Compound	Radioactivity, % of TRR in sample					
	Swiss chard	Red beets		Wheat		
		Roots	Tops	Forage	Straw	Grain
Rotation 1						
F	0.4	ND	0.4	ND	ND	ND
FSO	28.1	12.4	19.0	31.3	10.4	4.0
FSO ₂	20.2	7.3	11.1	18.0	14.1	7.8
FSOP	3.1	4.6	2.1	12.1	5.6	3.8
FSO ₂ P	4.9	8.1	3.4	10.6	5.8	15.1
OH-FSO ₂ P	1.2	1.0	0.2	2.4	0.5	11.2
FSOP-glu	2.0	5.5	4.1	3.4	10.3	9.4
FSO ₂ P-glu	1.0	4.1	3.7	5.2	13.5	15.9
FSO ₂ P-conj	15.7	7.0	18.0	1.4	1.7	ND
DAFSO	13.9	ND	13.2	2.3	ND	ND
Subtotal	90.5	50.1	75.2	86.7	62.0	67.2
Unidentified	8.9	23.2	14.9	2.2	11.6	3.9
Bound	0.5	20.4	2.5	5.5	9.9	24.2
Rotation 2						
F	ND	ND	ND	ND	ND	ND
FSO	1.4	3.1	7.2	19.1	5.3	1.1
FSO ₂	1.8	3.6	6.1	18.6	8.6	2.9
FSOP	0.3	0.4	0.2	10.3	3.3	2.2
FSO ₂ P	7.2	7.2	3.2	24.6	6.3	4.9

Compound	Radioactivity, % of TRR in sample					
	Swiss chard	Red beets		Wheat		
		Roots	Tops	Forage	Straw	Grain
OH-FSO ₂ P	1.3	4.6	ND	1.5	0.4	2.6
FSOP-glu	ND	8.8	10.4	0.9	8.1	2.6
FSO ₂ P-glu	2.5	ND	8.6	2.0	9.4	8.0
FSO ₂ P-conj	24.7	3.8	14.5	1.6	3.1	2.8
DAFSO	37.3	ND	5.6	3.3	ND	ND
Subtotal	76.5	31.5	55.8	81.9	44.5	27.1
Unidentified	14.0	38.8	14.4	2.5	9.6	5.6
Bound	9.5	29.8	10.7	3.4	11.7	67.7
Rotation 3						
F	0.1	ND	0.1	ND	<0.1	0.1
FSO	4.1	1.7	4.5	26.1	4.5	1.0
FSO ₂	5.8	2.1	4.6	29.1	8.5	1.9
FSOP	0.5	ND	0.4	5.8	1.1	0.2
FSO ₂ P	4.5	2.3	3.0	21.8	2.2	1.5
OH-FSO ₂ P	ND	1.4	ND	5.0	ND	0.6
FSOP-glu	ND	10.5	6.2	0.5	6.4	ND
FSO ₂ P-glu	2.8	2.8	10.9	0.5	11.6	5.1
FSO ₂ P-conj	25.0	7.9	11.5	0.3	3.1	1.9
DAFSO	28.5	ND	ND	ND	ND	1.9
Subtotal	71.3	28.7	41.2	89.1	37.4	14.2
Unidentified	18.0	25.4	42.2	2.0	6.4	5.2
Bound	10.9	45.9	16.7	9.0	20.6	78.6

In general there is an overall decrease in the levels of identified residues with time, although in Swiss chard the levels of the FSO₂P conjugates increase with each rotation. The percentages of bound residues increase with time in all the crops.

Nemacur 3 was sprayed onto soil at three sites at a rate equivalent to 6.72 kg ai/ha and incorporated immediately after application (Pither, 1991). Rotational crops were planted 1, 4 and 8 months after the soil treatment (plant-back period). Representative crops comprised cereals and root and leafy vegetables. All crops were sampled at harvest, and immature forage samples were collected approximately 45 days after planting. Soil core samples (0-15.2 and 15.2-30.5 cm depth) were taken immediately after treatment, at planting and at harvest. Residues in soil were determined on a dry weight basis. The results are shown in Tables 34-36.

Table 34. Fenamiphos residues in cereals planted as rotational crops.

Crop/Sample	Plant-back period, months	Residue, mg/kg		
		Mississippi	Texas	Kansas
Wheat				
forage	1	0.75, 0.88	0.02	
grain		<0.01	NA	
straw		0.18	<0.01	
forage	4		<0.01	
grain			<0.01	
straw			<0.01	
Sorghum				
forage	1			0.05
grain				<0.01
straw				0.03
forage	4	0.44, 0.68		0.01
grain		<0.01		<0.01
straw		0.02		<0.01

Crop/Sample	Plant-back period, months	Residue, mg/kg		
		Mississippi	Texas	Kansas
forage	8	<0.01		
grain		<0.01		
straw		<0.01		

Table 35. Fenamiphos residues in root and leafy vegetables planted as rotational crops.

Crop/Sample	Plant-back period, months	Residue, mg/kg		
		Mississippi	Texas	Kansas
Turnip				
tops	1	0.05	<0.01	0.02
roots		<0.01	<0.01	<0.01
tops	4		<0.01	<0.01
roots			<0.01	<0.01
Spinach leaves	1	0.02		0.10
	4			0.03
Mustard leaves	1		0.03	
	4		<0.01	

Table 36. Fenamiphos residues in soil at intervals before cropping.

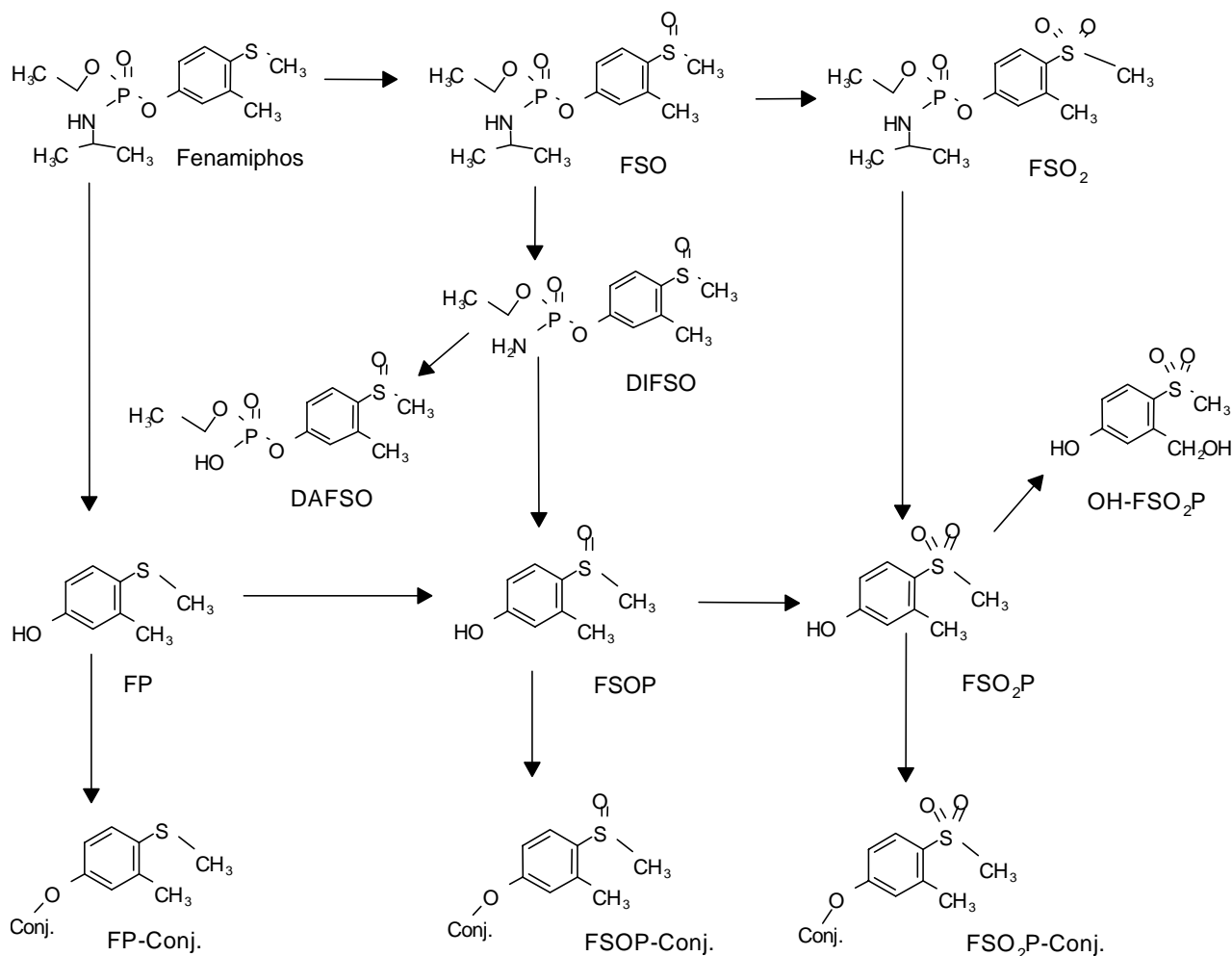
Soil core depth, cm	Plant-back period, months	Residue, mg/kg		
		Mississippi	Texas	Kansas
Cereal plots	1			
0-15.2		0.61	0.15	1.55
15.2-30.5		<0.01	<0.01	<0.01
Leafy plots				
0-15.2		0.73	0.24	0.79
15.2-30.5		<0.01	<0.01	<0.01
Root plot				
0-15.2		1.13	0.33	1.21
15.2-30.5		<0.01	<0.01	<0.01
Cereal plots	4			
0-15.2		0.96	<0.01	0.43
15.2-30.5		0.34	<0.01	0.01
Leafy plot				
0-15.2			<0.01	0.26
15.2-30.5			<0.01	<0.01
Root plot				
0-15.2			<0.01	0.39
15.2-30.5			<0.01	<0.01
Cereal plots	8			
0-15.2		0.56		
15.2-30.5		0.16		

Residues were present in the top core soil samples but were below the limit of detection in most of the lower cores. The results show that there is a correlation between residues in the top layer of the soil and the residues found in cereal forage, turnip tops and leafy vegetables planted 1 and 4 months after treatment.

In summary, the metabolism and crop rotation studies show similar results. After treatment of a crop, or planting in a first rotation after soil treatment, the major transformation of fenamiphos is via formation of the sulfoxide and sulfone, which are both soluble in organic solvents. As the crop matures or is planted in a second rotation, fenamiphos sulfoxide and sulfone are converted to their phenol conjugates after cleavage of the phosphate ester bond. Both phenol conjugates are extractable

in aqueous solvents. The transient metabolites DIFSO and DAFSO were not found in the metabolism studies but DAFSO was isolated in the rotation studies, and both may be envisaged as intermediates formed before the phenols. Proposed transformation pathways are shown in Figure 2.

Figure 2. Proposed metabolic pathway of fenamiphos in treated and rotational plants.



F	fenamiphos	FSO	fenamiphos sulfone
FSO ₂	fenamiphos sulfone	DAFSO	desamino-fenamiphos sulfoxide
DIFSO	desisopropyl fenamiphos sulfoxide	FP	fenamiphos phenol
FSOP	fenamiphos sulfoxide phenol	FSO ₂ P	fenamiphos sulfone phenol
FP-Conj.	fenamiphos phenol conjugate	FSO ₂ P-Conj.	fenamiphos sulfone phenol conjugate
FSOP-Conj.	fenamiphos sulfoxide phenol conjugate		
OH-FSO ₂ P	fenamiphos sulfone phenol, hydroxylated at the 3-methyl group		

Environmental fate in soil

Photolysis

Samples of sandy loam soil (0.5 g) were treated with 10 µg [1-*phenyl*-¹⁴C]fenamiphos and irradiated for 0, 1, 2, 4, 6, 12, 24 or 48 hours before radioassay (Dime *et al.*, 1983). The samples were extracted with acetone/water and centrifuged, and the supernatant was radioassayed. The radioactive compounds were further characterized by TLC.

The half-life was calculated to be 1.6 hours ($k_1 = 0.42 \text{ h}^{-1}$); fenamiphos was stable in the control soil. Approximately 99% of the radioactivity was extracted from both control and irradiated soils, except from the 48-hour sample where the recovery was 90%. The radioactivity during the 48 hours was due to fenamiphos, ranging from 7 to 91%, fenamiphos sulfoxide from 5 to 64%, and fenamiphos sulfone from 0.5 to 6.6% of the TRR.

In a similar study (Hanlon, 1988) [$^{13,14}\text{C}$]fenamiphos was applied to sandy loam soil at a concentration of 9.14 mg/kg. Samples were exposed to natural sunlight for 0, 1, 2, 3 or 4 hours after treatment, and control samples were kept in the dark for similar periods. All samples were extracted with acetonitrile/water and centrifuged, and the supernatant was assayed by liquid scintillation counting. Compounds were identified by HPLC. Extracted radioactivity ranged from 99.6% at 0 hours to 98.2% at 4 hours. The calculated half-life was reported as 2.7 hours, with a first-order rate constant of 0.25 h^{-1} . Fenamiphos accounted for 99.6 to 33.8% of the extracted radioactivity from 0 to 4 hours after treatment and fenamiphos sulfoxide for 34.8% at 1 hour to 64.4% at 4 hours.

In summary, the soil photolysis studies show that fenamiphos is rapidly degraded to fenamiphos sulfoxide according to first-order kinetics, with half-lives of 1.6 hours under laboratory conditions and 2.7 hours in natural sunlight.

Adsorption/desorption

The adsorption and desorption of labelled fenamiphos on sand, sandy loam, silt loam and clay loam was investigated by Daly (1988). Solutions of 250, 187, 125 and 25 $\mu\text{g/ml}$ of [1-*phenyl*- ^{14}C]fenamiphos were prepared in 0.01 molar CaCl_2 solution. Liquid scintillation counting was used to measure the concentrations of radioactivity in the aqueous solutions, and HPLC to determine the radiochemical purity of the [^{14}C]fenamiphos in the CaCl_2 solution at the beginning and end of the test period.

To 3 g samples of each soil were added 10 ml aliquots of each concentration of fenamiphos solution and the suspensions were shaken in darkness for 8 hours at 25°C . The soil suspensions were then centrifuged and the supernatants removed by decantation after the pH had been measured; aliquots were taken for LSC. For the desorption phase, aliquots of 0.01 molar CaCl_2 were added to each sample according to the volume removed after the adsorption experiment. The suspensions were shaken in darkness for 24 hours at 25°C , centrifuged, the supernatants removed, and the volumes measured. The wet soil was combusted for radioanalysis and the supernatant taken for liquid scintillation counting.

HPLC analyses of the supernatants from soils treated with the 250 $\mu\text{g/ml}$ solution showed that fenamiphos was stable, with [^{14}C]fenamiphos present at 97.8%, 98%, 97.4% and 97.6% of its initial level in sand, sandy loam, silt loam and clay loam respectively after 8 hours, and recoveries of total ^{14}C from the sand, sandy loam, silt loam and clay loam 99.4%, 102%, 104% and 111% respectively.

The adsorption constants (K_{oc}) and Freundlich constants (K_d , n) were calculated (Table 37).

Table 37. Adsorption and desorption of [^{14}C]fenamiphos (Daly, 1988).

Soil	% Organic C	Adsorption		Desorption	
		K_d	K_{oc}	K_d	K_{oc}
Sand	0.53	2.86	543.4	2.61	496.3
Sandy loam	0.58	0.96	165.6	0.68	117.9
Silt loam	1.53	3.46	226.5	4.29	281.3
Clay loam	1.16	1.98	171.0	1.47	127.1

The mobility of a compound is directly related to its adsorption properties, and K_{oc} is used to rank chemicals with respect to their leaching potential:

K_{oc}	>5000	immobile in soil
	2000-5000	slight mobility
	500-2000	low mobility
	150-500	medium mobility
	50-150	high mobility

From the adsorption K_{oc} values the mobility of fenamiphos is estimated to be low in sand and medium in sandy loam, silt loam and clay loam.

The investigation of the behaviour of fenamiphos in 16 soils from different locations was the subject of a Ph.D. dissertation (Simon, 1990). The degradation of fenamiphos in the soils was investigated, and the adsorption of fenamiphos, fenamiphos phenol sulfoxide and fenamiphos phenol sulfone was measured. The soils and their physicochemical properties are tabulated below.

Table 38. Properties of soils investigated (Simon, 1990).

Location	Clay, %	Silt, %	Sand, %	H ₂ O capacity	pH (CaCl ₂)	pH (H ₂ O)	% organic C
Canada	22.7	47.6	29.7	43.5	6.8	7.27	6.52
Sweden	8.5	9.0	82.5	11.7	6.6	6.33	1.23
Germany/Puch	14.1	74.9	11.0	25.7	6.4	6.98	1.21
Germany/Speyer	4.5	14.1	81.4	18.3	5.1	6.45	2.22
Netherlands	19.3	58.0	22.7	23.3	6.6	6.60	1.60
France	28.1	39.9	32.0	22.8	7.5	7.96	1.58
USA/Indiana	12.0	25.6	62.4	12.1	6.5	6.42	0.95
USA/Nebraska	27.0	69.8	3.2	30.8	5.3	6.68	1.51
Japan/Toyoda	9.8	48.1	42.1	58.3	5.9	5.97	3.53
USA/Florida	1.3	3.3	95.4	5.2	5.8	6.58	0.77
Costa Rica	29.6	41.6	28.8	51.8	4.9	6.03	4.76
Brazil/P. Fundo	44.4	24.4	31.2	23.1	4.8	5.79	1.63
Brazil/Parana	53.5	30.9	15.6	31.3	7.0	6.52	2.28
Thailand	55.3	43.0	1.7	37.0	4.5	5.72	1.63
Phillipines	15.2	42.3	42.5	24.9	5.5	5.83	0.73
Japan/Tsurug.	10.6	47.2	42.2	59.8	5.9	7.10	3.58

The labelled compounds were [1-*phenyl*-^{13/14}C]fenamiphos, [1-*phenyl*-¹⁴C]fenamiphos phenol, [1-*phenyl*-¹⁴C]fenamiphos sulfoxide phenol and [1-*phenyl*-¹⁴C]fenamiphos sulfone phenol. Concentrations of 0.5, 1, 2, 5 and 10 mg/l of fenamiphos in 0.01 molar CaCl₂ were prepared and 10 ml of each solution was added to 2 g of each soil. The samples were shaken for 4 hours at room temperature, then centrifuged. Aliquots of 0.5 ml of each supernatant were taken for liquid scintillation counting. The experiments with fenamiphos phenol sulfoxide and fenamiphos phenol sulfone were conducted in the same way with concentrations of 0.1, 0.5, 1, 2 and 5 mg/l.

Table 39. Calculated adsorption isotherms, adsorption constants (K_{oc}) and Freundlich constants (K_f).

Soil	Fenamiphos		Fenamiphos sulfoxide phenol		Fenamiphos sulfone phenol	
	K_f	K_{oc}	K_f	K_{oc}	K_f	K_{oc}
Canada	19.42	297.9	6.97	82.6	8.67	133.1
Sweden	3.43	279.7	0.15	12.5	0.61	49.4
Germany/Puch	1.35	111.6	0.35	28.8	0.57	47.2
Germany/Speyer	3.96	178.4	0.99	44.5	1.28	57.7
Netherlands	5.76	380.0	0.66	41.3	1.06	66.1
France	2.52	159.5	0.40	25.7	0.64	40.4
USA/Indiana	2.22	234.7	0.82	86.4	0.98	103.4
USA/Nebraska	4.72	312.6	1.78	118.3	2.31	152.9
Japan/Toyoda	2.69	76.2	0.96	27.2	1.09	31.0
USA/Florida	1.74	226.0	1.01	132.1	1.13	146.8
Costa Rica	17.30	363.4	7.88	165.6	9.83	206.6
Brazil/P. Fundo	2.28	140.5	0.92	56.3	1.11	68.0

Soil	Fenamiphos		Fenamiphos sulfoxide phenol		Fenamiphos sulfone phenol	
	K _f	K _{oc}	K _f	K _{oc}	K _f	K _{oc}
Brazil/Parana	6.02	264.1	2.77	121.7	3.70	162.4
Thailand	23.34	1432	1.67	102.4	3.23	198.5
Phillipines	2.47	339.7	0.33	44.7	0.38	52.0
Japan/Tsurugashima	7.27	205.9	3.89	108.8	4.35	121.5

The soil from Thailand with a high clay and silt content showed the highest adsorption of fenamiphos. The soils from Canada and Costa Rica showed high K_f values for fenamiphos and the two metabolites, with the highest for both metabolites in the Costa Rica soil. In general, the adsorption constants of fenamiphos sulfoxide phenol and fenamiphos sulfone phenol were lower than those of fenamiphos.

The adsorption and desorption of fenamiphos sulfoxide and fenamiphos sulfone on a clay loam from France and a silt loam from The Netherlands was investigated by Fent (1995a,b). The characteristics of the two soils are shown below.

	Netherlands	France
Clay	19.3%	28.1%
Silt	58.0%	39.9%
Sand	22.7%	32.0%
Organic C	1.6%	1.58%
pH (H ₂ O)	6.6	8.0

The soils (12 g) were treated with [¹⁴C]fenamiphos sulfoxide and sulfone at concentrations of 5.09, 1.10, 0.20 and 0.04 mg/l in 0.01 M CaCl₂ and 0.00018 M biocide and the mixtures shaken for 1, 5, 9, 24, 48, 53 or 72 hours at 22°C. After treatment, the soil mixtures were centrifuged and ¹⁴C was determined in the supernatants by liquid scintillation counting and in the soil by LSC after combustion; the purity of the radioactive substances was checked by TLC. To measure desorption the remaining soil from each test was suspended in 0.01 molar CaCl₂ and shaken for the same time as for the adsorption measurement. The radioactivity in the supernatant was determined after centrifugation. The results are shown in Table 40.

Table 40. Adsorption and desorption of fenamiphos sulfoxide and sulfone (Fent, 1995a,b).

Soil	Fenamiphos sulfoxide		Fenamiphos sulfone	
	K _d	K _{oc}	K _d	K _{oc}
Adsorption				
Silt loam (Netherlands)	3.60	225	4.98	311
Clay loam (France)	0.71	44.8	1.04	66
Desorption				
Silt loam	3.10	194	4.58	286
Clay loam	1.12	71.2	1.64	104

The results showed that the proportions of fenamiphos sulfoxide adsorbed to The Netherlands soil were 67.7 to 70.7% of the applied concentration and to the French soil 27% to 36.8%. The corresponding percentages of fenamiphos sulfone adsorbed were 74.3 to 77.3% and 35.2 to 47.1%. Analysis indicated that >96% of the measured radioactivity in both soil supernatants was due to fenamiphos sulfoxide and >99% and 88% of the ¹⁴C in the silt and clay loam soils respectively was accounted for by fenamiphos sulfone.

Desorption ranged from 34.5% to 64.3% of the applied concentrations of fenamiphos sulfoxide and 23.4 to 55.7% of the applied concentrations of fenamiphos sulfone.

Mobility. The leaching of aged residues of [1-*phenyl*-¹⁴C]fenamiphos incorporated into a sandy loam and a silt loam soil at a concentration equivalent to 10 kg ai/ha was investigated by Spiteller (1987). The soils were weighed into incubation vessels and water added to adjust the moisture content to 40%. The vessels were fitted with traps for ¹⁴CO₂ and other volatiles and stored at 21°C for 15, 30 or 63 days. After ageing, the samples were extracted with acetone/H₂O followed by CH₃Cl/MeOH and centrifuged. The supernatants were analysed by TLC and the unextracted residues by combustion and LSC.

In the leaching experiments, columns of each soil were prepared and saturated with water. The corresponding soil containing the aged residues was added to the top of each column, the columns were watered evenly for 48 hours, and the leachate was collected in fractions. The columns were drained, and the soil was divided into segments and analysed for radioactivity. The leachate fractions were centrifuged and ¹⁴C in the supernatant was measured by LSC. Fractions containing more than 1% of the applied radioactivity were worked up further. The extracts were partitioned into CH₃Cl and analysed by TLC.

Table 41. Distribution of residue components in aged soils (Spiteller, 1987).

Compound	¹⁴ C, % of applied, after ageing period (days)					
	Silt loam			Sand loam		
	0	15	63	0	15	63
F	80.3	8.5	2.6	78.5	23.9	5.0
FSO	6.5	39.6	19.1	5.1	57.3	49.0
FSO ₂		24.5	20.8		6.6	16.5
FP						
FSOP			1.0			2.8
FSO ₂ P		7.8	16.8			6.7
Unidentified	7.5	4.3	6.6	10.6	6.6	4.6

Less than 1% of the applied radioactivity was found in the eluates from the silt soil aged for 0, 15 or 63 days, but 4.3 and 15.9% of the applied radioactivity was recovered in the volatile traps after 15 and 63 days respectively. From the sandy soil, 3.5%, 56.8% and 52.9% of the applied radioactivity was recovered in the eluate fractions after 0, 15 and 63 days ageing respectively.

Table 42. Radioactive compounds in the organic and aqueous fractions from extracts of the leachates, expressed as a percentage of applied radioactivity.

Compound	Organic			Aqueous		
	0 days	15 days	63 days	0 days	15 days	63 days
F						
FSO	2.7	47.9	33.3			0.2
FSO ₂		4.7	7.7			0.1
FP						
FSOP					0.2	0.7
FSO ₂ P						0.2
Unidentified	0.4	2.8	6.2		1.1	1.1

The results show that fenamiphos is transformed mainly into fenamiphos sulfoxide and sulfone. Although fenamiphos phenol was not found, fenamiphos phenol sulfoxide and sulfone were both found after 63 days ageing.

In a subsequent laboratory leaching study (Mulford, 1987a), a sandy loam soil from Kansas was treated with [1-*phenyl*-^{13,14}C]fenamiphos at a rate of 11 mg/kg and incubated for 30 days at 22-24°C under aerobic conditions. The moisture content of the soil was increased to 75% and volatiles were collected in NaOH traps. Sub-samples of the treated soil were analysed for radioactivity by combustion analysis followed by liquid scintillation counting.

The main radioactive compounds in the aged soil were fenamiphos and fenamiphos sulfoxide, accounting for 45.6 and 46.9% of the applied radioactivity respectively. The remaining radioactivity was due to fenamiphos sulfone, fenamiphos phenol sulfoxide and fenamiphos phenol sulfone, contributing 2.3, 2.7% and 1% of the applied radioactivity respectively.

Three soils were used in the leaching study, a Californian sandy loam, sand from Indiana and the Kansas sandy loam. The aged soil treated with fenamiphos was introduced into columns containing the three soils at a rate equivalent to 22.3 kg ai/ha. The samples were stored frozen for 15 days before leaching and prepared by saturating with 0.01 molar CaCl₂ solution before adding the aged soil. Each column was then leached continuously for 2 days with 1160 ml of 0.01 molar CaCl₂. The leachate was collected in fractions and assayed by liquid scintillation counting. The fractions were combined and extracted with CH₂Cl₂/CH₃CN (2:1). The organic extracts were concentrated and analysed by TLC and HPLC. After leaching the soils were sectioned, dried and assayed by combustion and liquid scintillation counting. The results are shown in Table 43.

Table 43. Properties of three leached soils and distribution of residues in the extracted leachate (Mulford, 1987a).

Properties	Soil		
	California sandy loam	Indiana sand	Kansas sandy loam
% sand	69	90	66
% silt	21	8	32
% clay	10	2	2
% organic C	1.2	0.8	2.4
pH (0.01 molar CaCl ₂)	5.4	4.3	5.1
CEC (meq/100 g)	12	6	17
Particle density (g/cm ³)	2.6	2.6	2.6
	% of applied ¹⁴ C as		
F	1.0	8.6	0
FSO	40.8	48.8	14.6
FSO ₂	2.0	2.5	0.7
FSOP	1.9	2.1	0.3
FSO ₂ P	0.7	0.8	0
Unidentified	0	0	0.3
Aqueous fraction	0.8	1.0	0.4
Total	47.2	63.8	16.2

The predominant radioactive component in the leachates was fenamiphos sulfoxide, with minor amounts of fenamiphos sulfone and the phenol derivatives. Fenamiphos was present in the Indiana and California soils. The results are in agreement with the study reported by Spittler.

In the three soils from the leaching experiment, the remaining radioactivity was from fenamiphos and fenamiphos sulfoxide, with the levels decreasing down the column. Overall, the data show that not all of the adsorbed fenamiphos and fenamiphos sulfoxide was leached from the soils.

Degradation

The degradation of [1-*phenyl*-¹⁴C]fenamiphos in Florida sand was investigated under aerobic conditions (Lane and Clay, 1989). Soil samples were treated with fenamiphos at 18 mg/kg and mixed thoroughly. The moisture contents were increased to 75% by addition of water. Samples taken immediately after application and after 1, 3, 7, 14, 21, 28, 59, 86 and 120 days were extracted with CH₃CN/H₂O (7:3) at room temperature and the CH₃CN evaporated. The remaining aqueous solution was partitioned with CH₂Cl₂/CH₃CN (2:1), evaporated, and assayed by liquid scintillation counting. The residues were characterized by TLC. The half-life of fenamiphos was calculated to be 30 days.

The results are shown in Table 44. At 120 days after treatment, 7.8% of fenamiphos remained in the soil.

The soil properties are summarized below.

% sand	93
% silt	1
% clay	6
% organic C	1.8
pH	4.9
CEC (meq/100 g in 0.01 molar sodium acetate at pH 8.2)	5
density (g/cm ³)	2.6

Table 44. Residues in Florida sand soil after aerobic degradation (Lane and Clay, 1989).

Compound	% of applied ¹⁴ C at days after application									
	0	1	3	7	14	21	28	59	86	120
F	97.8	97.8	93.5	82.4	68.4	54.0	47.9	24.5	9.8	7.8
FSO	1.0	3.2	8.3	15.3	24.8	30.2	33.6	35.1	41.4	27.3
FSO ₂			0.5	2.0	4.4	9.5	11.9	19.2	21.8	22.3
FSOP				0.7	1.1	1.6	1.7	3.5	2.4	1.5
FSO ₂ P					0.7	1.1	2.9	11.0	14.1	14.0
H ₂ O-soluble	0.4	0.3	0.4	0.5	0.3	0.6	0.6	1.2	1.4	1.7
Origin/diffuse	0.7	0.7	0.3	0.2	0.5	0.4	0.3	0.3	0.5	1.2
Solids	0.1	0.6	0.9	1.7	2.1	3.0	3.4	6.9	12.7	17.4
Total	100	102.6	103.9	102.8	102.3	100.4	102.3	101.7	104.1	93.2

Fenamiphos was largely degraded to fenamiphos sulfoxide and fenamiphos sulfone during the 120 days of the study. Fenamiphos phenol sulfone was found at 11 to 14% of the applied radioactivity after 59-120 days. The results are in agreement with those previously found for the degradation of fenamiphos in aged soils.

In a degradation study with a sandy loam soil (Spiteller, 1989a) [1-*phenyl*-^{13,14}C]fenamiphos was applied at a rate equivalent to 10 kg ai/ha. The moisture content of the soil was adjusted to 75% and the samples were maintained under aerobic conditions for 6 days with traps for the collection of ¹⁴CO₂. The systems were then made anaerobic by purging with nitrogen and sampled 20, 36, 52 and 66 days after application of the fenamiphos.

The samples were extracted by sonification with acetone/MeOH (1:1) followed by CH₃Cl/MeOH. After each extraction, the samples were centrifuged and the supernatants characterized by TLC and HPLC. The remaining insoluble residues and soil samples were combusted to determine the radioactivity.

The half-life for the anaerobic phase of the study was calculated to be 87.9 days.

Table 45. Residues in treated sandy loam soil after aerobic and anaerobic incubation (Spiteller, 1989a).

Compound	% of applied radioactivity at days after application					
	0	6	20 (14)	36 (30)	52 (46)	66 (60)
F	93.3	36.3	28.6	27.1	23.9	22.2
FSO	3.5	46.5	44.9	39.7	28.8	25.8
FSO ₂	<0.1	<0.1	1.1	0.6	0.8	0.9
FP	<0.1	<0.1	0.4	<0.1	4.2	3.2
FSOP	<0.1	2.5	5.8	5.8	3.2	2.6
FSO ₂ P	<0.1	<0.1	3.5	7.1	10.1	12.8

Unknown	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Total	96.8	85.3	84.3	80.2	71.0	67.5

Figures in parentheses show days under anaerobic conditions.

The results show that the degradation of fenamiphos is slow under anaerobic conditions. This is not unexpected as the degradation of fenamiphos yields mainly oxidation products. The half-life in the aerobic degradation study of Lane and Clay was reported as 30 days, whereas under anaerobic conditions it was almost 90 days.

In a companion study of aerobic degradation (Spiteller, 1989b), [1-*phenyl*-^{13,14}C]fenamiphos was applied to a sandy loam at a rate equivalent to 10 kg ai/ha and monitored for 365 days. The soil properties are shown below.

% sand	65.5
% silt	26.3
% clay	8.2
% organic C	1.29
pH	6.8
CEC (meq/100 g in 0.01 molar sodium acetate, pH 8.2)	10
density (g/cm ³)	2.24

The experimental details were as described above and treated soil samples were analysed 0, 1, 3, 7, 14, 31, 63, 100, 123, 184, 274 and 365 days after application.

The distribution of fenamiphos and its degradation products in the extracted samples at various times is shown in Table 46, which shows that degradation was much faster under aerobic than under anaerobic conditions.

Table 46. Distribution of radioactive residues after aerobic degradation of fenamiphos in sandy loam soil (Spiteller, 1989b).

DAT	% of applied radioactivity									
	F	FSO	FSO ₂	FP	FSOP	FSO ₂ P	FSO ₂ A	Total	Unextracted	Volatiles
0	90.0	7.6	<0.1	<0.1	<0.1	<0.1	<0.1	97.6	3.4	nd
1	75.8	18.1	<0.1	<0.1	<0.1	<0.1	<0.1	93.9	4.9	<0.1
3	53.8	34.4	0.6	<0.1	1.0	<0.1	<0.1	89.8	9.1	0.1
7	28.5	48.1	2.4	<0.1	2.5	2.1	<0.1	83.6	11.6	0.4
14	13.3	51.4	3.5	<0.1	4.6	6.8	<0.1	79.6	16.2	1.1
31	4.0	36.9	2.3	<0.1	5.4	17.5	0.7	66.8	23.8	4.2
63	1.7	17.7	1.2	<0.1	3.0	24.3	1.9	49.8	38.2	10.5
100	1.1	9.3	0.6	<0.1	2.0	21.3	2.7	37.0	44.3	16.6
123	1.0	7.5	0.2	<0.1	1.5	19.7	3.0	32.9	48.4	19.2
184	0.7	4.0	<0.1	<0.1	1.1	13.9	4.3	24.0	49.4	23.8
274	0.5	1.5	<0.1	<0.1	1.0	7.7	4.3	15.0	51.1	31.8
365	0.4	1.0	<0.1	<0.1	1.1	6.6	4.4	13.5	50.5	34.2

FSO₂A: fenamiphos sulfone anisole

The proportion of extractable radioactivity decreases with time and that of volatile and unextractable materials increases. The results show that the degradation of fenamiphos proceeds via the formation of fenamiphos sulfoxide, followed by fenamiphos sulfoxide phenol and fenamiphos sulfone phenol. Most of the radioactivity after 100 days was unextractable. The main pathways of degradation in soil are oxidation at the methylthio group followed by cleavage of the phosphate bond with the formation of phenol derivatives (Figure 3).

The degradation of fenamiphos was studied in several soils from different geographical locations (Simon, 1990; Simon *et al.*, 1992). The properties and origins of the soils are given in Table 38 above. [1-*phenyl*-^{13/14}C]fenamiphos was applied to the soils at a rate equivalent to 10 kg ai/ha (0.77 mg/100 g sample). Control samples were stored in the dark at 22°C, while the test samples were incubated at either 16 and 22°C or 22 and 28°C to represent cool/moderate or subtropical/tropical climates. The samples were incubated for 15, 50 or 90 days.

The extraction procedures included sonication with 1:1 acetone and MeOH followed by 1:1 CH₃Cl and MeOH, centrifugation of each solvent mixture and liquid scintillation counting, with TLC and GC-MS of the organic extracts and combustion analysis of the remaining dry soil samples. The results are shown in Tables 47 and 48 for the cool/moderate climate and subtropical/tropical climate respectively.

Table 47. Aerobic degradation of fenamiphos in nine soils from cool to temperate geographic regions: recovery and distribution of applied radioactivity at 16 and 22°C (Simon *et al.*, 1992)

°C	Days	Radioactivity, % of applied (mean of triplicate samples)										
		Total extracted ¹	F	FSO	FSO ₂	FSOP	FSO ₂ P	FSO ₂ A	TTR ²	Un-extracted	¹⁴ CO ₂	Recovery
Canada (I)												
22	15	74.5	14.6	45.0	6.2	3.9	4.8	--	65.8	18.5	1.3	94.3
	50	53.5	2.9	24.3	6.3	3.7	14.9	1.4	33.5	36.2	8.3	98.0
	90	45.3	2.2	27.7	4.3	2.7	16.7	2.7	34.2	34.8	17.0	97.1
16	15	79.9	33.5	41.2	2.1	2.1	1.0	--	76.8	14.8	0.6	95.3
	50	70.1	10.0	37.8	7.2	4.8	10.3	--	55.0	21.5	3.4	95.0
	90	54.3	5.3	25.4	4.5	3.4	14.4	1.3	35.2	31.6	10.4	96.3
Sweden (II)												
22	15	85.7	7.8	65.3	9.0	1.3	2.3	--	82.1	6.3	1.7	93.7
	50	70.8	1.5	35.6	19.0	1.1	11.9	1.7	56.1	12.2	8.1	91.1
	90	55.7	0.8	21.0	14.1	0.9	14.8	3.9	35.9	18.5	16.2	90.4
16	15	87.8	18.0	65.7	3.6	0.5	--	--	87.3	3.7	0.6	92.1
	50	81.5	8.6	53.9	11.7	2.1	5.2	--	74.2	8.1	3.1	92.7
	90	77.7	4.9	49.8	14.3	2.0	6.0	0.7	69.0	9.1	4.8	91.6
Germany/Bavaria (III)												
22	15	77.4	2.6	56.6	5.3	2.6	10.3	--	64.5	19.8	4.0	101.2
	50	41.8	0.1	24.1	2.1	0.8	12.3	2.4	26.3	40.3	21.2	103.2
	90	19.2	0.4	7.1	0.5	0.4	7.0	3.3	8.0	48.2	32.9	100.3
16	15	79.8	3.9	66.8	3.0	2.4	3.7	--	73.7	12.7	1.2	93.7
	50	52.6	0.2	29.6	3.1	1.1	17.3	1.3	32.9	23.7	11.3	87.6
	90	33.9	0.5	12.6	1.2	0.4	17.0	1.7	14.3	35.1	21.0	90.0
Germany/Rheinland Pfalz (IV)												
22	15	84.5	15.7	52.3	4.3	7.6	4.1	--	72.3	10.1	2.0	96.6
	50	68.4	2.3	34.9	5.9	11.1	12.3	1.2	43.1	19.8	8.7	96.9
	90	57.7	0.8	23.4	5.2	10.1	16.2	2.0	29.4	25.4	13.2	96.3
16	15	86.5	24.0	54.2	2.5	4.2	0.9	--	80.7	8.5	0.1	95.1
	50	74.9	4.4	44.0	7.4	9.1	8.9	0.5	55.8	13.6	4.1	92.6
	90	66.6	1.7	28.7	7.3	8.1	18.4	1.7	37.7	20.6	9.0	96.2
Netherlands (V)												
22	15	89.3	2.6	68.1	9.6	1.1	7.9	--	80.3	12.7	3.4	105.4
	50	49.4	0.3	24.3	5.7	0.7	14.9	3.1	30.3	29.5	21.8	100.7
	90	22.0	0.5	10.8	2.2	0.4	5.2	2.9	13.5	33.8	39.0	94.8
16	15	90.1	5.1	79.1	3.9	--	2.0	--	88.1	9.0	1.1	100.2
	50	72.5	1.7	37.8	11.6	1.2	19.0	1.2	51.1	18.3	7.8	98.6
	90	42.7	--	18.7	4.7	--	15.8	3.5	23.4	28.6	23.4	94.7
France (VI)												
22	15	65.3	5.5	36.8	1.8	5.4	14.2	0.8	44.1	29.8	2.5	97.6
	50	23.1	1.1	4.8	0.3	0.8	12.2	3.3	6.2	69.1	16.8	109.0

°C	Days	Radioactivity, % of applied (mean of triplicate samples)										
		Total extracted ¹	F	FSO	FSO ₂	FSOP	FSO ₂ P	FSO ₂ A	TTR ²	Un-extracted	¹⁴ CO ₂	Recovery
	90	10.5	0.6	1.5	0.2	0.2	2.9	3.7	2.3	61.8	32.2	05.5
16	15	79.4	6.8	52.9	0.9	4.4	14.4	--	60.6	23.3	1.6	104.3
	50	52.2	3.8	27.7	0.8	2.8	16.0	1.1	32.3	42.5	7.2	101.9
	90	47.3	3.9	22.4	0.6	2.8	15.2	1.5	26.9	46.4	9.8	104.0
USA/Indiana (VII)												
22	15	79.2	7.3	47.3	6.4	3.7	14.5	--	61.0	18.4	1.5	99.1
	50	56.6	1.3	25.5	6.6	2.1	19.5	1.6	33.4	29.1	7.7	93.4
	90	45.9	1.1	12.5	2.5	1.8	24.7	3.3	16.1	33.8	14.0	93.7
16	15	90.8	24.7	58.6	2.6	2.3	2.6	--	85.9	11.7	0.8	103.3
	50	77.8	13.1	48.7	4.8	3.2	8.0	--	66.6	19.6	2.8	100.2
	90	72.6	4.4	45.5	5.4	4.3	12.3	0.7	55.3	25.9	4.1	102.6
USA/Nebraska (VIII)												
22	15	70.7	6.1	58.1	5.5	1.0	--	--	69.7	13.1	1.0	84.8
	50	67.9	2.1	33.8	22.6	1.6	7.1	0.7	58.5	23.5	6.6	98.0
	90	58.0	0.8	23.4	19.2	1.4	11.5	1.7	43.4	26.3	12.8	97.1
16	15	79.4	12.3	65.5	1.6	--	--	--	79.4	13.0	0.4	92.8
	50	70.9	3.8	54.3	11.4	0.8	0.6	--	69.5	14.9	1.7	87.5
	90	71.0	2.2	37.8	22.8	1.8	6.4	--	62.8	18.9	4.8	94.7
Japan/Toyoda (IX)												
22	15	82.9	48.5	34.4	--	--	--	--	82.9	17.5	0.1	100.5
	50	76.7	17.6	48.9	7.1	2.0	1.1	--	73.6	25.6	0.3	102.6
	90	73.4	7.3	45.6	13.9	2.8	4.1	--	66.8	35.5	1.1	110.0
16	15	86.9	69.1	17.8	--	--	--	--	86.9	10.4	--	97.3
	50	85.2	34.7	48.8	0.6	1.1	--	--	84.1	19.0	0.1	104.3
	90	80.3	24.8	49.3	3.1	2.5	0.6	--	77.2	23.9	0.4	104.6

¹ Including polar products (1%)² Total toxic residues (F + FSO + FSO₂)

-- not detected

F fenamiphos

FSO fenamiphos sulfoxide

FSO₂ fenamiphos sulfone

FSOP fenamiphos sulfoxide phenol

FSO₂P fenamiphos sulfone phenolFSO₂A fenamiphos sulfone anisoleTable 48. Aerobic degradation of fenamiphos in seven soils from subtropical and tropical geographic regions: recovery and distribution of applied radioactivity at 22 and 28°C (Simon *et al.*, 1992)

°C	Days	Radioactivity, % of applied (mean of triplicate samples)										
		Total extracted ¹	F	FSO	FSO ₂	FSOP	FSO ₂ P	FSO ₂ A	TTR ²	Un-extracted	¹⁴ CO ₂	Recovery
USA/Florida (X)												
22	15	74.5	30.7	34.4	8.1	1.3	--	--	73.2	10.0	0.6	85.1
	50	60.2	7.6	30.7	15.5	2.0	4.4	--	53.8	22.7	2.7	85.6
	90	52.7	6.7	20.2	16.3	1.8	6.9	0.5	43.2	24.8	4.8	82.3
28	15	69.6	16.5	38.1	11.4	1.7	1.9	--	66.0	14.3	1.2	85.1
	50	51.3	5.3	21.0	16.2	1.8	6.2	0.5	42.5	24.3	5.2	80.8
	90	43.7	2.9	16.4	13.4	2.0	7.6	1.0	32.7	36.3	8.6	88.6
Costa Rica (XI)												
22	15	73.0	15.5	45.4	5.9	3.7	2.5	--	66.8	21.6	2.0	96.6
	50	55.8	5.9	28.9	7.0	5.2	8.2	0.6	41.8	37.1	7.5	100.4
	90	47.5	3.5	21.3	5.5	5.4	10.5	1.3	30.3	39.6	12.0	99.1
28	15	66.9	14.0	37.1	5.2	6.5	4.1	--	56.3	28.9	4.4	100.2
	50	43.2	4.6	20.9	3.6	6.3	6.8	1.0	29.1	47.3	12.4	102.9
	90	36.8	2.6	17.6	3.9	5.0	6.2	1.5	24.1	39.3	16.1	92.2
Brazil/P. Fundo (XII)												
22	15	81.1	11.2	53.5	6.7	5.4	4.3	--	71.4	18.1	2.7	101.0

°C	Days	Radioactivity, % of applied (mean of triplicate samples)										
		Total extracted ¹	F	FSO	FSO ₂	FSOP	FSO ₂ P	FSO ₂ A	TTR ²	Un-extracted	¹⁴ CO ₂	Recovery
	50	58.8	2.0	26.2	9.7	5.5	13.5	1.9	37.9	33.4	13.3	105.5
	90	44.3	1.2	13.0	3.5	4.2	18.9	3.5	17.7	32.1	20.8	97.2
28	15	74.5	4.0	49.6	9.2	5.9	5.8	--	62.8	21.4	5.0	100.9
	50	42.6	1.7	15.2	3.7	3.9	14.6	3.5	20.6	33.3	22.4	98.3
	90	23.3	0.7	5.8	1.0	2.4	8.4	5.0	7.5	46.4	36.5	106.2
Brazil/Parana (XIII)												
22	15	71.6	16.9	40.6	5.2	4.5	4.4	--	62.7	28.2	1.1	99.9
	50	49.2	5.1	17.6	5.2	3.8	16.6	0.9	27.9	39.4	9.4	98.0
	90	32.4	2.2	7.8	2.0	2.7	15.6	2.1	12.0	48.5	18.1	99.0
28	15	63.7	11.0	32.3	6.9	5.1	8.4	--	50.2	35.7	3.8	103.2
	50	33.7	2.7	10.6	2.1	2.9	13.3	2.1	15.4	50.5	19.0	103.2
	90	18.2	1.1	4.3	0.9	1.3	8.0	2.6	6.3	52.3	33.7	104.2
Thailand (XIV)												
22	15	86.9	24.6	60.9	0.3	1.1	--	--	85.8	13.8	0.4	101.1
	50	74.3	5.0	52.5	7.7	5.2	3.9	--	65.2	20.6	3.0	97.9
	90	61.2	2.0	39.6	8.5	6.2	4.0	--	51.0	24.6	4.3	90.1
28	15	94.2	25.0	64.2	2.5	2.5	--	--	91.7	16.1	0.6	110.9
	50	70.3	3.7	45.6	11.5	4.9	4.6	--	60.8	27.1	4.4	101.8
	90	50.1	1.9	28.0	9.7	4.2	6.3	--	39.6	30.7	9.9	90.7
Philippines (XV)												
22	15	75.4	6.4	61.7	4.4	1.6	1.3	--	72.5	11.5	1.8	88.6
	50	56.8	1.2	31.7	10.6	1.7	9.6	1.4	43.5	27.0	11.2	95.0
	90	37.7	0.3	18.0	6.7	0.9	9.3	2.1	25.0	35.7	24.2	97.6
28	15	73.4	3.5	57.2	7.5	2.2	3.0	--	68.2	18.3	4.6	96.3
	50	38.8	0.4	22.5	6.4	0.9	5.6	2.5	29.3	30.8	23.3	92.9
	90	19.9	0.2	11.1	2.8	0.6	1.8	3.0	14.1	39.7	40.0	99.6
Japan/Tsurug (XVI)												
22	15	73.4	14.8	56.6	1.1	0.9	--	--	72.5	20.0	0.3	93.7
	50	66.2	4.8	45.1	9.3	3.1	3.9	--	59.2	25.4	1.2	92.8
	90	58.7	1.9	33.7	11.3	3.2	8.0	0.6	46.9	33.2	3.5	95.4
28	15	64.0	5.6	51.0	4.0	2.3	1.1	--	60.6	31.4	0.5	95.9
	50	51.3	2.3	29.3	9.5	2.7	6.7	0.8	41.1	32.8	4.4	88.5
	90	43.7	1.2	22.0	6.6	3.3	8.5	2.1	29.8	44.8	9.9	98.4

¹ Including polar products (1%)

² Total toxic residues (F + FSO + FSO₂)

-- not detected

F fenamiphos
 FSO fenamiphos sulfoxide
 FSO₂ fenamiphos sulfone
 FSOP fenamiphos sulfoxide phenol
 FSO₂P fenamiphos sulfone phenol
 FSO₂A fenamiphos sulfone anisole

From the results, the half-life of fenamiphos was less than 15 days at 22°C in all the soils. If the residues of fenamiphos, fenamiphos sulfoxide and fenamiphos sulfone are combined, as all three compounds show nematocidal activity, residues are still detectable after 90 days at 22°C, at levels ranging from 2.3% of the applied radioactivity (France) to 66.8% (Japan/Toyoda). After 50 days in many soils, the proportion of fenamiphos sulfone phenol increased as corresponding levels of fenamiphos sulfoxide and sulfone decreased. The results are in agreement with those of previous studies, as the major degradation products are fenamiphos sulfoxide and fenamiphos sulfone which are then converted to the corresponding phenols by cleavage of the phosphate ester bond. The degradation pathways of fenamiphos in soils are shown in Figure 3.

The half-lives of fenamiphos and/or the sum of fenamiphos and fenamiphos sulfoxide in the studies by Simon (1990), Simon *et al.* (1992), Spiteller (1989a,b) and Lane and Clay (1989) are shown in Table 49.

Fenamiphos was rapidly degraded under aerobic conditions with half-lives of ≤ 30 days in two experiments. Half-lives of the sum of fenamiphos and fenamiphos sulfoxide (both compounds nematocidal) ranged from 12 to 166 days, depending upon soil type and incubation temperature.

Figure 3. Proposed degradation pathways of fenamiphos in soil under aerobic and anaerobic conditions.

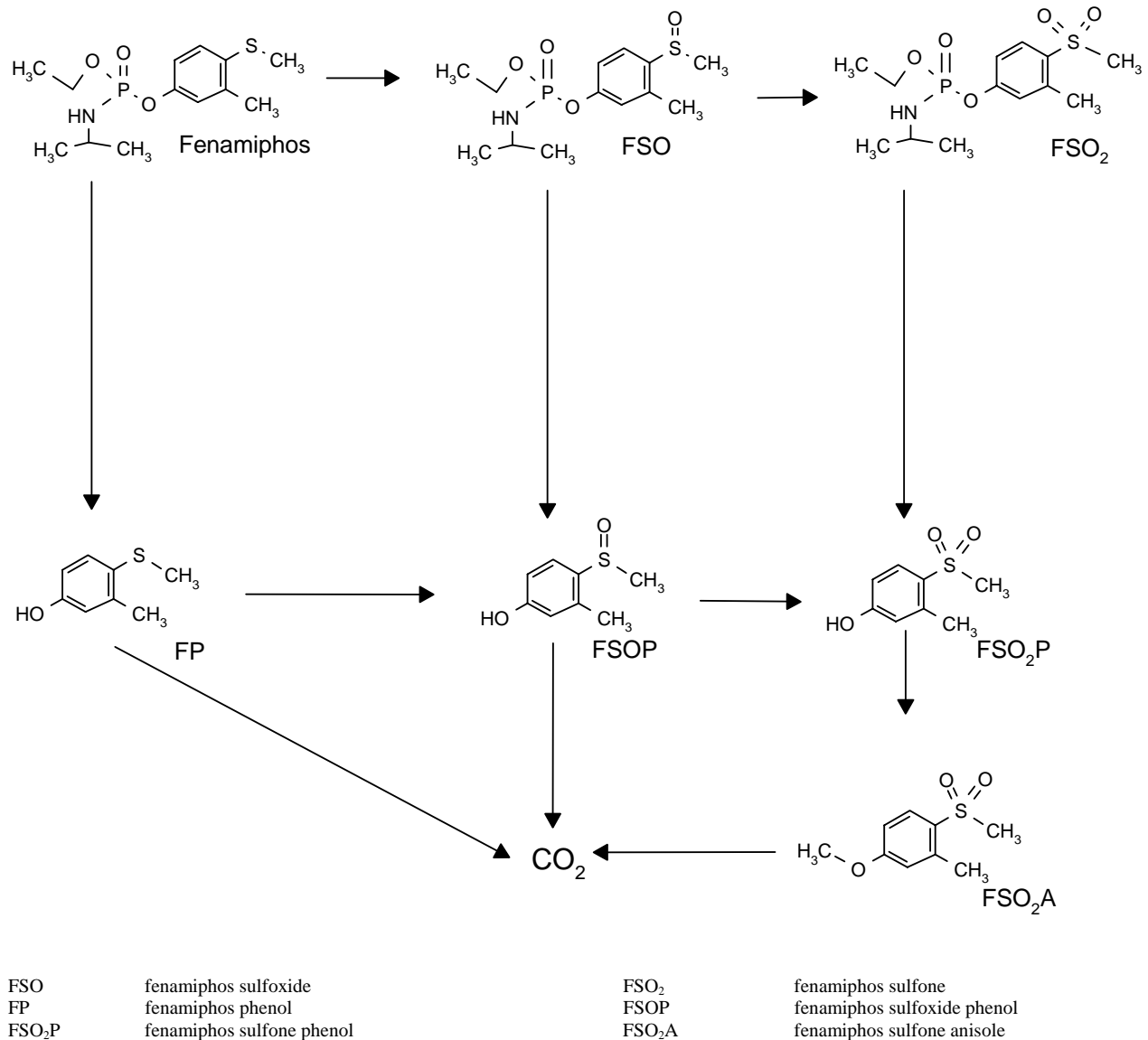


Table 49. Half-lives of the sum of fenamiphos and fenamiphos sulfoxide calculated according to first-order kinetics from experiments under aerobic laboratory conditions

Soil and conditions	Application rate, ai	Half-life, days, F + FSO	Reference
Aerobic			
sand (USA)	18 mg/kg	153 *	Lane and Clay, 1989
sandy loam (USA)	10 kg/ha	--	Spiteller, 1989b

Soil and conditions	Application rate, ai	Half-life, days, F + FSO	Reference
silty sand loam (Canada)	10 kg/ha	49/27 **	Simon, 1990
loamy sand (Sweden)	10 kg/ha	85/37 **	
loamy silt (Germany)	10 kg/ha	30/23 **	
silty sand (Germany)	10 kg/ha	50/36 **	
clay silt (Netherlands)	10 kg/ha	40/27 **	
clay loam (France)	10 kg/ha	29/12 **	
loamy sand (USA)	10 kg/ha	81/28 **	
silty loam sand (USA)	10 kg/ha	68/36 **	
silty loam sand (Japan)	10 kg/ha	166/86 **	
sand (USA)	10 kg/ha	35/25 ***	
clay loam (Costa Rica)	10 kg/ha	32/24 ***	
sandy clay loam (Brazil)	10 kg/ha	26/18 ***	
silty clay (Brazil)	10 kg/ha	21/14 ***	
silty clay (Thailand)	10 kg/ha	70/52 ***	
silty sand loam (Philippines)	10 kg/ha	33/23 ***	
loamy sand (Japan)	10 kg/ha	48/28 ***	

¹ Not calculated owing to rapid degradation

* F+FSO+FSO₂

** 1st value at 16°C, 2nd value at 22°C

*** 1st value at 22°C, 2nd value at 28°C

F fenamiphos

FSO fenamiphos sulfoxide

FSO₂ fenamiphos sulfone

In a US study (Kasper and Shadrack, 1993), the half-life of fenamiphos was determined in soils at two sites in California. At Chualar and Fresno, soils placed in vessels located in outdoor plots were treated with [U-*phenyl*-¹⁴C]fenamiphos at a rate equivalent to 10.9 kg ai/ha and sampled 6 and 12 hours and 1, 2, 4, 7, 10, 18, 30 and 60 days after treatment. At each sampling the top 3 cm layer of soil was homogenized, extracted and analysed by TLC. Extraction procedures were similar to those used in other studies.

The properties of the soils from the two sites are shown below.

	Fresno soil	Chualar soil
% sand	64.7	70.0
% silt	31.3	18.0
% clay	4.0	12.0
% organic C	0.5	1.3
pH	7.5	6.4
CEC (meq/100 g)	6.9	17.3
Density (g/cm ³)	1.41	1.32

The calculated half-lives were 19.9 and 18.2 days in the Fresno and Chualar soils respectively. These values compare well with the 15.7 days reported by Spiteller. Fenamiphos sulfone phenol was the product of interest in this study and it was not detected until day 7. Its levels ranged from 0.4-1.9% and 0.3-3.5% of the applied radioactivity from days 7 to 60 in the Fresno and Chualar soils respectively. The main components of the radioactive residue were fenamiphos, fenamiphos sulfoxide and fenamiphos sulfoxide phenol.

In an earlier study also at sites in Fresno and Chualar (Grace *et al.*, 1990), fenamiphos was applied twice to soil at a rate equivalent to 11.2 kg ai/ha; the treatment interval was 6 months. Half-lives of 16.2 days at Chualar and 17 days at Fresno were reported. These values are in good agreement with previously reported half-lives of fenamiphos ranging from 15 to 30 days. Total residues of the compounds showing nematicidal activity (fenamiphos, fenamiphos sulfoxide and fenamiphos sulfone) were below the limit of determination of 0.01 mg/kg at depths below 61 cm at all

sampling times after a single application. The data from both sites showed that under conditions of field use fenamiphos residues would not readily reach depths greater than 90 cm before dissipation.

In a field dissipation study in North Carolina (Halarnkar *et al.*, 1996), two sprays of fenamiphos were applied to soil at a rate equivalent to 12.3 kg ai/ha, at an interval of 3 months. Soil core samples were taken immediately before and 2 hours after the first application, then 1, 3, 5, 10, 12, 30, 60 and 90 days after the first spray, then 3 hours and 1, 3, 5, 10, 14, 28, 61, 88, 180, 276, 361, 452 and 561 days after the second application. Soil cores were also collected within 21 days after significant rainfall (>7.5 cm in 24 hours). Cores were a minimum depth of 15.2 cm, but not more than 122 cm. Untreated controls were collected within 2 hours after the first spray and 88, 276 and 561 days after the second spray. The treated plots were irrigated and the total rainfall plus irrigation was 145% of the 10-year average rainfall for the study period.

At each sampling the cores were sub-sampled to depths of 0-15.2, 15.2-30.5, 30.5-61, 61-76.2, 76.2-91.4 and 91.4-122 cm. All soil cores down to depths of 61 cm were extracted and analysed by GLC and reversed phase, ion-pair and normal HPLC. The limits of detection by HPLC were 0.01 µg/g for fenamiphos, FSO, FSO₂, FSOP, FSO₂P and FSO₃HP (fenamiphos sulfonic acid phenol). As the soil samples were held in frozen storage for a maximum of 842 days before analysis frozen storage stability studies were also conducted.

The properties of the soil used in the study at depths from which cores were routinely analysed are shown below.

	Core depth (cm)		
	0-15.2 cm	15.2-30.5	30.5-61
% organic C	0.49	0.38	0.16
pH	6.6	5.5	5.4
CEC	1.02	1.30	0.84
density (g/cm ³)	1.66	1.66	1.68
% moisture (0.33 bar)	3.02	3.01	3.47
% sand	89.2	89.2	85.2
% silt	6.4	6.4	8.4
% clay	4.4	4.4	6.4
Class	sand	sand	loamy sand

The half-life of fenamiphos was determined to be 15.9 days with a first-order rate constant of -0.0437. The half-life is similar to those found in other laboratory degradation studies (15-30 days). The results are shown in Table 50.

Table 50. Distribution of residues after two spray applications of fenamiphos (Halarnkar *et al.*, 1996).

Compound	Maximum residue, mg/kg, at (DAT1)	Dissipation, minimum mg/kg, at (DAT1)	Maximum residue, mg/kg, at (DAT2)	Dissipation, minimum mg/kg, at (DAT2)
F	2.70 (0)	<0.01 (60) all depths	2.52 (0)	<0.01 (180) all depths
FSO	4.10 (3)	0.13 (90)	1.95 (3)	<0.01 (361) all depths
FSO ₂	0.44 (12)	<0.01 (90) all depths	0.10 (5)	<0.01 (61) all depths
FSOP	0.13 (30)	0.02 (90)	0.41 (14)	<0.01 (61) all depths
FSO ₂ P	0.23 (30)	0.03 (90)	0.13 (14)	<0.01 (28) all depths
Total F equivalents	4.68 (3)	0.28 (90)	4.03 (3)	<0.01 (361)

DAT1: days after first spray

DAT2: days after second spray

Recoveries of fenamiphos, fenamiphos sulfoxide, fenamiphos sulfone, fenamiphos sulfoxide phenol, fenamiphos sulfone phenol and fenamiphos sulfonic acid phenol were determined at a level of 1 mg/kg of each compound in fortified field samples. The recoveries were 82-108%, 68-128%, 75-

117%, 69-91%, 78-103% and 72-95% respectively. In a storage stability study samples spiked with fenamiphos, fenamiphos sulfoxide and fenamiphos sulfone were stored for 365 days, when 86, 100 and 100% of the three compounds remained respectively. In another stability study samples of sandy loam soil fortified with fenamiphos sulfoxide phenol, fenamiphos sulfone phenol and fenamiphos sulfonic acid phenol were stored for 731 days. The recoveries were 89, 84 and 95% respectively.

In a similar dissipation study in California (Antle *et al.*, 1996), a sandy loam soil was treated with two sprays of fenamiphos at a rate of 12.3 kg ai/ha, with an interval of 93 days between applications. Soil core samples were taken immediately before and 1, 3, 5, 10, 14, 28, 60 and 93 days after the first application, then 1, 3, 5, 9, 14, 28, 61, 90, 182, 274, 365, 454 and 548 days after the second application. The core depths were as in the previous study.

The soil characteristics are shown below.

	Core depth		
	0-15.2 cm	15.2-30.5	30.5-61
% organic C	0.22	0.33	0.27
pH	8.1	7.5	7.0
CEC	5.18	4.79	4.97
density (g/cm ³)	1.63	1.57	1.59
% moisture (0.33 bar)	10.77	10.94	9.62
% sand	63.2	61.2	63.2
% silt	28.0	30.0	28.0
% clay	8.8	8.8	8.8
Class	sandy loam	sandy loam	sandy loam

Table 51. Distribution of residues after two spray applications of fenamiphos (Antle *et al.*, 1996).

Compound	Maximum residue, mg/kg, at (DAT1)	Dissipation, minimum mg/kg, at (DAT1)	Maximum residue, mg/kg, at (DAT2)	Dissipation, minimum mg/kg, at (DAT2)
F	1.47 (3)	<0.01 (93) all depths	2.03 (1)	<0.01 (28) all depths
FSO	3.28 (10)	0.08 (93) all depths	2.52 (3)	<0.01 (454) all depths
FSO ₂	0.44 (28)	<0.01 (93) all depths	0.09 (3)	<0.01 (9) all depths
FSOP	0.34 (28)	0.02 (93)	0.38 (5)	<0.01 (9) all depths
FSO ₂ P	0.29 (28)	0.05 (93)	0.07 (1, 5)	<0.01 (9) all depths
FSO ₃ HP			0.14 (182)	<0.01 (548)
Total F equivalents	4.44 (10)	0.26 (93)	4.31 (3)	<0.01 (548)

Recoveries at a fortification level of 0.1 mg/kg were 63-95% for fenamiphos, 79-129% for fenamiphos sulfoxide, 82-108% for fenamiphos sulfone, 81-117% for fenamiphos sulfoxide phenol, 89-101% for fenamiphos sulfone phenol and 86-113% for fenamiphos sulfonic acid phenol.

A half-life of 15 days was calculated from samples taken between the first and second applications. This is in agreement with the results of other field degradation studies (Table 52).

Table 52. Half-lives of fenamiphos in various field studies.

Location	Application rate, kg ai/ha	Half-life, days	Reference
Chualar, California	11.2	16.2	Grace <i>et al.</i> , 1990
Fresno, California	11.2	17.0	
Fresno, California	12.3	15.0	Antle <i>et al.</i> , 1996
North Carolina	12.3	15.9	Halarnkar <i>et al.</i> , 1996

Summary of results of soil studies

In laboratory experiments fenamiphos was degraded rapidly under aerobic conditions with half-lives less than 30 days, and more rapidly in sunlight on soil surfaces under laboratory and natural conditions with half-lives of 1.6 and 2.7 hours respectively. Adsorption studies with a variety of soils from cold and tropical regions showed that the adsorption of fenamiphos was dependent upon the clay and silt content. In several degradation studies, fenamiphos was shown to be converted to fenamiphos sulfoxide and sulfone with subsequent transformation to the phenol derivatives.

Hydrolysis

The hydrolysis of [1-*phenyl*-¹⁴C]fenamiphos was examined in aqueous buffer solutions (pH 3, 7 and 9) for 30 days at concentrations of 1 and 10 mg/l and temperatures of 30° and 50°C (McNamara and Wilson, 1979). Samples were taken at regular intervals up to 30 days. Each solution was radioassayed and extracted with CH₂Cl₂. The organic fractions from each sample were radioassayed, concentrated, radioassayed again and analysed by TLC. Aqueous fractions from the first extraction which showed much radioactivity were treated with NaCl and HCl before re-extraction with CH₃Cl.

At 30°C more than 95% of the applied radioactivity was extracted into CH₃Cl at pH 7 and 9 at both concentrations, but at pH 3 the extractable radioactivity decreased from 99% at day 0 to 10% at day 30. After incubation at 50°C for 30 days the extractable radioactivity was 4% at pH 3, 97% at pH 7 and 57% at pH 9 at 1 mg/l, and 6% at pH 3, 97% at pH 7 and 67% at pH 9 at 10 mg/l.

Table 53. Rate constants and half-lives for hydrolysis of fenamiphos in buffer solutions.

pH	°C	Concentration, mg/l	k ₁ , days ⁻¹	Half-life, days
3	30	1	0.079	8.8
		10	0.066	10
3	50	1	0.211	3.3
		10	0.232	3.0
9	30	1	0.003	220
		10	0.003	230
9	50	1	0.032	22
		10	0.030	23

The main hydrolysis products at pH 9 at a concentration of 10 mg/l were fenamiphos phenol, fenamiphos sulfoxide phenol and fenamiphos sulfoxide, formed by phosphate ester hydrolysis in basic solution and oxidation at the methylthio group. In pH 3 buffer the major hydrolysis product (50% of the initial radioactivity) was deaminated fenamiphos, with fenamiphos phenol and deaminated fenamiphos sulfoxide accounting for less than 10%.

The hydrolytic degradation of fenamiphos was investigated in buffer solutions at pH 4, 7 and 9 and temperatures of 60, 70 and 80°C by Andersen (1985b) at a concentration of 0.05 molar fenamiphos. The solutions were sealed and incubated in darkness. The concentration of fenamiphos was determined by HPLC.

Table 54. Half-lives and hydrolysis rate constants for degradation of fenamiphos in buffer solutions (Andersen, 1985b).

pH	°C	Hydrolysis constant, k	Half-life
4	60	0.071 day ⁻¹	9.8 days
	70	0.217 day ⁻¹	3.2 days
	80	0.408 day ⁻¹	1.7 days
7	60	0.010 day ⁻¹	67.1 days
	70	0.019 day ⁻¹	37.2 days
	80	0.050 day ⁻¹	13.8 days

pH	°C	Hydrolysis constant, k	Half-life
9	60	0.010 h ⁻¹	70 h
	70	0.034 h ⁻¹	20.5 h
	80	0.133 h ⁻¹	5.2 h

The results show that the hydrolysis of fenamiphos is accelerated by elevated temperatures and is most rapid at pH 9.

The stability of [1-*phenyl*-^{13,14}C]fenamiphos in buffer solutions at pH 5, 7 and 9 in the dark and under sterile conditions was investigated by Mulford (1987b). The labelled fenamiphos was dissolved in sterile acetate, phosphate or borate buffer and assayed for soluble radioactivity. The solutions were sealed in vials, kept in the dark, and analysed 0, 5, 10, 14, 18, 24 and 31 days after preparation. At each sampling the total radioactivity was assayed by liquid scintillation counting and fenamiphos and the hydrolysis products were quantified by HPLC. Each sample was extracted with dichloromethane and analysed by TLC. The recovered radioactivity ranged from 93 to 99%, 94.4 to 96.5% and 98.3 to 100% at pH 5, 7 and 9 respectively. The distribution of the radioactivity is shown in Table 55.

Table 55. Distribution of radioactive compounds in buffered solutions in the dark (Mulford, 1987b).

PH, compound	% of recovered radioactivity at days after application						
	0	5	10	14	18	24	31
pH 5							
F	99.3	97.3	95.4	97.4	97.2	93.4	89.6
FSO	0.1	1.8	4.5	1.9	2.3	6.3	9.9
pH 7							
F	99.6	99.0	96.8	97.6	97.4	95.4	91.9
FSO	0.2	0.6	2.2	1.8	1.7	4.6	8.1
pH 9							
F	99.5	98.4	96.8	96.4	96.1	93.7	90.1
FSO	0.3	0.5	1.7	1.6	1.6	2.5	4.1
FP	0	0.8	1.3	1.7	2.1	3.4	5.2

Fenamiphos sulfoxide was the major hydrolysis product, but fenamiphos phenol was also formed at pH 9. At all sampling times the products accounted for less than 10% of the recovered radioactivity, indicating that fenamiphos is stable in sterile solutions in the dark.

The calculated half-life values are shown in Table 56.

Table 56. Half-lives calculated for fenamiphos in sterile buffer solutions in the dark (Mulford, 1987).

pH	°C	Concentration, mg/l	Rate constant, days ⁻¹	Half-life, days
5	25	34.5	2.81 x 10 ⁻³	245
7	25	34.0	2.30 x 10 ⁻³	301
9	25	35.4	2.95 x 10 ⁻³	235

The half-lives found in the studies described are compared in Table 57 .

Table 57. Half-lives of fenamiphos found in hydrolysis studies.

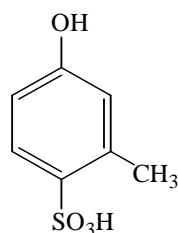
pH	°C	Fenamiphos concentration, mg/l, and label	Half-life, days	Reference
3	30	1 [1- <i>phenyl</i> - ¹⁴ C]	8.8	McNamara and Wilson, 1979, revised 1981
		10	10	
3	50	1 [1- <i>phenyl</i> - ¹⁴ C]	3.3	
		10	3.0	
9	30	1 [1- <i>phenyl</i> - ¹⁴ C]	220	

pH	°C	Fenamiphos concentration, mg/l, and label	Half-life, days	Reference
		10	230	
9	50	1 [1- <i>phenyl</i> - ¹⁴ C]	22	
		10	23	
4	60	7.5 unlabelled	9.8	Anderson, 1985
	70		3.2	
	80		1.7	
7	60	7.5 unlabelled	67.1	
	70		37.2	
	80		13.8	
9	60	7.5 unlabelled	2.9	
	70		0.9	
	80		0.2	
5	25	34.5 [1- <i>phenyl</i> - ¹⁴ C]	245	Mulford, 1987
7	25	34.0 [1- <i>phenyl</i> - ¹⁴ C]	301	
9	25	35.0 [1- <i>phenyl</i> - ¹⁴ C]	235	

Photolysis

Aliquots of a 12 mg/l solution of [1-*phenyl*-¹⁴C]fenamiphos in phosphate buffer were irradiated with a mercury lamp at 27-28°C for 0, 1, 2, 4, 6, 12 or 24 hours (Dime *et al.*, 1983). Controls were kept in the dark. The aqueous solutions were extracted with CH₃Cl and both organic and aqueous phases were radioassayed. TLC was used to identify the radioactive components.

The half-life was 3.6 hours with a first-order rate constant of 0.19 h⁻¹; fenamiphos was shown to be stable under similar conditions in the absence of light. The light intensity was compared to that of a July day in Kansas, and it was reported that the half-life under Kansas light intensity would be 6.8 hours. Recoveries of radioactivity were 94% from the irradiated samples and 99% from the controls. TLC of the organosoluble fractions showed that 34 to >99% of the radioactivity was present in the organic phase and was largely due to fenamiphos, ranging from 94 to 4.3% from 0 to 24 hours of irradiation, and fenamiphos sulfoxide ranging from 4 to 17.3 % over the same period. The water-soluble fractions contained 0.3 to 56.4% of the radioactivity with fenamiphos phenol sulfonic acid (4-hydroxy-2-methylbenzenesulfonic acid), the major component, accounting for 2.3 to 18.6% of the recovered radioactivity. The results are shown in Table 58.



fenamiphos phenol sulfonic acid
(4-hydroxy-2-methylbenzenesulfonic acid)

Table 58. Distribution of ¹⁴C in extracts of irradiated aqueous solutions of [1-*phenyl*-¹⁴C]fenamiphos (Dime *et al.*, 1983, revised 1985).

Component	¹⁴ C, % of applied, after (hours)						
	0	1	2	4	6	12	24
Organosoluble							
F	94.3	78.7	64.1	43.7	28.1	9.6	4.3
FSO	4.0	2.7	7.0	8.4	11.2	16.0	17.3
Rf > Fenamiphos	--	2.2	2.7	2.0	2.4	1.1	1.3
Rf < Fenamiphos	--	1.9	2.8	3.7	5.5	2.9	2.8
Diffuse	1.4	3.6	6.0	10.9	11.4	10.9	5.3
Origin	<0.1	0.4	1.2	2.2	3.1	4.1	3.3

Component	¹⁴ C, % of applied, after (hours)						
	0	1	2	4	6	12	24
Subtotal	99.7	89.5	83.8	70.9	61.7	44.6	34.3
Water-soluble							
Band 7 (Rf = 0.95)		0.1	0.1	0.2	0.4	0.4	0.1
Band 6 (Rf = 0.91)		0.1	0.2	0.2	0.3	0.5	0.4
Band 5 – FSOP		0.3	0.6	1.1	1.3	1.1	1.1
Band 4 (Rf = 0.54)		0.2	0.5	0.7	1.0	1.0	1.1
Band 3 – FSA		0.7	1.4	2.1	3.1	4.4	6.1
Band 2 – FPSA		2.3	5.6	10.5	14.3	17.1	18.6
Band 1 (Rf = 0.10)		0.4	0.7	1.5	1.9	2.3	3.3
Origin ²		1.4	3.2	6.0	9.1	14.4	20.8
Diffuse ¹		0.6	1.2	2.6	3.4	4.8	4.9
Subtotal	0.3	6.1	13.5	24.9	34.8	46.0	56.4
Total recovered ¹⁴ C	100	95.6	97.3	95.8	96.5	90.6	90.7

FSA

fenamiphos sulfonic acid

FPSA

fenamiphos phenol sulfonic acid

The rapid degradation of fenamiphos was confirmed in another irradiation study (Andersen, 1985a). Solutions of fenamiphos were irradiated with a TQ 150 high-pressure mercury vapour lamp fitted with a Duran 50 filter tube to pass wavelengths >290 nm in a rotary carousel irradiation apparatus. Analysis by HPLC indicated a half-life of 15 minutes. Photoproducts were not identified. The UV spectrum of fenamiphos (3.8 mg/l in distilled water) over the range 200-249 nm showed a maximum at 284 nm. The conclusion of the study was that the UV absorption properties of the compound indicate that fenamiphos would undergo photodegradation under environmental conditions.

Environmental fate in water/sediment systems

The degradation of [1-phenyl-^{13,14}C]fenamiphos was investigated in a laboratory study (Wilmes, 1987) in two water/sediment systems obtained from a reclaimed gravel pit (Lienden) and an orchard drainage ditch (Ijzendoorn) in The Netherlands. Both systems were treated with 10 mg of labelled fenamiphos/l applied to the sludge, and samples were analysed 1, 7, 26, 54 and 98 days after application. Water and sludge were separated and the water samples analysed by reversed-phase HPLC with a radioactivity detector. The sediment samples were extracted with MeOH and with MeOH/H₂O (1:1) containing 0.2% H₃PO₄. Measurement of ¹⁴C in the extracts by liquid scintillation counting was followed by HPLC. The remaining solids were compressed and analysed by combustion and LSC. Volatiles were collected in traps.

In both systems, the distribution of the radioactivity between the water and sediment remained constant for the duration of the study. The results are shown in Table 59. In the Lienden samples, the radioactivity in the water ranged from 76 to 86% and in the Ijzendoorn samples from 51 to 62% of the applied radioactivity. The rate of degradation was faster in the Lienden samples, with almost complete transformation from fenamiphos to fenamiphos sulfoxide and fenamiphos sulfoxide phenol by day 98. The major identified product was fenamiphos sulfoxide.

Table 59. Distribution of ¹⁴C from fenamiphos in water/sediment systems (Wilmes, 1987).

Phase, site, DAT	Distribution of radioactive residues as % of recovered radioactivity (mean of duplicates)					
	F	FSO	FSOP	FSO ₂ P	Unidentified	Total % found
Water						
Lienden						
1	81.1	2.5				83.6
7	69.1	12.2				81.3
26	66.1	9.8				75.9
54	7.2	69.3	5.1	1.7 ¹	2.6	85.9

Phase, site, DAT	Distribution of radioactive residues as % of recovered radioactivity (mean of duplicates)					
	F	FSO	FSOP	FSO ₂ P	Unidentified	Total % found
98	–	66.7	10.9	4.6	3.4 ¹	85.6
Ijzendoorn						
1	56.8	2.1				58.9
7	54.7	7.5				62.2
26	51.9	2.7				54.6
54	35.1	15.6	2.5		2.6	55.8
98	31.1	15.7	1.0	1.8	1.6	51.2
Sediment						
Lienden						
1	14.0	0.1 ¹			1.5	15.6
7	15.3	0.5 ¹			2.2	18.0
26	19.3	0.4			1.6	21.3
54	3.3	3.1	0.4 (total FSOP + FSO ₂ P)		2.4	9.2
98	0.8	3.2	0.3 (total FSOP + FSO ₂ P)		6.3	10.6
Ijzendoorn						
1	35.1	0.4			3.8	39.3
7	31.7	0.9	0.7 (total FSOP + FSO ₂ P)		2.5	35.8
26	37.3	2.8			2.8	42.9
54	30.6	2.3			2.3	33.2
98	29.3	2.4			2.4	34.1

¹Single analyses

In an addendum to the above study the degradation of [1-*phenyl*-^{13/14}C]fenamiphos was investigated at two concentrations in the Lienden system after a 29-day period (Wilmes, 1988). The results after 29 and 26 days are compared in Table 60.

Table 60. Comparison of degradation of fenamiphos in water/sediment system from Lienden after 26 and 29 days (Wilmes, 1987, 1988).

Sample	¹⁴ C, % of applied		
	29 days, 10 mg/l	29 days, 1 mg/l	26 days*, 10 mg/l
Water phase			
F	47.4	21.2	66.1
FSO	43.8	58.6	9.8
Sediment extract			
F	8.6	7.5	19.3
FSO	2.3	4.6	0.4
Total F	55.9	28.7	85.4
Total FSO	46.1	63.2	10.2

* Original experiment

At day 29, 44% of the radioactivity in the water was due to fenamiphos sulfoxide compared to 10% at day 26 in the original study. There was also increased degradation of fenamiphos at the lower concentration.

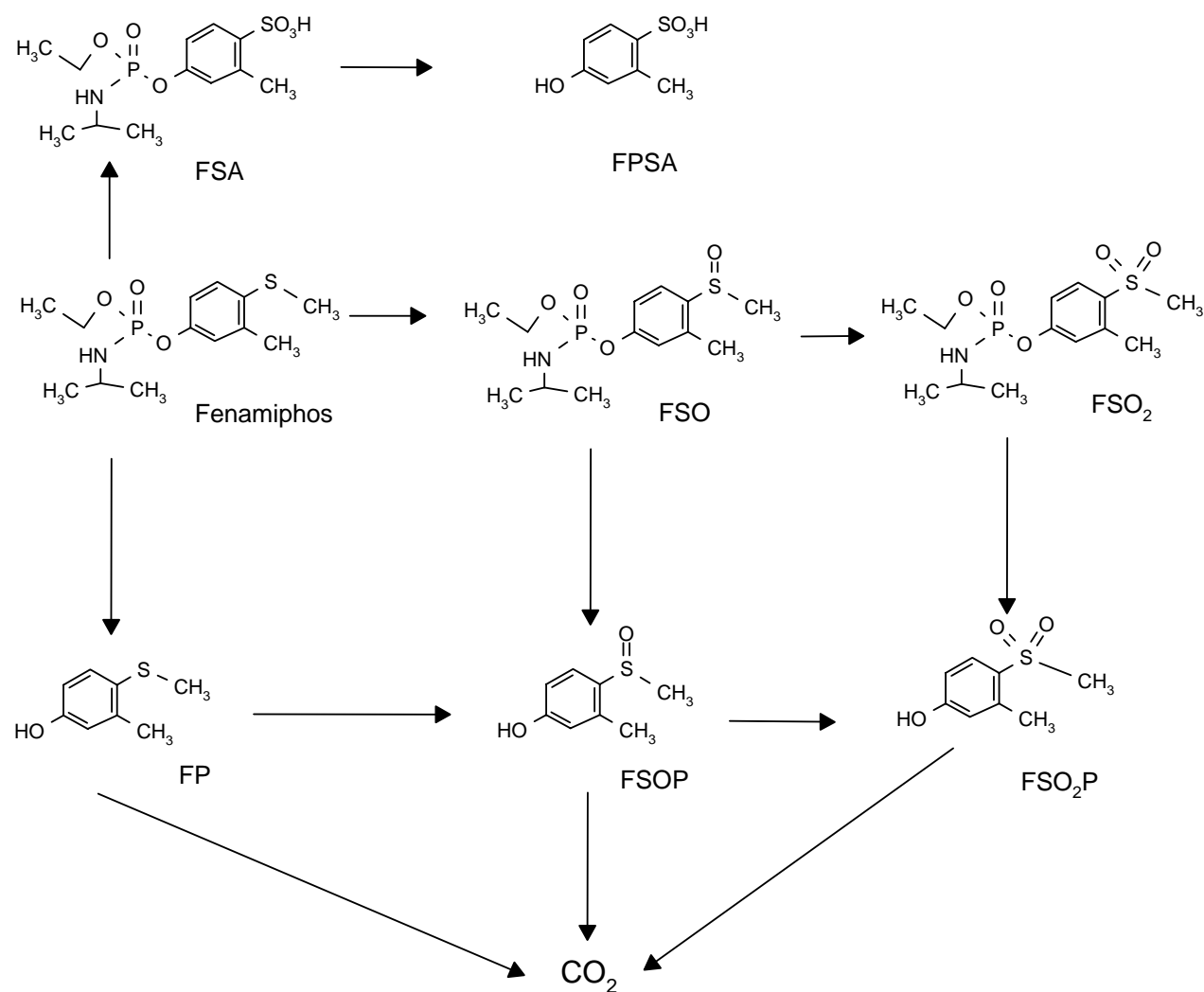
Proposed degradation pathways of fenamiphos in aquatic systems are shown in Figure 4. In summary, fenamiphos is degraded by the familiar mechanisms of oxidation of the methylthio group, cleavage of the phosphate ester bond and further degradation to CO₂. An additional pathway involves the formation of fenamiphos phenol sulfonic acid by oxidative demethylation of the methylthio group before cleavage of the ester.

METHODS OF RESIDUE ANALYSIS

Methods for the determination of fenamiphos and its sulfoxide and sulfone in various crops, animal tissues, water and soil were reported by the manufacturer. In addition, multi-residue enforcement methods were submitted by the governments of The Netherlands and Australia.

Enforcement methods

In the Official Methods of Analysis in The Netherlands, fenamiphos residues are determined by GLC with an ion-trap detector. The limit of determination for fenamiphos is reported as 0.05 mg/kg for various types of sample, and recoveries from lettuce were 55-70%, with fortifications at 0.07-0.35 mg/kg. In fatty foods the limit of determination was also reported as 0.05 mg/kg; recoveries were not Figure 4. Proposed degradation pathways of fenamiphos in the aquatic environment.



FSO fenamiphos sulfoxide
 FP fenamiphos phenol
 FSO₂P fenamiphos sulfone phenol
 FPSA fenamiphos phenol sulfonic acid

FSO₂ fenamiphos sulfone
 FSOP fenamiphos sulfoxide phenol
 FSA fenamiphos sulfonic acid

reported. The LOD in the multi-residue method appears to be for fenamiphos alone and not total fenamiphos residues.

In a multi-residue method provided by the Australian government fenamiphos is determined by GLC with flame photometric detection in the phosphorus mode. The limit of determination is reported as 0.02 mg/kg.

The published method devised by Thornton (1971) was included as an enforcement method by the manufacturer. It is described in detail below.

Published methods for enforcement also include the general methods for the determination of organophosphorus pesticides of Hild and Thier (1979) and Specht and Thier (1987).

Other general methods

A general method for the determination of organophosphorus pesticide residues in non-fatty foods was reported by Storherr *et al.* (1971). This method was used in Brazil for the determination of fenamiphos in tomatoes, bananas, cotton and coffee.

Samples containing less than 5% sugar are blended with CH₃CN and Celite, then filtered by vacuum. Samples containing about 5-15% sugar (e.g. fruits) are blended with CH₃CN and water and filtered. An aliquot of the filtrate is extracted three times with CH₃Cl. The CH₃Cl extracts are combined and drained through a Celite-charcoal column. The column is eluted with CH₃CN/C₆H₆ (1:1) and the eluate concentrated to about 1.0 ml by evaporation. Isopropanol is added and the mixture distilled to remove the CH₃CN. This procedure is repeated once. Finally, the residue is concentrated to about 0.5 ml and made up to 1.0 ml with ethyl acetate.

About 3-8 µl are injected onto a 300 x 2.2 mm i.d. glass column, 2% stabilized diethylene glycol succinate on 80-100 mesh Chromosorb W (IIP) (column temperature, 200-220°C). Organophosphorus pesticides are detected by their retention times with a thermionic alkali detector (TID). At fortification levels of 0.05-0.2 mg/kg, recoveries from various crops were in the range 72-122%.

Forty-one organophosphorus pesticides were evaluated, but they did not include fenamiphos or its metabolites.

Specialized methods

Methods for the determination of fenamiphos and its metabolites in numerous crops, animal tissues, water and soil were reported. All are modified versions of the method of Thornton (1969, 1971). The original method was validated for citrus peel and pulp, pineapple fruit, peanut kernels and hulls, cured tobacco, peanut vines, pineapple bran and pineapple forage. The basic procedure involves homogenisation of the sample with dry ice and acetone, filtration to remove solids, and extraction of the filter cake with CH₃Cl. The CH₃Cl extract is added to the filtrate and the phases are separated. The CH₃Cl phase is dried and concentrated to near dryness. The remaining residue is extracted twice with CH₃Cl and washed with 0.05 N H₂SO₄. The CH₃Cl extracts are combined and evaporated to dryness. The residue is dissolved in acetone and treated with 0.1 molar KMnO₄ and 20% MgSO₄. The mixture is extracted with CH₃Cl and the extracts dried and evaporated. The remaining residue is dissolved in a measured volume of acetone and quantified by GLC as fenamiphos sulfone. With oily substrates such as oilseeds and fats, the oxidized residues are cleaned up on a Florisil column before evaporation and quantification.

Peanut kernels and animal tissues are chopped, blended with acetone and filtered. The filter cake is extracted twice with CH₃Cl, and the extracts are combined and partitioned. The CH₃Cl phase is filtered through Celite and evaporated to dryness. The residue is dissolved in petroleum ether and partitioned against CH₃CN. The lower CH₃CN phase is separated and washed with petroleum ether, which is itself washed with CH₃CN. The combined CH₃CN phases are evaporated to dryness in a rotary evaporator at 40°C.

After oxidation, the samples and the standard are dissolved in acetone and an appropriate aliquot is injected into the gas chromatograph (30 cm x 4 mm borosilicate glass column, 6% QF-1 solution coated on 80-100 mesh Gas Chrom Q, helium as carrier gas at 100 ml/min, phosphorous-sensitive alkali-flame detector).

Recoveries of fenamiphos, fenamiphos sulfoxide and fenamiphos sulfone from numerous crops are shown in Table 61. All analyses were with the original Thornton method (1969, Report 25402), or one of the variants listed by Report number. Where no Report number is given, the original method was used.

Table 61. Recoveries from crop samples by method 00024/I8 or a modification.

Sample	Fortification level, mg/kg	% recovery			Report no.	
		F	FSO	FSO ₂		
Orange peel	0.02	95,105	100, 100	95, 100	91361	
	0.05	90	108	102		
	dry peel	0.05	69			45952
		0.1	89	125	110	
	pulp	0.02	85, 90	85, 90	75, 85	91361
		0.05	108	86	96	
		0.1		79	94	
	leaves	0.5	71	114	131	45952
		0.1	121			
oil	1	70			45952	
wash water	0.05	127			45952	
Peanut kernels	0.1	96	96	79		
	hulls	0.1	71	89		
		0.5	84	97		
Pineapple pulp	0.05	76, 84	74, 104	74, 88	80476, 82388	
	0.1	95, 100	91	94		
	bran	0.05	80, 84	74, 86	74, 84	80476, 82388
		0.1	87, 91		108	
	forage foliage crown	0.1	76	79	82	80476
		0.05	88	86	88	
		0.05	86, 92	76, 88	78, 84	
	0.1	101			80476, 82388	
Apples	0.1	96, 86, 98, 97, 76	95	75	80664, 43979	
	0.05	82, 95	90, 76	80, 79		
Cherries	0.01			130	80432	
	0.02			105	80432	
	0.03			103	80432	
	0.05	77, 84, 98	82, 86, 112	78, 88, 92, 102	43979, 80432	
	0.1	83, 92, 92	73, 77, 81	82, 87, 90, 103	43979	
Peaches	0.05	78, 97	96, 97	66, 78	80704, 43979	
	0.1	82	80	72		
Broccoli	0.05	110	80	73	43979	
	0.05	94		94	35778	
Cauliflower	0.05	86, 80	88	80, 88	43979, 35778	
	0.1	100			35778	
Brussels sprouts	0.01	50,50	70, 70	60, 70	87381,	
	0.02	50	65	85		
	0.05		98	78, 92, 96	35778	
	0.05	84		70		
	0.1	95				
Cabbage	0.05	114		86	35778	
Carrot	0.05	94		90	35778	
	0.1	93		68	35778	
Sugar beet	tops	0.05	90, 106	92, 118	35778	
	root	0.05	100, 102, 116	78, 104, 116		
	juice	0.1	88, 94		110	
		0.05	98			
	pulp	0.1	76		105	

Sample	Fortification level, mg/kg	% recovery			Report no.	
		F	FSO	FSO ₂		
Sweet potatoes	0.05	78		76	35778	
	0.1	81, 90				
Coffee beans	0.05	86	70	78	84626	
	0.1	79, 86				
	0.5	77				
Cotton seed	0.05	135	118	84	66584	
Grapes	0.01	80			66584, 69911, 68204, 69399	
	0.02	80				
	0.03	97				
	0.05	102, 108, 110	92, 92	94, 108		
	0.1	98, 101	100	102		
Raisins	0.05	80	53	84	69459	
Raisin trash	0.05	67	124	57		
Grapefruit	0.1	87, 96, 97, 121	93, 94, 120, 121	99,93	69913	
Lemons	pulp	0.02	90, 100	75, 90	70, 95	91360
		0.05	82	72	92	
	peel	0.02	65, 90	80, 90	85, 85	91360
		0.05	80	102	84	

Modifications to the published method of Thornton (1971) for specific crops are described below.

Ohs (1988a) reported a modification (M002) for the determination of fenamiphos and its sulfoxide and sulfone in tomatoes. Samples are homogenized and blended with acetone. The homogenate is filtered and the filter cake is blended again with acetone. The combined filtrates are concentrated by rotary evaporation (60°C), leaving an aqueous phase which is cleaned up on an Extrelut 20 cartridge. The parent compound and metabolites are eluted with CH₂Cl₂ and the eluate is concentrated to dryness. The residue is taken up in acetone and a 20% MgSO₄ and 0.2 M KMnO₄ solution is added. After 20 minutes, the reaction mixture is cleaned up on Extrelut 20 as described above. The evaporated residue is taken up in ethyl acetate. GLC is on a Megabore column (SE 54 L, 30 m, 0.53 mm i.d.) at a temperature of 200°C (injector temperature 270°C) with an FPD. Recoveries were 91% at 0.02 mg/kg and 100% at 0.20 mg/kg. The typical limit of determination is given as 0.02 mg/kg.

Specht (1995) reported a modification to M002 for bananas (Supplement E019). Fenamiphos sulfone is determined by gas chromatography on a DB-5 30 m fused silica capillary column, 0.53 mm i.d., with a thermionic alkali FID. Temperatures: column 150°C to 250°C; injector 250°C; detector 270°C. Retention time for fenamiphos sulfone: 18 min. Recoveries of fenamiphos sulfone at 0.02, 0.045 and 0.45 mg/kg ranged from 64 to 97%.

Ohs (1998b) reported in Supplement E020 recoveries from bananas at fortification levels of 0.02 and 0.1 mg/kg fenamiphos of 86-88% and 82-91% respectively, with a limit of determination of 0.02 mg/kg, and in Supplement E022 recoveries from watermelons at 0.02 and 0.1 mg/kg of 93 and 87% respectively. The limit of determination was again 0.02 mg/kg.

Recoveries from carrots at fortification levels of 0.02 and 0.1 mg/kg were 88 and 91% (Ohs, 1989, Supplement E024). The limit of determination was 0.02 mg/kg.

Method 0024/18 and modification M002 were revised for the extraction of tobacco, banana pulp and peel, strawberries, melons, peppers, tomatoes and summer squash (zucchini) by Blass (1997a,b). After clean-up of the raw extract on an Extrelut cartridge as in M002, the evaporated residue is transferred to a silica gel cartridge with diethyl ether. Fenamiphos and its sulfoxide and sulfone are eluted with methanol. The oxidation procedure follows.

At fortification levels of 0.02 to 5.0 mg/kg the recoveries were 74 to 101%. The limit of determination is 0.02 mg/kg for carrots and 0.1 mg/kg for tobacco (green and dried). It should be noted that the oxidation may be affected by small volumes of water co-eluted from the Extrelut cartridge. It is recommended that the eluate from the cartridge is dried with anhydrous sodium sulfate and filtered. Recoveries at fortification levels of 0.02, 0.10 and 1.0 mg/kg ranged between 72 and 110% for banana pulp and peel, strawberries, melons, peppers, tomatoes and summer squash.

Other modifications to method 0024/18 include the following.

Brussels sprouts, Report 87381. Celite 545 was substituted for Hyflo Super-Cel; Whatmann 1 was substituted for Whatmann 42 filter paper.

Cherries, Report 80432. After oxidation a heptane/acetonitrile clean-up was incorporated, with 20 ml heptane + 20 ml acetonitrile followed by a second extraction of the heptane phase with 10 ml of acetonitrile.

Lemon pulp, Report 91360. The sample (25 g) is extracted with 150 ml acetonitrile/water (3:2). The extrelut cartridge of M002 is eluted with chloroform instead of dichloromethane. After oxidation the residue is taken up in acetonitrile and partitioned against hexane.

Orange pulp, Report 91361. Orange pulp (25 g) is extracted twice with 150 ml of acetone. CH₂Cl₂ replaces CH₃Cl in the extraction and oxidation steps.

Many of the modifications were to the gas-chromatographic conditions. The modifications (including proposed confirmatory columns) are shown in Table 62, together with conditions reported in the original method.

Table 62. Gas-chromatographic conditions for method 00024/18 and its modifications.

Report No., Sample	Column dimensions	Column packing	Carrier gas (ml/min)	Temperatures, ° C, column injector detector
25402 Method 00024/18	30.5 cm x 4 mm i.d. borosilicate glass	6% QF-1, 80/100 mesh, Gas Chrom Q	Helium (100)	230 225 240
Modifications:				
80664 Apples	20 cm x 4 mm i.d.	6% QF-1, 80/100 mesh Chromosorb W,HP	Nitrogen (80)	205 260 245
87381 Brussel sprouts	61 cm x 2 mm i.d. glass	5% OV-17, 80/100 mesh Gas Chrom Q	Nitrogen	203 230 240
80432 Cherries	38 cm x 2 mm i.d. glas	Ultradond II (Supelco)		210 280 250
84626 Coffee	80 cm x 4 mm i.d.	10% DC-200, 80/100 Chromosorb CW, HP	Nitrogen (100)	215 255 210
69399 Grapes	40 cm x 2 mm i.d.	10% DC-200 + 1.5% OV-210 80/100 mesh Chromosorb W, HP		
69459 Dried raisins	61 cm x 6 mm o.d.	3% OV-210, 80/100 mesh Gas Chrom Q	Nitrogen (110)	240 250 200
69911 Grapes	20.3 cm x 6.3 mm o.d.	3% OV-210, 80/100 mesh Gas Chrom Q	Nitrogen (100)	225 230 200
69913 Grapefruit	40.6 cm x 2 mm i.d. glass	15% OV-210, 80/100 mesh	Helium (40)	200 280

Report No., Sample	Column dimensions	Column packing	Carrier gas (ml/min)	Temperatures, ° C, column injector detector
		Chromosorb W, HP		250
91361 Orange pulp	61 cm x 2 mm i.d.	15% OV-12 x 1.95% OV-210		
80704 Peaches	45.7 cm x 6.3 mm o.d.	3% OV-225 100/120 Gas Chrom Q	Nitrogen (135)	235 230 200
	91.4 cm x 6.3 mm o.d.	3% OV-1 100/120 Chrom W, HP	Nitrogen (150)	240 200 230
	91.4 cm x 6.3 mm o.d.	3% OV-1 100/120 Chrom W, HP	Nitrogen (150)	240 220 230
	45.7 cm x 6.3 mm o.d.	3% OV-210 100/120 Chrom W, HP	Nitrogen (130)	235
80476 Pineapple	91.4 cm x 6.3 mm o.d.	3% OV-1 100/120 mesh Chrom W, HP	Nitrogen (150)	245
82388 Pineapple	20 cm x 4 mm i.d.	6% QF-1 80/100 mesh Chromosorb W, HP	Nitrogen (90)	195 255 220

Method 00224/I389 for the determination of des-isopropyl fenamiphos sulfoxide (DIFSO) and fenamiphos and its sulfoxide and sulfone in crops was reported by Sandie and Gronberg (1981). DIFSO has been detected as a minor and transient metabolite in grapes and citrus. The method has been used in processing studies on grapes, pineapples, peanuts and cotton seed.

Chopped fruit samples are blended with acetone and diluted with water. The extract is filtered and divided into two equal portions, both of which are partitioned three times with CH₃Cl. The CH₃Cl phases are combined and evaporated. The residues are taken up in ethyl acetate and partitioned against water. The ethyl acetate phase is discarded and the water phase partitioned against CH₃Cl. The CH₃Cl is evaporated and the residue dissolved in 20% CH₃CN/H₂O. Portion A (containing F, FSO and FSO₂) is oxidized as previously described and partitioned with CH₃Cl. Portion B is chromatographed in small volumes by HPLC on RP-18 in 20% CH₃CN/H₂O solvent (with UV detection at 220 nm) and the DIFSO fractions are collected and combined. CH₃CN is added before evaporation to dryness. The remaining residue is derivatized by dissolution in BF₃/methanol and allowed to stand overnight at room temperature. Water is added and the DIFSO is extracted with CH₃Cl.

For GLC the fraction containing F, FSO and FSO₂ is dissolved in ethyl acetate and injected onto a borosilicate column (40 cm x 2 mm i.d.; 10% C-200 + 1.5% OV-210 on 80/100 mesh Chromosorb W). The residue is quantified as the sulfone. The DIFSO residue is dissolved in ethyl acetate and injected onto a 25 cm x 2 mm i.d. borosilicate column packed with 5% OV-225 on 60/80 mesh Tenax. The methylated DIFSO is identified by its retention time.

Recoveries of DIFSO from orange peel and pulp at 0.05 mg/kg were 93-102% and from grapes at 0.1 mg/kg or above 83-84%. At levels of 0.01 to 0.05 mg/kg, recoveries ranged from 34 to 115%. The limit of determination was 0.01 mg/kg. Recoveries of F, FSO and FSO₂ from orange peel and pulp fortified at 0.1 mg/kg were 90-118%. The limit of determination was 0.01 mg/kg.

Animal tissues

The determination of fenamiphos and the metabolites FSO, FSO₂, FSOP, FSO₂P, DIF, DIFSO and DIFSO₂ in cattle tissues and milk was reported by Sandie *et al.*, (1978, Method I 270).

Chopped tissue samples except fat are homogenized with acetone and Super-Cel (Celite). The extract is filtered, the filter cake is again extracted with CH_2Cl_2 and acetone, and the extracts are combined. After phase separation, the lower phase is drained through Na_2SO_4 into a flask and evaporated. The residue is taken up in hexane and partitioned twice with acetonitrile. The combined CH_3CN layers are divided into two equal portions and both are evaporated to dryness.

Chopped fat samples are homogenized with hexane and Super-Cel and filtered. The filter cake is suspended in CH_3CN and again filtered. The filtrates are combined and the same procedure as for other tissues is then followed.

Milk is blended with acetone and Super-Cel. The extract is filtered and the filter cake is suspended in CH_2Cl_2 and acetone. The filtrates are combined and shaken, and after phase separation the lower phase is filtered through Na_2SO_4 and concentrated by rotary evaporation. The remaining aqueous layer is once more extracted with dichloromethane and acetone. After phase separation the lower phase is added to the previous one and the combined extract is evaporated to dryness. The residue is dissolved in hexane and partitioned twice with acetonitrile. The combined CH_3CN extracts are divided into two equal portions and evaporated to dryness as before.

One portion of the dry residue is oxidized with KMnO_4 (0.1 molar) in MgSO_4 (20% solution) and extracted with CH_2Cl_2 . The CH_2Cl_2 extract is dried over Na_2SO_4 and evaporated to dryness. The residue is partitioned between hexane and CH_3CN and the CH_3CN phase is washed with hexane. The combined acetonitrile phases are evaporated to dryness. The residue is dissolved in ethyl acetate and 5- μl aliquots are injected into a gas chromatograph equipped with a borosilicate glass column and flame photometric detector (FPD) in the phosphorus mode. Fenamiphos sulfone is identified by its retention time.

The residue from the other half of the sample is dissolved in ethyl acetate and hydrolysed for 10 min in a mixture of benzene and 0.5 N KOH in isopropyl alcohol. The reaction mixture is then transferred to a separatory funnel with isopropyl alcohol and water. After the addition of benzene and shaking, the phases are allowed to separate. The lower aqueous phase is drained into a second separatory funnel and the benzene phase is again partitioned against water. The combined aqueous phases are acidified by the addition of 3 N HCl and partitioned with CH_2Cl_2 . The lower phase is drained through Na_2SO_4 and the partitioning is repeated twice.

After adding 100 μl of 10% mineral oil in hexane, the combined CH_2Cl_2 fractions are evaporated to dryness on an analytical evaporator. The residue is then methylated by adding 0.5 ml of 0.2 M trimethylanilinium hydroxide in methanol and analysed by GLC with an FPD in the sulfur mode.

Recoveries from fortified tissues were 87-117% at 0.1 mg/kg and 73-158% at 0.05 mg/kg in the phosphorus mode, and 84-138% at 0.1 mg/kg and 58-148% at 0.05 mg/kg in the sulfur mode. The limit of determination for all tissues in both modes was 0.01 mg/kg.

Recoveries from fortified milk were 79-115% at 0.005 mg/kg in the phosphorus mode and 84-128% at 0.01 mg/kg and 70-120% at 0.005 mg/kg in the sulfur mode. The limit of determination in both modes was 0.001 mg/kg.

A method for the determination of F, FSO and FSO_2 in soil and turf was reported by Peterson and Winterlin (1986). Samples are homogenized and extracted with ethyl acetate/acetone (4:1). The extract is filtered and the filter cake again extracted. The filtrates are combined and filtered through anhydrous Na_2SO_4 . After evaporation to near dryness the residue, dissolved in benzene, is adsorbed onto a Florisil column. The column is washed with 2.5% acetone in benzene, and fenamiphos and FSO_2 are eluted with 25% acetone in benzene. Fenamiphos sulfoxide is eluted with 80% acetone in benzene. Fenamiphos and its sulfone are further chromatographed on a cellulose column with 25% acetone in benzene. The fraction containing fenamiphos and FSO_2 and that containing FSO are

separately evaporated to dryness and redissolved in appropriate volumes of ethyl acetate and acetone respectively. The samples are analysed by capillary GLC with an NPD (capillary: DB-5, 25 m x 0.251 mm i.d. at 235°C, hydrogen as carrier gas).

Recoveries of fenamiphos averaged 90.2% from soils fortified at 0.005-1.0 mg/kg wet soil and those of fenamiphos sulfoxide and fenamiphos sulfone averaged 83.3% and 95.4% respectively. In turf grass, recoveries of all three compounds were similar. The limit of determination in soil was 0.005 mg/kg and in turf grass 0.01 mg/kg.

The determination of fenamiphos and its sulfoxide and sulfone in water at a limit of 0.1 µg/l was reported by Vorkink (1989). A 500-ml sample is extracted three times with aliquots of CH₃Cl. The combined extracts are evaporated to a volume of 3-5 ml, then transferred to an N-EVAP tube evaporator with gas flow and taken to dryness under nitrogen. The residue is redissolved in toluene and analysed by GLC on a capillary column (25% cyanopropyl, 3 m x 0.2 mm i.d. fused silica) with an NPD.

The recoveries of fenamiphos, FSO and FSO₂ from spiked water were 86.5%, 95.9% and 78.3% respectively at 0.1 µg/l and 101%, 101%, and 102% at 0.5 µg/l. The limit of determination was 0.1 µg/l for all three compounds, with linearity in the range 0.1 to 10 µg/l.

An HPLC method for the determination of fenamiphos phenol sulfoxide, phenol sulfone and phenol sulfonic acid in soil to support terrestrial field dissipation studies was reported by Mattern and Parker (1994). Soil (50 g) is extracted with methanol/water (2:1) and filtered. The filter cake is extracted once more with methanol/water and the filtrates are combined. Methanol is removed by rotary evaporation and the remaining aqueous filtrate is partitioned three times with ethyl acetate. The aqueous phase and the combined ethyl acetate phases are cleaned up separately.

The aqueous phase is passed through an amino-SPE cartridge which is eluted with 0.01 M NH₄OH. The eluate is neutralized with 0.1 M acetic acid, evaporated to dryness at <40°C, and the residue dissolved in water. The organic fraction is evaporated to dryness, redissolved in ethyl acetate, concentrated to <0.1 ml under a stream of nitrogen, diluted in isopropanol/ethyl acetate (3:1) and passed through a 1-g silica gel SPE column which is eluted with the isopropanol/ethyl acetate solvent. The eluate is evaporated under nitrogen and the residue is dissolved in methanol/water (1:1).

The solution from the aqueous fraction is chromatographed by ion-pairing with 0.005 M tetrabutylammonium hydroxide (TBAH) on a reverse-phase C-18 column at pH 5 with a gradient of increasing acetonitrile. The cleaned-up organic fraction is chromatographed on a similar column with 0.1% acetic acid and an increasing proportion of acetonitrile. Detection of all compounds is by UV absorption at 240 nm.

Recoveries of fenamiphos phenol sulfoxide were 88.2 to 111.9% with an average of 97.4%, of fenamiphos phenol sulfone 101.6 to 107.0% with an average of 104.6%, and of fenamiphos phenol sulfonic acid 76.0 to 99.9% with an average of 86.0%. The limits of determination in soil were 0.01 mg/kg for the three analytes.

A method combining electrospray LC-MS-MS and conventional HPLC (Mattern *et al.*, 1998) was also developed to determine fenamiphos and five degradation products in soil in support of field dissipation studies. An internal deuterated fenamiphos standard (0.1 ml of 25 mg/l in acetonitrile) is added to the soil sample (25 g), followed by CH₃CN/water (4:1). The mixture is sonicated for 1 min and filtered, and this extraction is repeated. The CH₃CN is removed by evaporation and the aqueous solution diluted to 50 ml with 1% methanol in water. An aliquot of 1-2 ml of this solution is filtered through a 0.45 µm acrodisc into an autosampler vial for LC-MS-MS analysis. A 100-µl aliquot of this is injected onto an HPLC column (Phenomenex Prodigy 5 ODS-2, 50 x 3.2 mm, 5 µm) and chromatographed with a gradient system from 5 mM NH₄OAc/10% methanol with an increasing

proportion of methanol. The mass spectrometer is operated in the positive-ion mode and optimized to monitor the daughter ions of each analyte.

The remaining aqueous phase from the original acetonitrile/water extract (45 ml) is passed through a 2-g C-18 cartridge conditioned with methanol followed by water. The aqueous eluate is collected and the cartridge rinsed with 7 ml water which is added to the eluate. The cartridge is then eluted with CH₃CN. The organic eluate is evaporated on a Turbovap evaporator and the residue dissolved in 0.5 ml of acetonitrile and diluted to 2.5 ml with water. An aliquot of this is filtered through an acrodisc and transferred into an autosampler vial for the HPLC determination of fenamiphos and its sulfoxide and sulfone. The aqueous eluate from the C-18 cartridge is evaporated to dryness on a rotary evaporator, the residue is dissolved in 2.5 ml of water, and an aliquot is filtered through an acrodisc into an autosampler vial for HPLC determination of the phenol sulfonic acid.

A 200- μ l aliquot is injected into a switched 2-column HPLC (column 1 Alltech Econosil C-18 cartridge; 250 x 4.6 mm i.d., 10 μ m; column 2 MetaChem Inertsil ODS-2, 250 x 4.6 mm i.d., 5 μ m; column-switching system) and chromatographed with a gradient system of 0.1% H₃PO₄ and increasing proportions of acetonitrile. Detection is by UV absorption at 240 nm. The recoveries of the six analytes are shown in Table 63.

Table 63. Average recoveries of fenamiphos and 5 degradation products from soil.

Analyte	Fortified at 0.01 mg/kg	Fortified at 0.1 mg/kg
Fenamiphos	106	102
Fenamiphos sulfoxide	104	104
Fenamiphos sulfone	106	106
Fenamiphos phenol sulfoxide	92	95
Fenamiphos phenol sulfone	87	95
Fenamiphos phenol sulfonic acid	77	84

The limit of determination of each analyte in the test soil was 0.01 mg/kg.

Stability of pesticide residues in stored analytical samples

Lenz (1995) fortified 12 crop commodities with a mixed standard containing fenamiphos, fenamiphos sulfoxide and fenamiphos sulfone at 1.0 mg/kg of each component. The samples were held in frozen storage ($\leq -5^\circ$ C) and analysed after 1, 3, 6, 12, and 18 months. The method used measured all the residues as fenamiphos sulfone by GLC with a thermionic alkali detector.

There was <10% loss or decomposition of fenamiphos residues in garlic and wet and dry grape pomace after 12 months, and in asparagus, bananas, grapes, grape juice, cotton seed, cotton seed meal, and refined cotton seed oil after 18 months. There was <20% decomposition in raisins and cotton seed hulls after 18 months. The results are shown in Table 64.

Freeseaman and Zoloty (1995) conducted a storage stability study of fenamiphos in cow tissues for 89 days and in milk for 61 days.

Fat was extracted with hexane, and kidney, liver, and muscle were individually extracted with acetone, and the extracts fortified at 1 mg/kg with fenamiphos (F), des-isopropyl fenamiphos (DIF), fenamiphos sulfoxide (FSO), des-isopropyl sulfoxide (DIFSO), sulfone (FSO₂), or des-isopropyl sulfone (DIFSO₂). The fortified extracts were stored at -25° C for 3 months, then analysed by HPLC. Cow milk was fortified at 1 mg/kg with F, FSO, or FSO₂ and stored at -25° C for 2 months before analysis.

Fenamiphos and its metabolites were relatively stable (<20% degradation) in kidney and muscle extracts. In the liver extracts only DIFSO and DIFSO₂ showed extensive degradation, 52% and 45% respectively. In the fat extracts only fenamiphos, DIF, and DIFSO₂ showed >50% degradation. The fenamiphos residues were stable in milk (<5% degradation).

Table 64. Stability of fenamiphos residues (sum of fenamiphos, fenamiphos sulfoxide and sulfone) in fortified plant material at -5°C (Lenz, 1995).

Commodity	Storage period, days	% recovery
Asparagus	43	94
	92	93
	180	89
	364	93
	553	92
Banana	92	86
	180	94
	364	100
	553	98
Cotton seed	43	51
	96	91
	180	100
	380	100
	538	100
Cotton seed hulls	43	98
	98	75
	180	90
	384	96
	538	81
Cotton seed meal	40	68
	93	92
	177	73
	377	84
	535	96
Cotton seed oil, refined	40	96
	95	98
	177	100
	377	100
	535	100
Garlic	34	96
	102	98
	208	99
	368	96
Grapes	34	93
	92	73
	184	84
	366	99
	547	99
Grape juice	34	99
	92	97
	184	92
	366	83
	547	100
Wet grape pomace	41	96
	155	99
	418	100
Dry grape pomace	41	99
	155	97
	418	81
Raisins	36	74
	95	82
	175	99
	359	92
	548	84

Under the conditions used to store the samples in the dairy cattle feeding study described later (Wargo, 1978), fenamiphos, if present, could have been degraded to FSO, but all FSO and FSO₂ residues would have remained relatively stable.

In the first part of the analytical method described above for the determination of fenamiphos and its metabolites in animal tissues and milk (Sandie *et al.*, 1978), both fenamiphos and FSO are oxidized to FSO₂, which is quantified as part of the total fenamiphos residue. Any degradation of fenamiphos and FSO to FSO₂ in the original extracts would not affect the total level of FSO₂ determined.

In the second part of the method, the total fenamiphos residues are determined by converting the oxidized fenamiphos, des-isopropyl-fenamiphos and DIFSO to sulfone phenols and determining the methylated phenols. Since DIF and DIFSO are both oxidized to DIFSO₂ before hydrolysis and methylation the observed degradation of DIF to DIFSO would not affect the total amount of methylated phenols.

DIFSO and DIFSO₂ are degraded to the corresponding phenols, but neither DIFSO nor DIFSO₂ was detected in the liver or kidney in the most recent goat metabolism study (Weber and Ecker, 1993), and DIFSO₂ was present in flank muscle but not detected in loin or round muscle. DIFSO and DIFSO₂ would therefore not be expected to be present at significant concentrations in the tissues of cattle, and their instability would not have a marked effect on the overall results of the analyses.

In an earlier storage stability study (Lenz, 1977), ¹⁴C ring-labelled DIF, DIFSO and DIFSO₂ were added to tissues and milk at 1 mg/kg and stored at -10°C. DIFSO and DIFSO₂ were unstable in the liver, kidney and fat, where degradation was predominantly to the corresponding phenols, but were relatively stable in the milk and muscle. The findings are in agreement with the Freeseaman and Zoloty study and are recorded in Table 65.

Table 65. Stability of fenamiphos residues in milk and animal tissues stored at -10°C.

Sample	Storage, days	% recovery of					
		F	FSO	FSO ₂	DIF	DIFSO	DIFSO ₂
Milk	0	96	100	99			
	1				100	95	100
	23						
	38				100	74	87
	61	96	100	100			
	78				74	63	88
Fat	0	95	100	100	96	98	97
	1				100	95	85
	21				94	74	45
	30				81	85	46
	83	42					
	88				3		
	89		92	99		85	47
Kidney	0	95	100	99	80	99	100
	3				98	89	81
	7				93	94	71
	51				81	80	48
	83	79					
	88				88		
	89		100	99		96	91
Liver	0	90	100	99	80	97	95
	1				100	91	82
	7				96	60	46
	14				100	57	45
	83	75					
	84				81	80	48
	88				88		
	89		98	99		40	49

Sample	Storage, days	% recovery of					
		F	FSO	FSO ₂	DIF	DIFSO	DIFSO ₂
Muscle	0	94	100	99	95	99	100
	1				100	92	90
	83	79					
	88				86, 87	82	83
	89		100	99		97	92

F	fenamiphos	DIF	Desisopropyl-fenamiphos
FSO	fenamiphos sulfoxide	DIFSO	Desisopropyl-fenamiphos sulfoxide
FSO ₂	fenamiphos sulfone	DIFSO ₂	Desisopropyl-fenamiphos sulfone

Definition of the residue

The existing definition of the residue is “sum of fenamiphos, its sulfoxide and sulfone, expressed as fenamiphos”. The analytical methods allow all three compounds to be included in the determined residue because fenamiphos and the sulfoxide are both oxidized to the sulfone, so the existing residue definition is appropriate on the basis of the metabolism studies reviewed.

USE PATTERN

Fenamiphos is an organophosphorus nematicide which is registered for use in more than sixty countries. It provides effective control of free-living, root-knot and cyst-forming nematodes. The main target species are *Meloidogyne spp.*, *Pratylenchus spp.*, *Radopholus similis*, *Rotylenchus spp.*, *Rotylenchus reniformis*, *Heterodera spp.* and *Xiphenma spp.* Fenamiphos provides crop protection against nematode damage through systemic activity in the plant and also contact action in the soil.

Fenamiphos is marketed as “Nemacur” and formulated into a granular product (5, 10, 12 and 15 GR) or an emulsion concentrate at 400 g ai/l. In the USA the EC product is formulated at 360 g ai/l.

Fenamiphos may be applied pre-planting, at planting, in established crops or in seedbeds and nurseries. For effective control, it is important that the Nemacur formulations are incorporated into the soil in the zones of root growth, as these are exposed to nematodes. Fenamiphos, fenamiphos sulfoxide and fenamiphos sulfone all exhibit nematicidal activity, thereby providing prolonged activity.

Fenamiphos is predominantly used in tropical and sub-tropical areas in the USA, South America, South Africa, Spain, Australia, Costa Rica and Italy. The product is mainly applied to bananas, citrus fruit, grapes, peanuts, tobacco and vegetables.

Information on numerous registered use patterns was provided by the manufacturer. GAP was also reported by the Australian government. Registered uses are shown in Table 66.

Table 66. Registered uses of fenamiphos.

Crop	Country	Form.	Application				PHI, days	Notes
			Timing	Method	Rate, kg ai/ha	No.		
Apple	Chile	400 EC	Seedling stage	Pre-planting root immersion or spray	40 g ai/100 l	1		19
			Post-planting	In furrow irrigation Drip irrigation	0.4-0.6 g ai/plant 6-8 2.8-4.8	1	45	
	Dominican Republic	12 GR		Incorporation	0.8-1.2 (2.4-4.8 g ai/plant)		60	
	Honduras	12 GR		Incorporation	0.8-1.2 (2.4-4.8 g ai/plant)		60	
	Mexico	10 GR 400 EC		Spreading Spray	2 g ai/m height of tree 6 g ai/tree		45-72	

Crop	Country	Form.	Application				PHI, days	Notes
			Timing	Method	Rate, kg ai/ha	No.		
	Spain	10 GR 400 EC		Spreading/incorp. Spraying	5-10	1	90	
	USA	350 EC		Incorporation	5.45-8.2		72	47
Asparagus	Colombia	10 GR	At planting Established crop	Spreading/incorp.	0.1 g ai/plant 2.5 (0.2 g ai/plant)			
	Mexico	10 GR	After the final cutting	Spreading/drilling	1.2-1.5	2		
Avocado	Dominican Republic	12 GR		Incorporation	0.8-1.2 (2.4-4.8 g ai/plant)		60	
	Honduras	12 GR		Incorporation	0.8-1.2 (2.4-4.8 g ai/plant)		60	
Banana	Argentina	10 GR		Spreading	12 g ai/plant		60	1
	Australia	100 G		Spreading/incorp.	2.5 g ai/plant 1.8 g ai/plant	3		2 1,3
		400 liq.		Soil treatment	24 9.6-12	3		2,6 3,7
	Brazil	10 GR		Spreading/incorp.	2-3 g ai/plant	2	30	
	Colombia	10 GR	At planting At 3 months	Incorporation	1.5 g ai/plant 3 g ai/plant	1-3	60	20
	Costa Rica	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Dominican Republic	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Ecuador	10 GR 15 GR		Spreading	3 g ai/plant	3		25
	El Salvador	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Greece	10 GR 400 EC	At planting Subsequent applications	Incorporation	2 g ai/plant 3-4 g ai/plant 2-4 g ai/plant	1-3	28-42	27
	Guatemala	10 GR 15 GR 400 EC		Spreading/incorp.	5 5.1 2.8-6.8	1	60	
	Honduras	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Mexico	10 GR 400 EC	At planting 4 to 6 months after first application	Spreading Spreading Spraying	1.5-2.5 g ai/corm 3 g ai/irrigation channel 16-32 g ai/10 l	1 2-3	60	
	Nicaragua	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Panama	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Portugal	10 GR 400 EC	Growing fruit	Spreading Spraying	10 2-4 g ai/plant	1		
	South Africa	10 GR	At planting	Incorporation	30-50 g/mat	2		
	Spain	10 GR 400 EC		Spreading/incorp.	1.5-3 g ai/plant 10-20		60 90	
Beans	Argentina	10 GR		Spreading	3.2-4	1	90	1
	Italy	5 GR		Soil incorporation	10-15	1	20	
	Spain	10 GR 400 EC	Pre-planting	Spreading Spraying	5-10 8-10	1	90	
Beetroot	Australia	400 liq.	7 days before planting	Soil treatment	9.6 9	1	84	
Brassicas	Costa Rica	10 GR	At planting Established crops	Spreading/incorp. Band treatment	1 g ai/hole 2-3			
Brussels sprouts	Mexico	10 GR 400 EC		In drills Spraying	1.5-3 3-5 (total/season) 1.6-3.2			
	USA	15 GR	Before planting	Incorporation	6.7 (31-78 g ai/304.8 m 6.7 [1000 ft] row)	1		
Cabbage	Costa Rica	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	Dominican Republic	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	El Salvador	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	Guatemala	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	Honduras	10 GR 15 GR		Spreading/incorp.	4-6	1	60	

Crop	Country	Form.	Application				PHI, days	Notes
			Timing	Method	Rate, kg ai/ha	No.		
	Mexico	10 GR 400 EC		In drills Spraying	1.5-3 3-5 (total/season) 1.6-3.2			
	Nicaragua	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	Panama	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	USA	15 GR	Before planting	Incorporation	6.7 (31-78 g ai/304.8 m [1000 ft] row)			
Carrots	Australia	100 G	7 days before seeding	Incorporation Band treatment	9	1	84	
		400 liq.	7 days before planting	Soil treatment	9.6	1	84 9	
	Costa Rica	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	Dominican Republic	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	El Salvador	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	Guatemala	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	Honduras	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	Italy	5 GR	At planting	Soil incorporation	15	1	90	
	NZ	400 EC	At sowing	Incorporation	3			
	Nicaragua	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	Panama	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
Celery	Australia	400 liq.	7 days before planting	Soil treatment	9.6	1	84 9	
Cherry	Chile	400 EC	Seedling stage	Pre-planting root immersion or spray	40 g ai/100 l	1	19	
			Post-planting	In furrow irrigation Drip irrigation	0.4 0.6 g ai/plant 6-8 2.8-4.8	1	45	
	USA	350 EC		Incorporation	5.45-8.2		45 47	
Citrus	Australia	400 liq.	Spring		30 15	1 1	8	
	Dominican Republic	12 GR		Incorporation	0.8- 1.2 (2.4-4.8 g ai/plant)		60	
	Greece	10 GR 400 EC		Spreading	3-4 g ai/m ²		28-42	
	Honduras	12 GR		Incorporation	0.8- 1.2 (2.4-4.8 g ai/plant)		60	
	South Africa	10 GR 400 EC		Spreading Spraying	12 (40 g ai/tree) 12		150	
	USA	350 EC		Incorporation	1.63-8.2	1-2	30 50	
Cocoa	Brazil	10 GR	At transplanting	Soil treatment	0.05 g ai/plant		45	
	Mexico	10 GR 400 EC	Nursery plants Transplanting	Incorporation Spraying	1-3 g ai/m ² 6-8 g ai/tree 2-2.4 g ai/m ² 60-80 g ai/shrub		45	
Coffee	Brazil	10 GR		Incorporation	1-1.5 g ai/plant 7 g ai/plant	3	45 16 17	
	Colombia	10 GR	At planting 1st year	Spreading/incorp.	1-1.5 g ai/plant 2.5-4 (1-2 g ai/plant)	1-2 2	60 21	
	Costa Rica	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Dominican Republic	10 GR 12 GR 15 GR		Spreading/incorp.	5 2.4-3.6 5.1	1	60	
	El Salvador	12 GR 12.6 GR		Spreading/incorp.	2.4-3.6 3.5-5	1	60 26	
	Guatemala	10 GR 12 GR 12.6 GR 15 GR 400 EC		Spreading/incorp.	5 2.4-3.6 3.5-5 5.1 5.6-6.8; 2.8-3.4	1	60 26 33	
	Honduras	10 GR 12 GR 12.6 GR		Spreading/incorp.	5 2.4-3.6 3.52-5	1	60 26	

Crop	Country	Form.	Application				PHI, days	Notes
			Timing	Method	Rate, kg ai/ha	No.		
		15 GR			5.1			
	Mexico	10 GR	Nursery plants Transplanting	Incorporation	0.2 g ai/bag 1-1.5 g ai/plant	1	45	
	Nicaragua	10 GR 12.6 GR 15 GR		Spreading/incorp.	5 3.52-5 5.1	1	60 26	
	Panama	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
Cotton	Brazil	10 GR	At planting	Incorporation	3-5	1	98? nil	
	Greece	10 GR 400 EC	At sowing	Incorporation	1-2 8; 2-3.4		28-42 60 29	
	Mexico	10 GR 400 EC	At planting	In drill Spraying	1.5-2 1.5-2			
	South Africa	10 GR	At planting	In furrow	15 g ai/100 m row			
	Spain	10 GR 400 EC	Pre-planting	Spreading/incorp.	5-10 4.8-10	1	90	
	USA	150 GR 350 EC		In furrow Band spray	0.84-1.65 0.82-3.27	1	45	
Crucifers	Australia	100 G	7 days before transplanting	Incorporation into soil	9-11	1		
		400 liq.	Pre-planting; 7 days before planting	Soil treatment	9.6	1		
Cucumber	Argentina	10 GR		Spreading	3.2-4	1	90 1	
	Colombia	10 GR		Spreading	0.5-0.8 g ai/plant		60-90	
	Costa Rica	10 GR 15 GR		Spreading/incorp.	2.5-5 2.55-5.1	1	60	
	Dominican Republic	10 GR 15 GR		Spreading/incorp.	2.5-5 2.55-5.1	1	60	
	El Salvador	10 GR 15 GR		Spreading/incorp.	2.5-5 2.55-5.1	1	60	
	Guatemala	10 GR 15 GR		Spreading/incorp.	2.5-5 2.55-5.1	1	60	
	Honduras	10 GR 15 GR		Spreading/incorp.	2.5-5 2.55-5.1	1	60	
	Nicaragua	10 GR 15 GR		Spreading/incorp.	2.5-5 2.55-5.1	1	60	
	Panama	10 GR 15 GR		Spreading/incorp.	2.5-5 2.55-5.1	1	60	
	Spain	10 GR 400 EC	Pre-planting	Spreading/incorp.	5-10 8-10	1	90	
Cucurbits	Australia	400 liq.	7 days before planting	Soil treatment	9.6	1	9	
Egg plant	Italy	5 GR		Soil incorporation	10-15	1	20	
	USA	15 GR 350 EC	At transplanting	Band treatment Incorporation	2.25 2.18	1		
Garlic	Colombia	10 GR	At sowing and 60 days later	Spreading	0.5 g ai/m ² 0.5 g ai/plant	2	60	
	Mexico	10 GR 400 EC	At planting	In drill Incorporation/Spray	1.5-3 2.4-3.2			
	USA	15 GR	At planting	In furrow	2.5-5	1		
Ginger	Australia	100 G	Mid Nov, late Jan	Spreading	11	1		
	South Africa	10 GR	At planting	In furrow	100 g ai/100m row	1	250	
Grapes	Argentina	10 GR	At sprouting	Spreading	3.2-4	1	60 1	
	Australia	400 liq.	Late Sept.	Soil treatment to inter-vine row	12		10	
	Chile	400 EC	Seedling stage	Pre-planting root immersion or spray	40 g ai/100 l	1	19	
			Post-planting	In furrow irrigation Drip irrigation	0.4-0.6 g ai/plant 6-8 2.8-4.8	1	45	
	Colombia	10 GR	At planting	Spreading	0.5 g ai/plant	1	45	
			Young plants		1-2 g ai/plant		45	
			In production		2-3 g ai/plant		45	
	Ecuador	10 GR 15 GR		Spreading	5-10	1		
	Mexico	10 GR 400 EC	At nursery At planting At sprouting Pest activity	Spreading Band application	1-1.5 g/m ² 4-6 4-6 0.4-0.6		Nil 20	

Crop	Country	Form.	Application				PHI, days	Notes
			Timing	Method	Rate, kg ai/ha	No.		
	South Africa	10 GR 400 EC	Before bud burst or after harvest	Spreading/incorp.	1 g ai/m ² or 100 g ai/100 m row	1	100	
	Spain	10 GR 400 EC		Spreading Spraying	5-10 8-10	1	90	
	USA	350 EC		Incorporation	1.63-6.54		2	48
Grapefruit	Argentina	10 GR		Spreading	3.2-4	1	60	1
	Costa Rica	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Dominican Republic	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	El Salvador	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Guatemala	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Honduras	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Mexico	10 GR 400 EC		Spreading Spray	1-2 g ai/m height of tree 8 g ai/l water		180	
	Nicaragua	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Panama	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
Guava	South Africa	10 GR	In spring	Spreading	3 g ai/m ²		120	38
Kiwifruit	NZ	400 EC	At planting	Soaking	16 g ai/10 l soln.			
	USA (CA)	350 EC		Soil treatment	1.63-3.27		31	49
Lemons	Chile	400 EC	Seedling stage	Pre-planting root immersion or spray	40 g ai/100 l	1		19
			Post-planting	In furrow irrigation Drip irrigation	0.4-0.6 g ai/plant 6-8 2.8-4.8	1	45	
	Colombia	10 GR		Spreading	20 g ai/plant		180	
	Costa Rica	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Dominican Republic	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	El Salvador	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Guatemala	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Honduras	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Mexico	10 GR 400 EC		Spreading Spray	1-2 g ai/m height of tree 8 g ai/l of water		180	
	Nicaragua	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Panama	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
Lettuce (endive)	Australia	400 liq.	7 days before planting	Soil treatment	9.6	1	56	9
Litchi	South Africa	10 GR	In spring	Spreading	3 g ai/m ²	1	70	38
Lucerne	NZ	400 EC		Spot treatment	8.8-10			
Mandarins	Colombia	10 GR		Spreading	10-20 g ai/plant		180	
Mangoes	Dominican Republic	12 GR		Incorporation	0.8-1.2 (2.4-4.8 g ai/plant)		60	
	Honduras	12 GR		Incorporation	0.8-1.2 (2.4-4.8 g ai/plant)		60	
Melons/ watermelon	Argentina	10 GR		Spreading	3.2-4	1	90	1
	Brazil	400 EC	At sowing	Trickle irrigation	4	1		
	Colombia	10 GR		Spreading	0.5-0.8 g ai/plant		90	
	Costa Rica	10 GR 15 GR		Spreading/incorp.	2.5-5 2.5-5.1	1	60	
	Dominican Republic	10 GR 12 GR 15 GR		Spreading/incorp.	2.5-5 0.72-1.2 2.5-5.1	1	60	
	El Salvador	10 GR 12 GR 15 GR		Spreading/incorp.	2.5-5 0.72-1.2 2.5-5.1	1	60	

Crop	Country	Form.	Application				PHI, days	Notes
			Timing	Method	Rate, kg ai/ha	No.		
	Guatemala	10 GR 12 GR 15 GR		Spreading/incorp.	2.5-5 0.72-1.2 2.5-5.1	1	60	
	Honduras	10 GR 12 GR 15 GR		Spreading/incorp.	2.5-5 0.72-1.2 2.5-5.1	1	60	
	Italy	5 GR		Incorporation	10-15	1	20	
	Nicaragua	10 GR 15 GR		Spreading/incorp.	2.5-5 2.5-5.1	1	60	
	Panama	10 GR 15 GR		Spreading/incorp.	2.5-5 2.5-5.1	1	60	
	Spain	10 GR 400 EC	Pre-planting	Spreading Spraying	5-10 8-10	1	90	
Mushrooms	Australia	400 liq.		Spray to compost at turning	22-26 g ai/tonne compost	1	42	11
				Casing treatment	22 g ai/tonne of casing	1	42	12
Nectarines	South Africa	400 EC	At establishment	Spraying	1 g ai/m ²		100	42
	USA	350 EC		Incorporation	5.45-8.2		45	47
Olives	Colombia	10 GR		Spreading	20-30 g ai/plant			
Okra	Mexico	10 GR 400 EC		In drill Spray	1.2-1.5 1.2-1.6			
	USA	15 GR	At planting	Incorporation	2.25-2.8	1		
Onions	Argentina	10 GR		Spreading	3.2-4	1	90	1
	Australia	400 liq.	7 days before planting	Soil treatment	9.6	1	84	9
	Colombia	10 GR	At sowing and 60 days later	Spreading	0.5 g ai/m ² 0.5 g ai/plant	2	60	
	Italy	5 GR		Soil incorporation	10-15	1	20	
	South Africa	400 EC	At planting	Incorporation	3	1	80	
Oranges	Argentina	10 GR		Spreading	3.2-4	1	60	1
	Chile	400 EC	Seedling stage	Pre-planting root immersion or spray	40 g ai/100 l	1		19
			Post-planting	In furrow irrigation Drip irrigation	0.4 0.6 g ai/plant 6-8 2.8-4.8	1	45	
	Colombia	10 GR		Spreading	10-20 g ai/plant		180	
	Costa Rica	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Dominican Republic	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	El Salvador	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Guatemala	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Honduras	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Italy	5 GR		Soil incorporation	10-15	1	180	
	Mexico	10 GR 400 EC		Spreading Spray	1-2 g ai/m height of tree 8 g ai/l water		180	
	Nicaragua	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Panama	10 GR 15 GR		Spreading/incorp.	5 5.1	1	60	
	Spain	10 GR 400 EC		Spreading Spraying	5-10 8-10	1	90	
Papaya	South Africa	10 GR	Early spring	Incorporation	4 g ai/m ²			
Parsnips	Australia	100 G (100 g/kg)	7 days before seeding	Incorporation Band treatment	9	1	84	
		400 liq.	7 days before planting	Soil treatment	9.6	1	84	9
Peaches	Italy	5 GR		Soil incorporation	10-15	1	120	
	Mexico	10 GR 400 EC		Spreading Spray	2 g ai/m height of tree 6 g ai/tree		45-72	
	South Africa	400 EC	6 weeks after establishment	Spraying/incorporation	1 g ai/m ²		100	42
	USA	350 EC		Incorporation	5.45-8.2		45	47

Crop	Country	Form.	Application				PHI, days	Notes
			Timing	Method	Rate, kg ai/ha	No.		
Peanuts	Costa Rica	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	Dominican Republic	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	El Salvador	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	Guatemala	10 GR 15 GR 400 EC		Spreading/incorp.	4-6 2.8-3.4; 5.6-6.8	1	60	33
	Honduras	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	Mexico	10 GR 400 EC	At planting	Incorporation	1.5-3 1.6-3			
	Nicaragua	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	Panama	10 GR 15 GR		Spreading/incorp.	4-6	1	60	
	South Africa	10 GR 400 EC	At planting After planting	In furrow Spraying	15 g ai/100m row 1.6-3.2	1	63	39
USA	15 GR 400 EC	At planting	Incorporation Spray in row	1.68-2.85 1.63-2.70	1		44	
Peas	South Africa	400 EC	Pre-planting	Incorporation	1.6		90	41
Pecan nuts	South Africa	10 GR	In spring	Spreading	1 g ai/m ²	1	120	
Peppers	Argentina	10 GR		Spreading	3.2-4	1	90	1
	Dominican Republic	12 GR		Spreading/incorp.	0.72-1.2	1	60	
	Guatemala	12 GR		Spreading/incorp.	0.72-1.2	1	60	
	Honduras	12 GR		Spreading/incorp.	0.72-1.2	1	60	
	Spain	10 GR 400 EC	Pre-planting	Spreading Spraying	5-10 8-10	1	90	
Pineapples	Australia	100 G	Bed preparation	Soil incorporation	100 g ai/100m of bed	1		4
		400 liq.	Pre-plant bed treatment	Soil treatment	100 g ai/100m of bed	1		4
			Plant and ratoon crop	Foliar spray	2.4	5		13
			Ratoon crop	Foliar spray	4.8	2		14
	Colombia	10 GR	At planting Established crop	Spreading	0.1 0.2 g ai/plant 1-1.5 g ai/plant	1-2	60	22
	Costa Rica	10 GR 15 GR		Spreading/incorp.	7-14 7.5-14.1	1	60	
	Dominican Republic	10 GR 12 GR 15 GR		Spreading/incorp.	7-14 2.4-3.6 7.5-14.1	1	60	
	El Salvador	10 GR 12 GR 15 GR		Spreading/incorp.	7-14 2.4-3.6 7.5-14.1	1	60	
	Guatemala	10 GR 12 GR 15 GR 400 EC		Spreading/incorp.	7-14 2.4-3.6 7.5-14.1 2.8-6.8	1-2	60	
	Honduras	10 GR 12 GR 15 GR		Spreading/incorp.	7-14 2.4-3.6 7.5-14.1	1	60	
	Mexico	10 GR 400 EC	At planting At planting	Spreading/incorp. Spray	4-6 10 4		30	34
	Nicaragua	10 GR 15 GR		Spreading/incorp.	7-14 7.5-14.1	1	60	
	Panama	10 GR 15 GR		Spreading/incorp.	7-14 7.5-14.1	1	60	
	South Africa	400 EC	At root formation	Spraying	2.5	3	56	40
	USA*	15 GR 350 EC	Pre-planting	Incorporation Foliar spray	10 5.4-9.8		225	46 52
USA**		Pre-planting Post planting	Spraying	9.8 0.54-3.27		30	51	
Potato	Argentina	10 GR	At planting	Spreading	2	1	90	1
	Australia	100 G	Pre-planting	Soil incorporation	10	1	84	
		400 liq.	Pre-planting and emergence	Soil treatment	5.2	1	84	
	Brazil	10 GR	At planting	Spreading/incorp.	4	1	100	
	Chile	400 EC	To planted or sown crop	Spraying	4-10	1	45	
	Colombia	10 GR	At planting and after	Spreading	2-3	2	45-60	

Crop	Country	Form.	Application				PHI, days	Notes
			Timing	Method	Rate, kg ai/ha	No.		
			germination					
	Costa Rica	10 GR 15 GR		Spreading/incorp.	2.5-5 2.55-5.1	1	60	
	Dominican Republic	12 GR		Incorporation	0.72-1.2	1	60	
	Greece	10 GR 400 EC	At planting	Spreading/incorp.	6-8 4		28-42 60	28
	Guatemala	10 GR 12 GR 15 GR			2.5-5 0.72-1.2 2.55-5.1			
	Honduras	10 GR 12 GR 15 GR			2.5-5 0.72-1.2 2.55-5.1	1	60	
	Italy	5 GR		Soil incorporation	10-15	1	20	
	Mexico	10 GR 400 EC	At planting	In drill Incorporation	4-6			
	NZ	400 EC	At planting	Broadcast spray	8			
	Nicaragua	10 GR 15 GR			2.5-5 2.55-5.1	1	60	
	Panama	10 GR 15 GR			2.5-5 2.55-5.1	1	60	
	Portugal	10 GR	21 days before planting	Spreading	8-10			
	South Africa	400 EC	At planting	Spraying in furrow Aerial	10 1.6 1.2	1 6	42	43
	Spain	10 GR 400 EC	Pre-planting (20 days before sowing)	Spreading Spraying	10 8-10	1	120	
Raspberry	USA	350 EC		Incorporation	3.27-6.54	1	90	
Soya bean	Mexico	10 GR 400 EC		In drill Spray	1.2-2		270	
Strawberry	Australia	100 G	Within one month of planting	Spreading	0.1 g ai/plant	1	42	5
		400 liq.	7 days before planting	Soil treatment	9.6	1	42	15
	Italy	5 GR		Soil incorporation	10-15	1	20	
	USA	15 GR 350 EC	Before transplanting	Incorporation	2-3 1.96-2.94	1	110	
Sugar beet	Greece	10 GR 400 EC	At sowing	Soil treatment	3 8	1	28-42 60	30
	Italy	5 GR		Soil incorporation	10-15	1	20	
	Spain	10 GR 400 EC	Pre-planting	Spreading Spraying	5-10 10-20	1	90	
Sugar cane	Australia	100 G	From planting to early tillering	Band treatment	4	-		
		400 liq.	From planting to early tillering	Directed treatment	4			
Sweet potato	Australia	400 liq.	7 days before planting	Soil treatment	9.6	1	84	9
	NZ (kumara)	400 EC	At planting	Broadcast/incorporation	8			
Tomato	Argentina	10 GR		Spreading	3.2-4	1	90	1
	Australia	100 G	7 days before planting	Spreading; band treatment	11	1		
		400 liq.	7 days before planting	Soil treatment	9.6	1		15
	Brazil	10 GR	At planting	Incorporation	3-4	1	90	18
	Chile	400 EC	To planted or sown crop	Spraying	4-10	1	45	
	Colombia	10 GR	Before of after sowing At transplanting	Spreading/incorp.	1.5-2.5 g ai/m ² 0.2-0.4 g ai/plant	1 2	60-90	23
	Costa Rica	10 GR 15 GR		Spreading/incorp.	2.5-5 2.55-5.1	1	60	
	Dominican Republic	10 GR 12 GR 15 GR		Incorporation	2.5-5 0.72-1.2 2.55-5.1	1	60	
	Ecuador	10 GR 15 GR		Spreading	1.3-3 1.5-3			
	El Salvador	10 GR 15 GR		Spreading/incorp.	2.5-5 2.55-5.1	1	60	
	Guatemala	10 GR 12 GR 15 GR		Incorporation	2.5-5 0.72-1.2 2.55-5.1	1	60	
	Honduras	10 GR		Incorporation	2.5-5	1	60	

Crop	Country	Form.	Application				PHI, days	Notes
			Timing	Method	Rate, kg ai/ha	No.		
		12 GR 15 GR			0.72-1.2 2.55-5.1			
	Italy	5 GR		Soil incorporation	10-15	1	20	
	Nicaragua	10 GR 15 GR		Incorporation	2.5-5 2.55-5.1	1	60	
	Panama	10 GR 15 GR		Incorporation	2.5-5 2.55-5.1	1	60	
	Portugal	10 GR 400 EC		Spreading Spraying	3.4; 3 35 3.2 36	1 1	90 90	
	South Africa	10 GR 400 EC	Pre-planting	Incorporation	1 g ai/m 100 g ai/100 m row	1		
	Spain	10 GR 400 EC	Pre-planting	Spreading Spraying	5-10 8-10	1	90	
Vegetables ***	Greece	10 GR 400 EC	Before planting	Incorporation Spraying	6-8; 4 31 4-8; 6 8; 4 32		28-42 60	

*Puerto Rico only.

** Hawaii only.

***Vegetables include tomato, eggplant, peppers, melon, watermelon, cabbage and cauliflower.

- Can be applied by machinery connected to the tractor or in a steady trickle in the sowing furrow.
- For previously untreated ratoons, where re-treatment is late or where nematode populations are high.
- Treatment of plant crops and subsequent ratoon crops.
- 100 g ai/100 m of bed for a 1 m wide band; e.g. for 0.75 m bed apply 75 g ai/100 m of row.
- 100 g ai/1000 plants or 0.1 g ai/plant.
- 2.4 g ai/stool or 240 g ai/100 m of row or 2.4 g ai/m² of wetted area.
- 120 g ai/100 m of row or 1.2 g ai/stool or 1.2 g ai/m² of wetted area.
- Initial rate is 30 kg ai/ha or 3 g ai/m² for trickle irrigation and maintenance rate is 15 kg ai/ha or 1.5 g ai/m² for trickle irrigation.
- 9.6 kg ai/ha or 6.4 g ai/10 m of row.
- 12 kg ai/ha or 1.2 g ai/m² for trickle irrigation.
- Apply treatment to compost before peak heating at the last turn of compost (20 L of spray mixture/tonne of compost), or after peak heating to compost.
- 4 g ai/bale of peat weighing 50 to 60 kg or 22 g ai/tonne of casing. Do not treat both casing and compost.
- Plant crop and ratoon crop foliar spray. Apply 5 sprays at 2 to 3 month intervals beginning 1 month after planting and ending no later than 6 weeks after flower induction. Immediately after plant crop harvest, apply 2 sprays over the plants 2 to 3 months apart.
- Ratoon crop foliar spray only. If nematodes have infested roots during the plant cycle, apply after harvest of plant crop and an additional spray 4 to 6 weeks later.
- 9.6 kg ai/ha or 6.4 g ai/10 m of row (trickle irrigation included).
- When planting incorporate 1.5 g ai/plant into the hole. On 2 year old coffee plants apply 1 g ai/plant in October, January and April. On coffee plants 3 years old or over, apply 1.5 g ai/plant in October, January and April.
- Label not dated.
- On staked tomatoes apply 0.2 g ai/plant before transplanting the seedlings; the product should be scattered into the hole then incorporated in the soil.
- Root immersion at 40 g ai/100 l. Immerse for 30 minutes or spray for seedlings in bags.
- When in production, apply 3 g ai/plant every 4-5 months at the beginning of the rainy season.
- In the first year apply twice at 1-2 g ai/plant or 2.5-4 kg ai/ha; in subsequent years apply at the same rate twice a year.
- For established plants apply 1-1.5 g ai/plant every 5 to 6 months.
- 1.5-2.5 g ai/m², equivalent to 1.25 to 2.5 kg ai/ha.
- 1-1.5 g ai/m² of seedbed.
- Apply every 4 months.
- For plants less than 1 year old apply 0.6 g ai/plant and for plants older than 1 year apply 1 g ai/plant. Apply once a year in May, June or beginning of July.
- For existing infections apply at 4 g ai/plant then following applications every 3-4 months at 3 g ai/plant.
- Spraying in sowing row or with drip irrigation.
- Before sowing apply 8 kg ai/ha; for existing crops apply 2 to 3.4 kg ai/ha with a drip irrigation system.
- Apply 8 kg ai/ha as a soil treatment before sowing; apply 3 kg ai/ha with a drip irrigation system in existing plantations.
- Apply 6 to 8 kg ai/ha to the soil and incorporate; apply 4 kg ai/ha in the sowing or plantation row; apply 0.8 g ai/ha in the planting hole.
- Apply 4 to 8 kg ai/ha in the row at transplantation or 6 to 8 kg ai/ha as a soil spray before planting or apply 4 kg ai/ha as a spray to the planting row or for drip irrigation.
- Apply 5.6 to 6.8 kg ai/ha as a broadcast spray or 2.3 to 3.4 kg ai/ha as a band treatment.
- Apply to foliage in established plantations at 90 and 150 days.
- Apply 3.4 kg ai/ha 7 to 14 days before planting; apply 3 kg ai/ha 30 days after planting.
- For drip irrigation only; first application before planting; second application 30 days after planting.

37. Commence application at planting or in established plantations in August. In all cases one application should be made in August followed by another in January; applications should be repeated annually in August and January.
38. Apply follow-up treatment annually in spring at 1.5 g ai/m²
39. 63 days for harvest and for use of peanut fodder.
40. Use on the plant crop only. Apply at the first signs of root formation; 2 subsequent sprays are applied at 3 month intervals. A total of 3 crop sprays per crop cycle are required.
41. Do not harvest peas within 90 days of application; do not allow animals to graze pea hay within 120 days of application.
42. Apply within 6 weeks of establishment of young trees in spring and in March-April (after harvest). Apply in 1m² around each tree. Repeat the 2 sprays during the second year, thereafter a treatment after harvest may be made annually.
43. Western Cape only: apply a total of 6 sprays at 1.6 kg ai/ha; first 3 sprays should be applied at weekly intervals commencing at 7 to 10 days after emergence of the plants. The 4th and subsequent sprays are applied at 14-day intervals. For aerial application (1.2 kg ai/ha) a total of 7 sprays are required. Commence spraying 7 to 10 days after plant emergence and repeat at 7-day intervals.
44. Do not feed or graze green peanut vines or peanut hay. Do not hog down treated peanut fields.
45. Do not feed or graze cotton foliage.
46. Post-plant applications can be made in addition to the pre-plant application. Do not apply more than a total of 20 kg ai/ha per crop per season, regardless of the formulation or method of application. Do not use forage or fodder for animal feed.
47. Do not apply more than 8.2 kg ai/ha per year per planting site. Do not graze livestock in treated orchards. Do not feed cover crops grown in treated orchards to livestock.
48. Apply a maximum of 6.54 kg ai/ha per season as a band treatment or low pressure irrigation. The last application may be made up to 2 days before harvest. Do not graze or feed treated crop to livestock.
49. Apply a maximum of 6.54 kg ai/ha per year. Do not graze livestock in treated vineyards. Do not feed cover crops grown in treated vineyards to livestock.
50. For band treatment two applications may be made per season not exceeding 8.2 kg ai/ha. For low pressure irrigation do not exceed a maximum of 6.7 kg ai/ha per season (1.63 to 3.27 kg ai/ha). Do not graze livestock in treated areas. In Florida apply a maximum of 5.45 kg ai/ha for band treatment and for low pressure irrigation apply a maximum of 4.9 kg ai/ha per season.
51. Post-plant application: do not apply more than a total of 26.2 kg ai/ha per plant crop including a pre-plant application. Post-plant applications may be made to the plant crop at 1 to 3 month intervals by foliar spray or drip irrigation. Do not apply more than a total of 9.8 kg ai/ha to each ratoon crop. The first application to the ratoon may be made immediately after crop harvest. Do not use green forage or green fodder for animal feed (cannery waste, such as cull fruit, fruit skin or shells, crowns, cores and basal ends may be fed).
52. Puerto Rico only: apply specified dosage as a foliar spray; begin applications 1 to 3 months after planting. First ratoon crop: make the first application immediately after crop harvest. Do not apply more than a total of 20 kg ai/ha per crop per season regardless of the formulation or method of application. Do not use forage or fodder for animal feed.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Data were provided on supervised trials on numerous crops including carrots, onions, Brussels sprouts, cabbage, peppers, squash, tomatoes, melons, grapefruit, lemons, limes, oranges, apples, cherries, peaches, grapes, bananas, pineapple, peanuts, cotton and coffee. For each crop, the relevant GAP is tabulated with the residue data for ease of comparison.

Residue data, application rates and spray concentrations have been rounded to 2 significant figures, or for residues near the limit of detection to 1 significant figure. Although the trials included control plots, no control data are reported unless residues in the untreated samples exceeded the limit of determination. Results of trials according to GAP are underlined; results used to estimate STMRs are double underlined. All residues, unless otherwise stated, are defined as “sum of fenamiphos, its sulfoxide and sulfone, expressed as fenamiphos”. The limit of determination in each trial is indicated at the end of each Table or in the text if reference is made to validation of a specific method.

Carrots. Trials were conducted in Australia, Italy and Spain. Field reports were not provided for any of the trials in Australia or Spain, nor for some in Italy, so the results are tabulated without any further details (Table 67). Recoveries from carrots using standard method 00024/18 are reported in Table 61 (Report no. 35778) at fortification levels of 0.05 and 0.1 mg/kg; recoveries from other trials are reported in the footnotes to Table 67.

In trials in Italy (Heinemann and Ohs, 1997a) Namacur 10 GR was applied at 15 kg ai/ha to soil before sowing carrots. The granules and carrot seed were scattered manually and incorporated with a rotary cultivator at depths of 5 cm (trials 501581 & 506664) and 10 cm (trials 506672 &

506680). Trial plots were 100 m² and the soils were sand, clay, and sandy loam. The treatments were applied 90 or 91 days before harvest of the first carrots; samples of roots were prepared by lightly washing in cold water. Details are shown in Table 67.

In subsequent trials (Heinemann and Ohs, 1997b) Nemacur 10 GR was applied to the soil at 30 kg ai/ha, twice the rate in the first trials, before sowing carrots. The granules and carrot seed were scattered manually and incorporated with a rotary cultivator at depths of 5 cm (trials 602752 & 603562) and 10 cm (trials 603570 & 603589). Trial plots were 40 to 80 m² and the soil at three sites was sandy; no information was given for the fourth site. The treatments were applied 90 days before the first carrots were harvested. The samples were cleaned with paper before extraction.

Two further trials were conducted at two sites in Italy in 1996 (Ohs, 1998). Nemacur 5 GR or 10 GR was applied to seedbeds before sowing carrots at a rate of 15 kg ai/ha. The plot sizes were 98 m² and the soils were sandy (pH 7.9 and 0.2% C). The products were spread by hand and incorporated with a rotary hoe. Samples were taken at 90, 100, 131 and 143 days after application; adhering soil was removed by rinsing with cold water.

Table 67. Residues from supervised trials on carrots in Australia, Spain and Italy.

Location, year (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
Australia, (VIC), 1972	400 EC	8.96	1*	107 117 136	'baby' carrot	<u>0.05</u> 0.04 0.02	Bayer Australia 1971a 27/71a ¹
Australia, (VIC), 1971	400 EC	8.96	1**	93 103 122	'baby' carrot	<u>0.08</u> 0.07 0.04	27/71b ¹
Australia, (VIC), 1971	400 EC	13.44	1**	93 103 122	'baby' carrot	0.13 0.1 0.09	27/71c ¹
Australia, (VIC), 1986 (Western red)	10 GR	11	1	84	root (early maturity)	<u>0.02</u>	Bayer Australia 1986 18/86a ²
Australia, (VIC), 1986 (Western red)	10 GR	22	1	84	root (early maturity)	0.05	18/86b ²
Spain, (Cadiz), 1981 (Tim tom)	10 GR	10	1	136	root	0.05, <u>0.06</u>	Bayer 1981a 5200-81 ³
Spain, (Cadiz), 1981 (Tim tom)	10 GR	10	1	136	root	0.05, <u>0.06</u>	5201-81 ³
Spain, (Cadiz), 1981 (Tim tom)	400 EC	9.6	1	136	root	0.01, 0.01	Bayer 1981b 5202-81 ³
Spain, (Cadiz), 1981 (Tim tom)	400 EC	9.6	1	136	root	0.1, 0.11	5203-81 ³
Italy, (Latina), 1989 (Delta cuore rosso)	5 GR	10	1	150	root	< <u>0.02</u> , <0.02	Bayer 1988a 0187-88 ⁴
Italy, (Latina), 1989 (Delta cuore rosso)	5 GR	10	1	65 85	root	0.045, <u>0.05</u> 0.041, 0.048	Bayer 1988a 0411-88 ⁵
Italy, (Latina), 1995 (Nandor)	10 GR	15	1	90 100 130	root	< <u>0.02</u> , <0.02 <0.02 <0.02, <0.02	Heinemann and Ohs 1997a 501581 ⁶
Italy, (Latina), 1995 (Bolero)	10 GR	15	1	90 100 130	root	< <u>0.02</u> , <0.02 <0.02 <0.02, <0.02	506664 ⁶
Italy, (Sicily), 1995 (Nelson)	10 GR	15	1	91 100 130	root	<0.02, <0.02 <0.02 0.067, <u>0.070</u>	506672 ⁶
Italy, (Sicily), 1995	10 GR	15	1	90	root	1.19, 0.929 (0.40 c)	

Location, year (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
(Junior)				100 131		0.602 (0.19 <i>c</i>) 0.093 (0.10 <i>c</i>)	506680 ⁶
Italy, (Latina), 1996 (Nantes 3 Tip-top)	10 GR	30	1	90 100 130	root	<0.02, <0.02 <0.02 <0.02, <0.02	Heinemann and Ohs 1997b 602752 ⁷
Italy, (Latina), 1996 (Bolero F1))	10 GR	30	1	90 100 130	root	<0.02, <0.02 <0.02 <0.02, <0.02	603562 ⁷
Italy, (Ferrara), 1996 (Nanthia)	10 GR	30	1	90 100 130	root	<0.02, <0.02 <0.02 <0.02, <0.02	603570 ⁷
Italy, (Verona), 1996 (Nentes 5 Sansom)	10 GR	30	1	90 100 131	root	<0.02, <0.02 <0.02 <0.02, <0.02	605589 ⁷
Italy, (Sicily), 1997 (Nanco)	10 GR	15	1	90 100 131 143	root	0.023, 0.024 <0.02 <0.02, <0.02 <0.02	Ohs 1998 608572 ⁸
Italy, (Sicily), 1997 (Nanco)	5 GR	15	1	90 100 131 143	root	0.027, 0.022 <0.02 <0.02, <0.02 <0.02	608580 ⁸
GAP							
Australia	100 G	9	1	84			
	400 liq.	9.6	1	84			
Italy	5 GR	15	1	90			

c Controls.

*Applied 21 days before sowing.

**Applied 7 days before sowing.

¹ Results corrected for recovery; limit of detection 0.01 mg/kg; recovery 79% at 0.5 and 72% at 0.1 mg/kg.

² Applied at sowing. Limit of detection = 0.05 mg/kg; recovery with fortification at 0.5 mg/kg = 98%.

³ Applied 2 days after sowing by spreading and incorporation. Limit of determination = 0.01 mg/kg; recovery at that concentration = 86%.

⁴ Applied 10 days before sowing. Limit of determination = 0.02 mg/kg; recovery at that concentration = 87%.

⁵ Applied 1 day before sowing. Limit of determination = 0.02 mg/kg; recovery at that concentration = 81%.

⁶ Recoveries of fenamiphos 76-87% at 0.02 mg/kg (n = 6); 69-92% at 0.1 mg/kg (n = 11); 74-84% at 1 mg/kg (n = 6).

⁷ Limit of determination = 0.02 mg/kg. Recoveries of fenamiphos 71-82% at 0.10 mg/kg (n = 4).

⁸ Limit of determination = 0.02 mg/kg. Recovery of fenamiphos 89 and 104% at 0.02 and 73 and 78% at 0.1 mg/kg.

In three of the four Italian trials in 1995, residues in carrots were below the limit of determination. In the fourth however the residues were high and finite levels were found in the control samples. The higher residues were explained as being due to bad weather conditions and the bad condition of the plot. In trial 506680, the average unit weight of the samples was much lower than the average in the other trials: 2-28 g compared with 56-113g, 78-150 g and 14-54 g. The composite sample weights were 1.36 to 2.03 kg in trial 506880, compared to 2.2-2.4 kg, 2.0-3.3 kg and 2.0-2.1 kg in trials 501581, 506664 and 506672. The data from trial 506680 are not considered valid for estimating a maximum residue level or STMR. In the 1997 trials, residues above 0.02 mg/kg were found at day 90 but all later residues were below the limit of determination after treatment at 15 kg ai/ha.

From the 1996 trials it is evident that doubling the application rate still gives fenamiphos residues below the limit of determination in carrots sampled at 90 days or later. These results can be used in the estimation of the maximum residue level and STMR

The residue data are in accord with the findings in the metabolism study of Khasawin (1973c) where a high proportion of the radioactivity in carrots was present as conjugated derivatives

of fenamiphos phenol sulfoxide and fenamiphos phenol sulfone, neither of which is encompassed by the existing residue definition.

Potatoes. Supervised trials were conducted in Spain and Australia. All results were presented in summary form; in some cases recoveries were not reported. Chromatograms were not provided for any of the trials. The results are shown in Table 68.

Table 68. Residues from supervised trials on potatoes. Tubers analysed.

Location, year (variety)	Application			PHI, days	Residue, mg/kg	Reference
	Form.	kg ai/ha	No.			
Spain (La Puebla), 1975, (Royal Kidney)	5 GR	10	1	118	0.07	Bayer 1975 5200B-75 ¹
Spain (La Puebla), 1977, (Maris Piper)	5 GR	10	1	97 104 111 125	0.17 0.12 0.08 0.04	Bayer 1977 5204-77 ²
Spain (Mallorca), 1982, (Baris-Bard)	10 GR	10	1	71 92 105	<0.01 <0.01 <0.01	Bayer 1982 5200-82 ³
Spain (Mallorca), 1982, (Irish Pace)	10 GR	10	1	71 92 105	<0.01 <0.01 <0.01	5201-82 ⁴
Australia (WA), 1971, (Delaware)	400 EC	4.47	1	96	<0.05	Bayer Australia 1971b 35/71a ⁵
Australia (WA), 1971, (Delaware)	400 EC	8.96	1	96	<0.05	35/71b ⁶
GAP						
Australia	100 G	10	1	84		
	400 liq.	5.2	1	84		
Spain	10 GR	10	1	120		
	400 EC	8-10	1	120		

¹ Single application by spreading and incorporation; plot size 1700 m², sandy loam soil. Samples taken 1 week before harvest. Limit of detection = 0.01 mg/kg; recoveries not reported.

² Single application by spreading and incorporation; plot size 1700 m², sandy loam soil. Samples taken 2 weeks after harvest. Limit of detection = 0.01 mg/kg; recoveries not reported.

³ Single application at planting by spreading and incorporation; plot size 50 m², sandy soil, pH 7, 1% C. Samples taken at harvest. Limit of detection = 0.01 mg/kg, recovery at 0.05 mg/kg = 94%.

⁴ Single application at planting by spreading and incorporation; plot size 25 m², sandy soil, pH 7, 3% C. Samples taken at harvest. Limit of detection = 0.01 mg/kg, recovery at 0.05 mg/kg = 94%.

⁵ Single application by spraying at 24 hours before planting. Limit of detection 0.01 mg/kg, recovery at 0.1 mg/kg = 78%, recovery at 0.5 mg/kg = 80%. Results corrected for recovery.

⁶ Single application by spraying at 24 hours before planting. Limit of detection = 0.01 mg/kg, recovery at 0.1 mg/kg = 95%, recovery at 0.5 mg/kg = 98%. Results corrected for recovery.

The results of trials according to GAP for Australia or Spain ranged from <0.01 to 0.17 mg/kg.

Onions. Trials in Australia and South Africa were reported (Table 70). Field details of the Australian trial were not given.

In the South African trials, NemaCur 400 EC was applied at 3, 4 or 6 kg ai/ha 4 days before sowing onions or at planting. The plot sizes were 600 to 800 m². Samples of onion bulbs were taken at maturity. In trial 311/88475/E517 the bulbs were half-developed at the first sampling and fully-developed at the final sampling. The results are shown in Table 69.

Table 69. Fenamiphos residues in onions resulting from supervised trials in Australia and South Africa.

Location, year (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
Australia (SA), 1971, (Brown)	43.6% EC	9.7	1	151	mature bulb	<0.01	Bayer Australia 1971c 29/71 ¹
South Africa, (Transvaal) 1990, (Hojem)	400 EC	4	1	139	bulb	0.05, 0.05	S. Afr Bur. Stds. 1990a 311/88475/E517 ²
				151		<0.05, <0.05	
				161		<0.05, <0.05	
				172		<0.05, <0.05	
200	<0.05, <0.05						
South Africa, (Western Cape), 1988, (Caledon globe)	400 EC	4	1	78	bulb	0.05, 0.05	311/88608/F237 ³
				88		<0.05, <0.05	
				101		0.05, 0.05	
South Africa, (Western Cape), 1990, (Caledon globe)	400 EC	3	1	72	bulb	<0.05, <0.05	311/88817/G160 ⁴
				81		<0.05, <0.05	
		91	<0.05, <0.05				
		6	1	72		<0.05, <0.05	
		81		<0.05, <0.05			
91	<0.05, <0.05						
GAP							
Australia	400 liq.	9.6	1	84			
South Africa	400 EC	3	1	80			

¹ Applied to soil by boom spray, then incorporated with tynes and rolled 5 days before sowing. Limit of detection = 0.01 mg/kg; recovery 82% at 0.5 mg/kg, 77% at 0.1 mg/kg; results corrected for recovery.

² Limit of detection = 0.05 mg/kg; recovery of fenamiphos = 74%, fenamiphos sulfoxide = 93% and fenamiphos sulfone = 86% at 0.1 mg/kg; results corrected for recovery.

³ Limit of detection = 0.05 mg/kg; recovery of fenamiphos = 74%, fenamiphos sulfoxide = 93%, fenamiphos sulfone = 86% at 0.1 mg/kg; results corrected for recovery.

⁴ Limit of detection = 0.05 mg/kg; recovery of fenamiphos = 82%, fenamiphos sulfoxide = 94%, fenamiphos sulfone = 100% at 0.1 mg/kg.

The trials were at rates in accordance with or in excess of those registered in Australia or South Africa. Results considered acceptable for the estimation of a maximum residue level and STMR are indicated; most results were corrected for recovery. As the samples in the Australian trial were taken later than the specified PHI and field details were not provided, the result was not included in the estimation. In the South African trials, although samples were taken later than the label PHIs, as the crop was mature and the application was either pre-planting or at sowing the trials may be considered to comply with the specified GAP.

Brussels sprouts. Several trials were conducted in the USA; field details were not reported. The registered use pattern for Brussels sprouts in the USA allows a single application equivalent to 6.7 kg ai/ha with no specified PHI. Recoveries by Method 00024/I8 with fortification at 0.01-0.1 mg/kg are reported in Table 61. The results of the trials are shown in Table 70.

Table 70. Residues from supervised trials on Brussels sprouts in the USA.

Location, year, (variety)	Application			PHI, days	Residue, mg/kg	Reference.
	Form.	kg ai/ha	No.			
Washington, 1973 (Jade)	15 GR	10	1	107	0.02	Chemagro 1973a 35889
Virginia, 1973 (Catskill)	15 GR	6.7	1	133	<0.01	35947
California, 1973 (J. Cross)	359 g/l SC	6.7	1	113	<0.01	Chemagro 1973b 35946
Virginia, 1973 (Catskill)	359 g/l SC	6.7	1	133	<0.01	35948
California, 1986 (Lannette)	15 GR	6.7	1	124	<0.01	Mobay 1986a 87382
California, 1986 (Lannette)	15 GR	6.7	1	157	<0.01	87383
California, 1986 (Lannette)	15 GR	6.7	1	157	<0.01	87384
GAP						
USA	15 GR	6.7	1	NS		
Mexico	10 GR	3 or 5/season	1	NS		

All of the trials except 35889 were at the maximum label rate of 6.7 kg ai/ha and residues were below the limit of determination. Trial 35889 was at 10 kg ai/ha and the PHI was shorter.

Cabbage. Supervised trials were in Australia and the USA. In many cases field details were not given. Recoveries from cabbage in the US trials by Method 00024/I with fortification at 0.05 mg/kg were reported in Table 61. The results of the trials are shown in Table 71.

Table 71. Residues in cabbage from trials in Australia and the USA.

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.
	Form.	kg ai/ha	No.				
Australia (WA), 1971 (Comet)	5 GR	8.9	1	106	mature head	<0.01	Bayer Australia 1971d 21/71c ¹
Australia (WA), 1971 (Comet)	5 GR	17.9	1	106	mature head	<0.01	21/71d ¹
Australia (WA), 1971 (Comet)	400 EC	8.9	1	106	mature head	<0.01	Bayer Australia 1971e 21/71a ²
Australia (WA), 1971 (Comet)	400 EC	17.9	1	106	mature head	<0.01	21/71b ²
Australia (WA), 1971 (Comet)	400 EC	4.48	1	42	head	<0.01	22/71a ³
Australia (WA), 1971 (Comet)	400 EC	8.9	1	42	head	<0.01	22/71b ³
Australia (WA), 1971 (Comet)	400 EC	17.9	1	42	head	0.04	22/71c ³
USA (Minnesota), 1973 (Golden acre)	15 GR	6.7	1	55	head	0.02	Chemagro 1973c 35890 ⁴
USA (Kansas), 1973 (Golden acre)	15 GR	6.7	1	65	head	<0.01	35891 ⁴
USA (California), 1973 (Danish boldhead)	15 GR	6.7	1	83	head	<0.01	35892 ⁴
USA (Nth Carolina), 1973 (Market prize)	15 GR	10	1	108	head	0.02 (0.13c)	35893 ⁴
USA (Kansas), 1973 (Golden acre)	15 GR	10	1	65	head	<0.02	35894 ⁴
USA (Nth Carolina), 1973 (Market prize)	15 GR	10	1	108	head	0.05	35954 ⁴
USA (Nth Carolina), 1973 (Market prize)	15 GR	6.7	1	108	head	<0.01	35955 ⁴
USA (Virginia), 1973 (Market prize)	15 GR	6.7	1	84	head	0.02	35956 ⁴
USA (Minnesota), 1973 (Golden acre)	3 SC	6.7	1	55	head	0.02	Chemagro 1973d 35895 ⁴
USA (Kansas), 1973 (Golden acre)	3 SC	6.7	1	65	head	<0.01	35896 ⁴
USA (California), 1973 (Danish boldhead)	3 SC	6.7	1	79	head	0.01	35897 ⁴
USA (California), 1973 (Danish boldhead)	3 SC	10	1	79	head	<0.01	35898 ⁴
USA (Virginia), 1973 (Market prize)	3 SC	6.7	1	84	head	<0.01	35952 ⁴
USA (Nth Carolina), 1973 (Market prize)	3 SC	10	1	108	head	0.02	35953 ⁴
USA (Indiana), 1977 (Golden acre)	3 SC	3.7	1	68	head wrapper leaves field trash	<0.01 <0.01 <0.01	Chemagro 1977a 53228 ⁴

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.
	Form.	kg ai/ha	No.				
USA (Wisconsin), 1977 (Roundup)	3 SC	2	1	84	head	<0.01	53229 ⁴
					wrapper leaves field trash	0.03 0.12	
USA (Indiana), 1977 (Golden acre)	3 SC	3.7	1	68	head	<0.01	53230 ⁴
					wrapper leaves field trash	0.01 0.02	
USA (Wisconsin), 1977 (Roundup)	3 SC	3.7	1	84	head	<0.01	54350 ⁴
					field trash	0.19	
GAP							
Australia	100 G	9	1	NS			
	400 liq.	9.6	1	NS			
USA	15 GR	6.7	1	NS			

¹ Applied 1 day before transplanting seedlings. Limit of detection = 0.01 mg/kg; recovery = 86% at 1 mg/kg, 84% at 0.5 mg/kg and 80% at 0.1 mg/kg. Area of plot treated = 1.85 m²; granules distributed by hand then rotary hoed in.

² Applied 1 day before transplanting seedlings. Limit of detection = 0.01 mg/kg; recovery = 86% at 1 mg/kg, 84% at 0.5 mg/kg and 80% at 0.1 mg/kg. Area of plot treated = 1.85 m²; spray applied then rotary hoed in.

³ Applied at planting. Limit of detection = 0.01 mg/kg; recovery = 86% at 1 mg/kg, 84% at 0.5 mg/kg and 80% at 0.1 mg/kg. Area of plot treated = 0.18 ha; applied by boom spray then rotary hoed in; samples taken 1-2 weeks before normal harvest.

⁴ Limit of detection = 0.01 mg/kg.

Registered use patterns in Australia allow a single pre-planting application at 9 or 9.6 kg ai/ha; a PHI is not specified. Only three trials in Australia were in accordance with GAP, as rates in the remaining four trials were either half or twice the maximum label rate. After application at the maximum or twice the maximum rate, the fenamiphos residues in the cabbage heads in all but one of the Australian trials were below the limit of detection.

The registered use pattern for Namacur 15 GR in the USA allows a single application before or at planting at a rate equivalent to 6.7 kg ai/ha with no specified PHI. The rates in the trials were 0.5, 1 or 1.5 times the maximum rate. The residues in the cabbage heads were below the limit of determination in most of the trials. Wrapper leaves and field trash were also analysed in trials 53228 to 53230 and 54350, where half the GAP rate was applied. Higher residues were found in some of these samples, at levels up to 19 times the residues in the heads. Similar findings were reported in the cabbage metabolism study (Khasawinah, 1973d), where higher levels of radioactivity were present in the outer leaves than in the whole head. The radioactivity was predominantly due to fenamiphos sulfoxide and fenamiphos sulfone.

The data on outer leaves and field trash are useful for estimating any exposure to livestock, which may be fed waste products and non-commercial crop parts after harvest. It was noted that field details were not reported in these trials.

Peppers. Supervised trials were conducted in Italy, Spain and Portugal. The results are shown in Table 72.

In glasshouse trials in Italy and Spain, Namacur 240 CS (capsule suspension) was applied to pepper plants by drip irrigation (Heinemann and Ohs, 1997c). The product was applied at inflorescence emergence or at fruit development, at a rate equivalent to 10 kg ai/ha. Fruits were sampled at 30/31, 60 and 90 days after treatment. The plot sizes were 40 to 845 m² and the soils at all sites were sandy. The pH of the soil was 5.7 and 7.4 in trials 602779 and 603937 respectively, where the soil was covered with black foil and the treatment applied directly to the base of the plants; in trials 603945 and 603953 the product was applied via the irrigation system. The results are shown in Table 72.

In a subsequent set of glasshouse trials in Italy, Spain and Portugal (Blass, 1998a), Namacur 240 CS was applied by irrigation at a rate equivalent to 10 kg ai/ha. A single application was made to pepper plants at flowering or early fruiting stages and samples were taken 30, 60 and 90 days after

treatment. Plot sizes were 50 to 336 m² and at all sites the soil was sandy in the pH range 6-8.4. In the Italian trials, the soil was covered with black plastic foil.

Table 72. Residues in peppers from trials in Italy, Spain and Portugal

Location, year, (variety)	Application			PHI, days	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.			
Italy (Lonigo), 1980	5 GR	10 F	1	84	<u>0.05</u>	Bayer 1981c 5205-80 ¹
Italy (Lonigo), 1980	5 GR	10 F	1	84	<u>0.05</u>	5206-80 ¹
Spain (San Javier), 1985 (Gedeon)	10 GR	10 G	1	1 15 29 56 84	<0.05, <0.05 <0.05, <0.05 0.05, 0.1 0.09, 0.1 0.31, <u>0.35</u>	Bayer 1985a 5208-84 ²
Spain (Murcia), 1985 (Gedeon)	400 EC	10 G	1	1 15 29 56 85	<0.05, <0.05 0.18, 0.18 0.28, 0.37 0.19, 0.20 0.25, <u>0.26</u>	Bayer 1985b 5207-84 ³
Spain (Alicante), 1986 (Gedeon)	400 EC	5 G	2	90 118 153	0.13, 0.17 0.05, 0.06 0.05, 0.05	Bayer 1986a 5203-86 ⁴
Italy (Sabaudia), 1987 (Eldor)	5 GR	14.4 F	1	65 81	<0.02, <0.02 <u><0.02</u> , <0.02	Bayer 1987a 5224-87 ⁵
Spain (Alicante), 1987 (Lamuyo)	10 GR	10 F	1	63	<u><0.05</u> , <0.05	Bayer 1987b 5235-87 ⁶
Spain (Semillas Llad), 1987, (Hungaro)	10 GR	10 F	1	75	<u>0.06</u> , 0.06	5236-87 ⁷
Spain (Alicante), 1987, (Lamuyo)	400 EC	10 F	1	50	0.08, 0.08	Bayer 1987c 5217-87 ⁸
Spain (Semillas Llad), 1987 (Hungaro)	400 EC	10 F	1	75	0.05, <u>0.06</u>	5218-87 ⁹
Italy (Latina), 1996, (Sonar)	240 CS	10 G	1	30 60 90	0.119, 0.108 <0.02, <0.02 <0.02	Heinemann & Ohs 1997c 0277-96 ¹⁰
Italy (Ragusa), 1996, (Lux)	240 CS	10 G	1	31 60 90 (green)	0.041, 0.033 <0.02, <0.02 <0.02	0393-96 ¹⁰
Spain (Almeria), 1996 (Anibal)	240 CS	10 G	1	90 (yellow) 31 60 90	<0.02 0.067, 0.071 0.02, 0.02 <0.02	0394-96 ¹⁰
Spain (Almeria), 1996 (Drago)	240 CS	10 G	1	31 60 90	0.187, 0.177 0.07, 0.064 <0.02	0395-96 ¹⁰
Italy (Latina), 1997, (Gordo)	240 CS	10 G	1	30 60 90	<0.02, <0.02 <0.02, <0.02 <0.02	Blass 1998a 0099-97 ¹¹
Italy (Ragusa), 1996, (Soldi)	240 CS	10 G	1	30 60 90	0.091, 0.088 0.110, 0.096 <0.02	0558-97 ¹¹
Spain (Almeria), 1996 (Roldan)	240 CS	10 G	1	30 60 90	<0.02, <0.02 <0.02, <0.02 <0.02	0559-97 ¹¹
Portugal (Lissabon), 1997, (Sonar)	240 CS	10 G	1	30 60 90	0.306, 0.287 0.80, 0.065 <0.02	0560-97 ¹¹
GAP						
Spain	10 GR	5-10	1	90		
	400 EC	5-10	1	90		

F: field G: glasshouse

¹ Field trial. Limit of determination = 0.05 mg/kg; recoveries not indicated. Treatment applied 20 days before planting. Plot size = 10 m².

² Glasshouse trial; single application by spreading by hand and incorporation in soil followed by irrigation; applied at the fruiting stage; plot size = 112 m²; soil pH 7.5; loamy soil, 1% C. Recovery = 100% at 0.1 mg/kg. Limit of determination = 0.05 mg/kg.

³ Glasshouse trial; single application by drip irrigation; applied at the fruiting stage; plot size = 36 m²; soil pH 7.5; loamy sand, 1% C; recovery = 100% at 0.1 mg/kg. Limit of determination = 0.05 mg/kg.

⁴ Glasshouse trial; 2 applications by drip irrigation, 1st at planting and 2nd 14 days after planting; plot size = 36 m²; pH 7.5-8.0; sandy soil 1% C; recovery = 82% at 0.1 mg/kg, 95% at 0.01 mg/kg. Limit of determination = 0.05 mg/kg.

⁵ Field trial; single application 11 days after planting by spreading; plot size = 3000 m² (0.3 ha); sandy soil; irrigation every 8-10 days by sprinkler. Limit of determination = 0.02 mg/kg, recovery at 0.02 mg/kg = 103%.

⁶ Field trial; single application in-furrow at planting with slight incorporation; plot size = 100 m²; sandy soil pH 7.5, 2% C. Limit of determination = 0.05 mg/kg; recovery at 0.05 mg/kg = 70%.

⁷ Field trial; single application at sowing by spreading and incorporation; plot size = 10 m²; sandy soil pH 7, 1% C. Limit of determination = 0.05 mg/kg, recovery at 0.05 mg/kg = 70%.

⁸ Field trial; single application 13 days after planting (8-leaf stage) by drench spray; plot size = 100 m²; sandy soil pH 7.5, 2% C. Limit of determination = 0.05 mg/kg, recovery at 0.05 mg/kg = 70%.

⁹ Field trial; single application at sowing by spraying and incorporation; plot size = 10 m²; sandy soil pH 7, 1% C. Limit of determination = 0.05 mg/kg, recovery at 0.05 mg/kg = 70%.

¹⁰ Limit of determination = 0.02 mg/kg, recovery at 0.02 mg/kg 79-100% (n = 6), recovery at 0.1 mg/kg 76-94% (n = 10), recovery at 1 mg/kg 83-94% (n = 6).

¹¹ Limit of determination = 0.02 mg/kg, recovery at 0.02 mg/kg = 81, 86%, recovery at 0.1 mg/kg 75-93% (n = 5).

The trials with the GR and EC products can be compared with registered use patterns in Spain, which allow a single pre-planting application at a maximum rate of 10 kg ai/ha and a PHI of 90 days. In both field and glasshouse trials fenamiphos residues 90 days after treatment were <0.02-0.06 mg/kg, after application at sowing. In trials where the application was made after planting, at flowering or early fruiting, higher residues in the range 0.25-0.35 mg/kg were present 84 or 85 days after treatment. As the specified GAP for the 400 EC product indicates application before sowing or at transplanting these results may not provide an accurate indication of likely residues after treatment at an early stage, but they give an indication of residues that may occur after late application.

The results with the 240 CS formulation are included although registration of the product is pending and only draft labels were provided.

Tomatoes. Numerous supervised trials were conducted in Australia, Brazil, South Africa, Spain, Italy and Portugal. The formulations used included 5 and 10 GR, 400 EC, 250 EW and 240 CS. The results are shown in Table 73. The trials by Heinemann and Ohs and by Blass (below) were conducted and reported in accordance with GLP. For the other trials, where only data sheets were provided, the relevant details are given as footnotes to the Table.

Glasshouse trials on tomatoes were conducted in Italy, Portugal and Spain (Heinemann and Ohs, 1997d). Namacur 240 CS (capsule suspension) was applied by drip irrigation at a rate equivalent to 10 kg ai/ha at pre-flowering (inflorescence emergence) and fruit development. Fruit were sampled 60 or 61 days after treatment. The plot sizes at the four sites were 34 to 530 m² and the soils were sandy with the pH at two sites 5.7 and 7.7, 0.4 and 0.5% C. In trials 602787 and 603848 the soil surface was covered with black foil; in trial 603864 the upper parts of the plant were cut.

In subsequent glasshouse trials (Blass, 1998b) Namacur 240 CS was applied by drip irrigation at a rate equivalent to 10 kg ai/ha in three trials and at 9.4 in one trial owing to underdosing. The trials were in Italy, Portugal and Spain, with applications 21, 87, 76 and 44 days after planting in trials 700460, 705616, 705624 and 705632 respectively. Plot sizes were 8.5-525 m². The soils were typically sand, sandy loam or loamy sand, with pH 5.5-7.8 and 0.5-2.2% C.

Table 73. Residues in tomatoes from supervised trials in Australia, Brazil, Italy, Portugal, South Africa and Spain.

Location, year, (variety)	Application			PHI, days	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.			
Australia (QLD), 1971 (Grosse lisse)	5 GR	11.2 F	1	78	<0.05	Bayer Australia 1971f 11/71d ¹
Australia (QLD), 1971 (Grosse lisse)	400 EC	8.9 F	1	81	<0.05	Bayer Australia 1971g 11/71a ²
Australia (QLD), 1971 (Grosse lisse)	400 EC	11.2 F	1	81	<0.05	11/71 b ²
Australia (QLD), 1971 (Grosse lisse)	400 EC	13.4 F	1	81	<0.05	11/71c ²
Australia (SA), 1971 (Grosse lisse)	400 EC	8.7 F	1	127 161	0.15 <0.05	12/71a ³
Australia (SA), 1971 (Grosse lisse)	400 EC	8.7 F	1	127 161	<0.05 <0.05	12/71b ³
Brazil (Agrocica), 1985	10 GR	2 F	1	124	<0.1	Fundacao C. & T 1985 BRA-78900-85A ⁴
Brazil (Sao Paulo), 1989 (Santa Cruz-Okada)	10 GR	5 F	1	70 94	<0.1 <0.1	U. Sao Paulo 1989 BRA-LYPES89-1-A ⁵
Brazil (Sao Paulo), 1989 (Santa Cruz-Okada)	10 GR	10 F	1	94	<0.1	BRA-LYPES89-1-B ⁵
South Africa, 1976	10 GR	10 F	1	58 73 88	<0.05 <0.05 <0.05	S. Afr. Bur. Stds 1977 311/880/P163 ⁶
South Africa, 1976	400 EC	10 F	1	58 73 88	<0.05 <0.05 <0.05	
South Africa, 1984 (Hibberdene)	10 GR 10 GR + 400 EC 400 EC 250 EW	1 g ai /m, 30 cm band F 0.5 g/m 30 cm or 40 cm band 1 g ai/m, 30 cm band 1 g ai/m, 30 cm band	1 1 + 1 1 1	86 99 112 35 48 61 86 99 112 86 99 112	0.30 0.14 0.10 0.36 0.25 0.16 0.17 0.14 0.06 0.12 0.09 0.06	S. Afr. Bur. Stds 1985 311/88694/B40 ⁷
	400 EC 400 EC	1 g ai/m 30 cm band + 0.5 g ai/m 40 cm band 1 g ai/m 30 cm band + 0.5 g ai/m	1 + 1 1 + 1*	35 48 61 0 7 13 20 33	0.15 0.07 0.11 <0.05 0.10 <0.05 <0.05 <0.05	
Spain (Alicante), 1984 (Restino)	400 EC	10 G	1	1 15 29 62	<0.05, <0.05 0.33, 0.41 0.33, 0.37 0.19, 0.27	Bayer 1985c 5206-84 ⁸
Spain (San Javier), 1986 (Carmelo)	400 EC	5 G	2	55 84 112	0.1, 0.13 <0.01, 0.01 <0.01, <0.01	Bayer 1986b 5202-86 ⁹
Spain (Moreno), 1988 (A-7)	400 EC	10 F	1	60	<0.02, <0.02	Bayer 1988b 0077-88 ¹⁰
Spain (St. Boi de Llobregat, (1988) (A-7)	10 GR	10 F	1	60	<0.02, <0.02	Bayer 1989a 0075-88 ¹¹ F
Spain (Sr. Jordana), 1988 (Carmelo)	10 GR	10 F	1	66	<0.02, <0.02	0076-88 ¹²
Spain (El Masnou), 1988 (Carmelo)	400 EC	10 F	1	66	<0.02, <0.02	Bayer 1989b 0078-88 ¹³

Location, year, (variety)	Application			PHI, days	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.			
Italy (Latina), 1996 (Sidonia)	240 CS	10 G	1	30 60 90	0.381, 0.368 0.02, 0.02 <0.02	Heinemann & Ohs 1997d 0278-96 ¹⁴
Italy (Sicily), 1996 (Felicia)	240 CS	10 G	1	31 60 90	0.081, 0.070 <0.02, <0.02 <0.02	0384-96 ¹⁴
Portugal (Lissabon), 1996 (Indalo)	240 CS	10 G	1	50 61 70	<0.02, <0.02 <0.02, <0.02 <0.02	0385-96 ¹⁴
Spain (Almeria), 1996 (Garbo)	240 CS	10 G	1	32 60 90	0.066, 0.070 0.092, 0.080 0.042	0386-96 ¹⁴
Italy (Latina), 1997 (Arletta)	240 CS	10 G	1	30 60 90	<0.02, <0.02 <0.02, <0.02 <0.02	Blass 1998b 0046-97 ¹⁵
Italy (Ragusa), 1997 (Cencara)	240 CS	10 G	1	30 60 90	0.079, 0.081 <0.02, <0.02 <0.02	0561-97 ¹⁵
Spain (Barcelona), 1997 (Alboran)	240 CS	9.4 G	1	31 60 90	0.205, 0.167 0.142, 0.144 0.032	0562-97 ¹⁵
Portugal (Lissabon), 1997 (Indalo)	240 CS	10 G	1	30 60 90	<0.02, <0.02 <0.02, <0.02 <0.02	0563-97 ¹⁵
GAP						
Australia	100 G	11	1	NS		
	400 EC	9.6	1	NS		
Brazil	10 GR	3-4	1	90		
Italy	5 GR	10-15	1	20		
Portugal	10 GR	3.4	1	90		
	400 EC	3.2	1	90		
South Africa	10 GR	1 g ai/m	1	NS		
	400 EC	1 g ai/m	1	NS		
Spain	10 GR	5-10	1	90		
	400 EC	5-10	1	90		

F: field

G: glasshouse

* Second treatment applied 1 week before harvest.

¹ Scattered by hand to soil 3 days after transplanting; left on surface. Limit of detection = 0.05 mg/kg; recovery at 1 mg/kg = 72%, recovery at 0.5 mg/kg = 69%.² Applied by boom to soil 1 day before transplanting and rotary hoed in. Limit of detection = 0.05 mg/kg; recovery at 1 mg/kg = 72%, recovery at 0.5 mg/kg = 69%.³ Applied by boom spray 21 days before transplanting. Limit of detection = 0.05 mg/kg; recovery at 1 mg/kg = 72%, recovery at 0.5 mg/kg = 69%. Hot dry weather, little rainfall, regular irrigation.⁴ Applied at planting by spreading; no recovery data given.⁵ Field trial with spreading at transplanting. Plot size = 12 m², clay soil, pH 5.7 % C = 1.8. Limit of determination = 0.1 mg/kg, recovery at 0.1 mg/kg 83-87%.⁶ No field details given; limit of detection = 0.05 mg/kg. Recoveries for fenamiphos sulfone and sulfoxide conducted at 1 mg/kg = 98% fenamiphos sulfone and 92% for fenamiphos sulfoxide.⁷ No field details given; limit of detection = 0.05 mg/kg. Mean recoveries at 0.1 mg/kg = 104% for fenamiphos, 94% fenamiphos sulfone and 96% fenamiphos sulfoxide. All results corrected for recovery.⁸ Glasshouse trial. Applied by drip irrigation at fruit development stage. Plot size = 55 m² Loamy clay soil pH 7.5, % C = 0.53. Recovery = 100% at 0.1 mg/kg.⁹ Glasshouse trial. Product applied 28 and 42 days after planting; last application at fruit development. Trial plot 30 m². Loamy sand soil pH 7.5-8, % C = 0.53. Limit of determination = 0.01 mg/kg, recovery at 0.01 mg/kg = 101%, at 0.1 mg/kg = 96%.¹⁰ Field trial. Applied by drip irrigation at 31 days after planting, flowering stage. Limit of determination = 0.02 mg/kg, recovery at 0.02 mg/kg = 91%. Plot size 12.8 m², sandy soil pH 6.5.¹¹ Field trial. Applied by spreading at 30 days after transplanting at flowering stage. Plot size = 12.8 m², sandy soil type pH 6.5. Limit of determination = 0.02 mg/kg, recovery at 0.02 mg/kg = 91%.¹² Field trial. Single application by spreading at 6 days after transplanting. Plot size = 45.5 m², sandy soil pH 7.5 % C = 2. Limit of determination = 0.02 mg/kg, recovery at 0.02 mg/kg = 91%.

¹³ Field trial. Single application by drip irrigation 6 days after planting. Plot size = 53.8 m², sandy soil pH 7.5, 2% C. Limit of determination = 0.02 mg/kg, recovery at 0.02 mg/kg = 91%.

¹⁴ Glasshouse trials. Single application by drip irrigation 6 to 49 days after planting. Limit of determination = 0.02 mg/kg, recovery at 0.02 mg/kg = 87, 88%, recovery at 0.1 mg/kg 72-102% (n = 6), recovery at 1 mg/kg = 73, 79%.

¹⁵ Glasshouse trials. Single application by drip irrigation at 21-87 days after planting. Limit of determination = 0.02 mg/kg, recovery at 0.02 mg/kg = 95, 97%, recovery at 0.1 mg/kg 75-90% (n = 7).

and those with the CS formulation ranged from <0.02 mg/kg to 0.3 mg/kg, predominantly from field trials. In all trials except the South African trials and one CS glasshouse trial (705624), fenamiphos residues were below the limit of determination. Residues of 0.06 to 0.36 mg/kg were found in some of the trials in South Africa at various harvest intervals.

Zucchini. In a glasshouse trial in Italy Nemacur 240 CS (capsule suspension) was applied by drip irrigation to plants at the 3 to 5 leaf stage. The product was applied at a rate equivalent to 10 kg ai/ha directly to the base of each plant. Drip irrigation was used to water the plants. Plot sizes were 40 to 79 m². The soils in all the glasshouses were sandy, with a pH range of 5.7 to 8.1. In trials R602795 and R603988 the soil was covered with a black material. The results are shown in Table 74. Recoveries from zucchini by method 00024/I8 were reported in the modification M003 to M002 (Blass, 1997a,b) described above.

Table 74. Residues in zucchini from trials in Italy with Nemacur 240 CS

Location, year, (variety)	Application			PHI, days	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.			
Latina, 1996 (President)	240 CS	10	1	30	0.037, 0.034	Heinemann & Ohs 1997e 0279-96 ¹
				60	<0.02, <0.02	
				90	<0.02	
Ragusa, 1996 (Stor-green)	240 CS	10	1	30	<0.02, <0.02	0396-96 ¹
				60	<0.02, <0.02	
				90	<0.02	
Latina, 1996 (President)	240 CS	10	1	30	<0.02, <0.02	0398-96 ¹
				60	<0.02, <0.02	
				90	<0.02	
Ragusa, 1996 (Stor-green)	240 CS	10	1	30	0.107, 0.090	0399-96 ¹
				60	<0.02, <0.02	
				90	<0.02	
GAP Spain (cucurbits)	10 GR 400 EC	10 10	1 1	90 90		

¹ Limit of determination = 0.02 mg/kg; recovery at 0.02 mg/kg 85-110% (n = 3), at 0.1 mg/kg 75-112% (n = 6), at 1 mg/kg = 85, 87%.

Fenamiphos was found at levels above the limit of determination at 30 days in two trials (602795 and 603996). This was explained as being due to cold temperatures in the glasshouses in trials 603996 and 603961, resulting in retarded fruit growth and smaller fruits. This may be a factor but the average unit weights in 603961 were lower than those in 603996 and the residues were below the LOD. Black cloth covered the soil surface in trial 602795 which may have contributed to the finite residues at day 30.

Melons. Supervised trials in Australia, Brazil, Guatemala, Mexico and Italy were reported. The results are shown in Table 75.

Four trials were conducted at each of two sites, Coahuila and Durango, in Mexico. Nemacur 15 GR was applied to the soil in-furrow at planting at 3 kg ai/ha (Leslie, 1988a). Fruit were collected 62 or 64 days after planting and residues were determined in the pulp and peel and expressed on a whole fruit basis. Plots were 200 m², predominantly composed of clay or sandy soils with pH 6.5-7.5. Residues in the whole fruit were below the limit of determination of 0.05 mg/kg.

In glasshouse trials on melons in Italy (Heinemann and Ohs, 1997f) at four sites, NemaCur 240 CS was applied at a rate of 10 kg ai/ha by drip irrigation 20-36 days after planting. Plots were 40-61 m² and were composed of sand or sandy loam soils at pH 5.7-8. At all sites the soil was covered by black foil. Samples were taken at 50, 59/60 and 90 days after treatment. Residues were determined in the whole fruit and pulp.

In subsequent glasshouse trials in Italy (Blass, 1998c) NemaCur 240 CS was applied to five test sites at times from 1 to 65 days after planting. The growth stages ranged from four-leaf to flowering. The product was applied by drip irrigation at a rate equivalent to 10 kg ai/ha. Plot sizes were 66 to 82 m² and soils were typically clay sand or sandy loam, pH 7.5 to 8.5, and 0.6-1.7% C. In trials 700452, 705543 and 705551 the soil was covered with black foil. Samples of whole fruit were collected 50, 60/61 and 67/70 days after treatment; pulp was analysed in addition to whole fruit.

Table 75. Residues of fenamiphos in melons from trials in Australia, Guatemala, Mexico and Italy.

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.
	Form.	kg ai/ha	No.				
Guatemala (Zacapa), 1987 (Mayan sweet)	10 GR	10	1	85	fruit	<0.05	Bayer 1987d GUA-36-87-A ¹ F
Guatemala (Zacapa), 1987 (Mayan sweet)	10 GR	10	1	71	fruit	<0.05	Bayer 1987d GUA-36-87-B ¹ F
Guatemala (Zacapa), 1987 (Mayan sweet)	10 GR	4	1	85	fruit	<0.05	Bayer 1987d GUA-36-87-C ¹ F
Guatemala (Zacapa), 1987 (Mayan sweet)	10 GR	4	1	71	fruit	<0.05	Bayer 1987d GUA-36-87-D ¹ F
Mexico (Durango), 1983 (Sierra gold)	15 GR	3	1	64	pulp peel whole fruit	≤0.01 <0.01 ≤0.01	Leslie 1988a 96784
Mexico (Durango), 1983 (Sierra gold)	15 GR	3	1	64	pulp peel whole fruit	≤0.01 <0.01 ≤0.01	
Mexico (Coahuila), 1983 (Imperial 45)	15 GR	3	1	62	pulp peel whole fruit	≤0.01 <0.01 ≤0.01	
Mexico (Coahuila), 1983 (Imperial 45)	15 GR	3	1	63	pulp peel whole fruit	≤0.01 0.02 ≤0.01	
Italy (Porgo Piave), 1989 (Charantes)	5 GR	15	1	85 100 105	pulp peel whole fruit pulp peel whole fruit pulp peel whole fruit	≤0.02 <0.02 ≤0.02 <0.02 <0.02 <0.02 ≤0.02	Bayer 1990a 0064-89 ³ F
Australia (QLD), 1971 (Hales best)	400 EC	8.9	1	112	mature fruit	<0.01	Bayer Australia 1971h 33/71a ⁴ F
Australia (QLD), 1971 (Hales best)	400 EC	8.9	1	77	mature fruit	<0.01	33/71b ⁵ F
Brazil (Sao Paulo), 1995 (Valenciano)	400 EC	4	1	90	whole fruit	<0.02	U. Sao Paulo 1996 BRA-2009-96-A ⁶ F
Brazil (Sao Paulo), 1995 (Valenciano)	400 EC	8	1	90	whole fruit	<0.02	BRA-2009-96-B ⁶ F
Italy (Latina), 1996, (Proteo)	240 CS	10	1	50 60 90	whole fruit	0.041, 0.042 <0.02, <0.02 <0.02	Heinemann and Ohs 1997f 0281-96 ⁷ G

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.
	Form.	kg ai/ha	No.				
				50 60 90	pulp	<0.02, <0.02 <0.02, <0.02 <0.02	
Italy (Ravenna), 1996, (Drake)	240 CS	10	1	50 60 90 50 60 90	whole fruit pulp	0.034, 0.036 0.02, 0.021 <0.02 0.022, 0.021 <0.02, <0.02 <0.02	0376-96 ⁷ G
Italy (Latina), 1996, (Mambo)	240 CS	10	1	50 60 90 50 60 90	whole fruit pulp	<0.02, <0.02 <0.02, <0.02 <0.02, <0.02 <0.02, <0.02 <0.02	0377-96 ⁷ G
Italy (Verona), 1996, (Golden Star)	240 CS	10	1	50 59 80 90 50	whole fruit pulp	0.046, 0.044 0.025, 0.028 <0.02 <0.02 0.021, 0.025	0378-96 ⁷ G
				59 80 90		<0.02, <0.02 <0.02 <0.02	
Italy (Verona Sth), 1997, (Super market)	240 CS	10	1	50 60 70 50 60 70	whole fruit pulp	0.03 <0.02 <0.02 0.03 <0.02 <0.02	Blass 1998c 0045-97 ⁸ G
Italy (Verona Sth), 1997, (Super market)	240 CS	10	1	50 61 70 50 61 70	whole fruit pulp	<0.02 <0.02 <0.02 <0.02 <0.02 <0.02	0554-97 ⁸ G
Italy (Ravenna), 1997, (Drake)	240 CS	10	1	60 70 60 70	whole fruit pulp	<0.02 <0.02 <0.02 <0.02	0555-97 ⁸ G
Italy (Ravenna), 1997, (Crido)	240 CS	10	1	50 60 67 50 60 67	whole fruit pulp	<0.02 <0.02 <0.02 <0.02 <0.02 <0.02	0557-97 ⁸ G
Italy (Ravenna Nth), 1997	240 CS	10	1	50 60 70	whole fruit	0.03 <0.02 <0.02	0801-97 ⁸ G

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.
	Form.	kg ai/ha	No.				
				50 60 70	pulp	0.03 <0.02 <0.02	
GAP							
Australia	400 liq.	9.6	1	NS			
Brazil	400 EC	4	1	NS			
Guatemala	10 GR	2.5-5	1	60			
	12 GR	0.72- 1.2	1	60			
	15 GR	2.5- 5.1	1	60			
Italy	5 GR	10-15	1	20			
Spain	10 GR	10	1	90			
	400 EC	10	1	90			

F: field G: glasshouse

¹ Field trial. Single application by spreading and incorporation at sowing or 14 days before sowing. Plot size 552 m². Soil type described as "Franco-arenoso" pH 6.7, 1.85% C. Limit of determination = 0.05 mg/kg, recovery at 0.05 mg/kg = 68%.

² Field trials. Recovery at 0.05 mg/kg = 102, 88, 76% from pulp, 88, 88, 90% from peel for F, FSO and FSO₂ respectively. Recoveries of fenamiphos were 89 and 109% at 0.1 and 0.5 mg/kg from pulp and 87% at 0.5 mg/kg from peel. Limit of determination = 0.05 mg/kg. All chromatograms provided.

³ Field trial. Single application at 18 days before planting by spreading. Plot size = 100 m², sandy soil pH 7, 1.53% C. Limit of determination = 0.02 mg/kg, recovery from pulp = 88% at 0.02 mg/kg.

⁴ Applied by boom spray and incorporated with rotary hoe 35 days before sowing. Limit of detection = 0.01 mg/kg, recovery at 0.1 and 0.5 mg/kg = 73% and 78% respectively.

⁵ Applied by boom spray and incorporated with rotary hoe 4 days before sowing. Limit of detection = 0.01 mg/kg, recovery at 0.1 and 0.5 mg/kg = 73% and 78% respectively.

⁶ Field trial. Applied by spraying at planting. Loamy sand soil pH 5.9, 1.3% C. Limit of determination = 0.02 mg/kg, recovery at 0.02 mg/kg 86-98%.

⁷ Glasshouse trial. Limit of determination = 0.02 mg/kg, recovery of fenamiphos at 0.02 mg/kg 82-93% (n = 6), at 0.1 mg/kg 75-91% (n = 14), at 1 mg/kg 78-89% (n = 6).

⁸ Glasshouse trial. Limit of determination = 0.02 mg/kg. Recovery from whole fruit = 92% at 0.02 mg/kg, 76-92% at 0.1 mg/kg (n = 5). Recovery from pulp = 88% at 0.02 mg/kg, 72-92% at 0.1 mg/kg (n = 5).

There were 13 field and 9 glasshouse trials. The residues in the trials according to GAP in Australia, Brazil and Guatemala were all below the limit of determination, both in pulp and whole fruit. In the trials in Mexico according to GAP in Guatemala, fenamiphos residues in the pulp and whole fruit were all below the limit of determination. The underlined residues were used in the estimation of the maximum residue levels and the double-underlined residues in the estimation of the STMR for the edible portion.

After treatment with the CS product the residues in the whole fruit were below the limit of determination in all but one trial (603783) where levels of 0.025 and 0.028 mg/kg were found in whole fruit, although residues in the pulp were <0.02 mg/kg.

Watermelons. Results from trials in Italy were in summary form. Fenamiphos residues were determined in the pulp, peel and whole fruit. The results are shown in Table 76. Recoveries from watermelons were reported in supplement E022 (Ohs, 1988b) to method 00024/I8 as 93% at 0.02 mg/kg and 87% at 0.1 mg/kg.

Table 76. Residues in supervised trials on watermelons in Italy.

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
Latina, 1988, (Crimson Sweet)	5 GR	10	1	99	fruit	<0.02, <0.02	Bayer 1989c 0197-88 ¹ F
				109		<0.02, <0.02	
Borgo Piave, 1989, (Crimson Sweet)	5 GR	15	1	85	pulp	<0.02, <0.02	Bayer 1990b 0062-89 ² F
				100		<0.02, <0.02	
				105	<0.02, <0.02		
				85	<0.02, <0.02		
					peel		

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
				100 105 85 100 105	whole fruit	<0.02, <0.02 <0.02, <0.02 <u><0.02</u> <0.02 <0.02	
GAP Italy (melon) Spain	5 GR 10 GR	10-15 5-10	1 1	20 90			

¹ Single application by spreading 20 days before planting. Plot size = 100 m², sandy soil, pH 7, 1.53% C. Limit of determination = 0.02 mg/kg, recovery at 0.02 mg/kg = 93%.

² Single application by spreading 2 days before planting. Plot size = 200 m², sandy soil, pH 7, 1.53% C. Limit of determination = 0.02 mg/kg, recovery at 0.02 mg/kg from pulp and peel = 88% (n = 3).

Although the Italian label provided does not specifically refer to use on watermelon, the use pattern on melons was taken as relevant GAP. The trials in Italy were also compares with registered uses in Spain. After a single application of fenamiphos at 10 or 15 kg ai/ha, residues in the whole fruit, pulp and peel were below the limit of determination 85 days after treatment.

Citrus fruit

Grapefruit. Data from trials in the USA are shown in Table 77. The results were in the form of summary sheets with chromatograms. In early trials (1972), residues were determined in the whole fruit and in later work (1981) the residues were determined in the pulp and peel and in the whole fruit. Recoveries at a fortification level of 0.1 mg/kg were reported in Table 61.

Table 77. Residues from supervised trials on grapefruit in the USA.

Year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
Arizona, 1972	15 GR	33.6	1	184	fruit	<0.01	Chemagro 1972a 33085 ¹
Texas, 1972	15 GR	33.6	1	183	peel pulp	<0.01 <0.01	33086 ¹
Florida, 1972	15 GR	33.6	1	184	fruit	<0.01	33147 ¹
Arizona, 1972	3 SC	33.6	1	184	fruit	<0.01	Chemagro 1972b 33075 ²
Florida, 1972	3 SC	33.6	1	186	fruit	0.5, 0.56	33082 ²
California, 1981 (Marsh)	15 GR	33.6	1	30 60 126 182 30 60 126 182 30 60 126 182	peel pulp whole fruit	0.02 0.05 0.06 0.02 <0.01 <0.01 <0.01 <0.01 <0.01 0.02 0.02 <0.01	Mobay 1981a 69914 ³
Texas, 1981, (Ruby Red)	15 GR	33.6	1	30 59 124 169 30 59 124	peel pulp	0.01 0.01 0.02 0.04 <0.01 <0.01 <0.01	69915 ⁴
				169 30 59 124 169	whole fruit	<0.01 <0.01 <0.01 <0.01 0.01	

Year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
Florida, 1981, (Marsh Seedless)	15 GR	33.6	1	30	peel	0.24	69916 ⁵
				61		0.43	
				122		0.73	
				184		0.80	
				243	pulp	0.25	
				30		0.03	
				61		0.05	
				122		0.08	
				184	whole fruit	0.03	
				243		<0.01	
				30		0.09	
				61		0.13	
				122		0.28	
184	0.29						
243	0.12						
California, 1981, (Marsh)	360 SC	33.6	1	30	peel	0.07	Mobay 1981b 69917 ⁶
				60		0.14	
				126		0.22	
				182	pulp	0.05	
				30		0.02	
				60		0.02	
				126	whole fruit	0.01	
				182		<0.01	
				30		0.04	
				60		0.06	
				126		0.09	
				182	0.02		
				Texas, 1981, (Ruby Red)	360 SC	33.6	
59	0.03						
124	0.03						
169	pulp	0.01					
30		0.01					
59		<0.01					
124	whole fruit	<0.01					
169		<0.01					
30		0.01					
59		0.02					
124		0.01					
169	<0.01						
Florida, 1981, (Marsh Seedless)	360 SC	33.6	1				30
				61	0.13		
				122	0.43		
				184	pulp	0.68	
				243		0.28	
				30		0.03	
				61	whole fruit	0.03	
				122		0.04	
				184		0.03	
				243		0.02	
				30		0.11	
				61	0.05		
				122	0.15		
184	0.26						
243	0.09						
GAP for citrus fruit	350 EC	5-8.4	1	30			

¹ Single broadcast application (within dripline).² Single application by soil drench (within dripline).³ Single application by surface broadcast; plot size = 144 m²; growth stage at application 50 mm fruit.⁴ Single broadcast application; plot size = 222.8 m²; growth stage at application ½-grown green fruit.⁵ Single surface broadcast application; plot size = 44 m²; growth stage at application ¼ to ½-grown green fruit.⁶ Single application by surface broadcast; plot size = 573.4 m²; growth size at application 50 mm fruit.⁷ Single application by surface broadcast; plot size = 222.8 m²; growth stage at application ½-grown green fruit.⁸ Single application by surface broadcast; plot size = 44 m²; growth stage at application ¼ to ½-grown green fruit.

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference or report no.
	Form.	kg ai/ha	No.				
South Africa (Nelspruit), 1989	10 GR	2.5 g ai/m ²	1	60	pulp	<0.05, 0.05	311/88812/G155 ⁷
				100		<0.05, <0.05	
				120		<0.05, <0.05	
				150		<0.05, <0.05	
				60	peel	0.08, 0.08	
				100		0.05, 0.05	
				120		<0.05, <0.05	
150	<0.05, <0.05						
GAP							
Australia	400 liq.	30	1	NS			
South Africa	10 GR	12 or 2 g ai/m²		150			
USA (for citrus fruit)	350 EC	5-8.4	1	30			

¹ Single application by soil drench (within dripline).

² Single application by soil broadcast, raked into soil within 1 day of application; growth stage at application: fruit set.

³ Single application by soil broadcast (within dripline), raked in within 1 day after application; growth stage at application: fruit set.

⁴ Limit of detection = 0.02 mg/kg, recovery at 0.1 mg/kg = 90%.

⁵ Single application by soil broadcast incorporation into the ground; growth stage at application mature fruit; sandy soil, pH 6.5-7.5, <1% C.

⁶ Single application by soil broadcast incorporation into the ground; growth stage at application mature fruit; sandy soil, pH 6.5-7.5, <1% C.

⁷ Single application by spreading by hand at flowering stage; area treated 62.5 m², sandy loam soil, pH 5.2, 1-2% C, Limit of detection = 0.05 mg/kg, recovery at 0.1 mg/kg = 98, 103, 65% of F, FSO and FSO₂ respectively from pulp, 76, 80 and 105% from peel.

The trials on lemons were also at 2.6 or 4 times the maximum registered rate in the USA. The residues were <0.01-0.44 mg/kg in whole fruit, <0.01-1.15 mg/kg in peel, and <0.01-0.05 mg/kg in pulp. Residues from trials according to GAP were <0.02 mg/kg in whole fruit in Australia and <0.05 mg/kg in pulp and peel in South Africa.

Limes. Limited data from US trials were provided. Again, excessive rates were used (Table 79).

Table 79. Residues from supervised trials on limes in Florida, USA.

	Application			PHI, days	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.			
1972 (Tahiti)	15 GR	33.6	1	147	<0.01	Chemagro 1972d 33087 ¹
1972 (Tahiti)	360 SC	33.6	1	147	<0.01	Chemagro 1972e 33088 ²
GAP for citrus fruit	350 EC	1.6-8.2	1	30		

¹ Single application by broadcast (within dripline). Chromatograms provided.

² Single application by soil drench (within dripline). Chromatograms provided.

Oranges. Supervised trials were conducted in Australia, South Africa and the USA. All data were provided in summary form and relevant field details are given as footnotes to Table 80. Recoveries from whole fruit, pulp, peel, leaves, oil and molasses at fortification levels of 0.02-1 mg/kg were reported in Table 61.

Table 80. Residues from trials on oranges in Australia, South Africa and the USA.

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
USA (California), 1970, (Navel)	10 GR	22.4	1	366	peel	<0.01, 0.02	Chemagro 1970a 27442 ¹
					pulp	<0.01, <0.01	
USA (California), 1972, (Navel)	15 GR	33.6	1	182	fruit	<0.01	Chemagro 1972f 33148 ²
USA (Arizona), 1972, (Navel)	360 SC	33.6	1	184	fruit	<0.01	Chemagro 1972g 33077 ³

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
USA (California), 1972, (Navel)	360 SC	33.6	1	182	peel pulp	<0.01 <0.01	33080 ³
USA (Florida), 1986, (Valencia)	15 GR	22.4	1	31	pulp	<0.01	Mobay 1986d 91352 ⁴
				60		<0.01	
				119		<0.01	
				181		<0.01	
				213		<0.01	
				242		<0.01	
				273		<0.01	
				304		<0.01	
				364		<0.01	
				31	peel	<0.01	
				60		<0.01	
				119		0.03	
				181		0.07	
				213		<0.01	
				242		0.04	
				273		0.01	
				304		0.03	
				364		0.02	
31	whole fruit	<0.01					
60		<0.01					
119		0.02					
181		<0.01					
213		0.01					
242		<0.01					
273		<0.01					
304		<0.01					
364		<0.01					
USA (Texas), 1986, (Hamlin)	15GR	22.4	1	30	pulp	0.02	91354 ⁵
				63		<0.01	
				123		<0.01	
				189		<0.01	
				219		<0.01	
				252		<0.01	
				272		<0.01	
				305		<0.01	
				378		<0.01	
				30	peel	<0.01	
				63		0.71	
				123		0.03	
				189		0.06	
				219		0.07	
				252		0.03	
				272		<0.01	
				305		<0.01	
				378		<0.01	
30	whole fruit	<0.01					
63		0.17					
123		<0.01					
189		0.01					
219		0.02					
252		0.01					
272		<0.01					
305		<0.01					
378		<0.01					
USA (California), 1986, (Valencia)	15 GR	22.4	1	21	pulp	<0.01	91356 ⁶
				62		<0.01	
				112		<0.01	
				151		<0.01	
				232		<0.01	
				300		<0.01	
332	<0.01						

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
				21 62 112 151 232 300 332	peel	0.01 <0.01 0.02 0.01 0.02 0.13 0.01	
				21 62 112 151 232 300 332	whole fruit	<0.01 <0.01 <0.01 <0.01 <0.01 0.03 <0.01	
USA (Florida), 1986, (Valencia)	360 SC	22.4	1	31 60 119 180 212 241 273 304 364 31 60 119 180 212	pulp peel	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 0.02 0.02 0.03	Mobay 1986e 91351 ⁷
				241 273 304 364 31 60 119 180 212 241 273 304 364	whole fruit	0.02 0.02 0.02 0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	
USA (Texas), 1986, (Valencia)	360 SC	22.4	1	30 63 123 189 219 252 272 305 378 30 63 123 189 219 252 272 305 378 30 63 123 189	pulp peel whole fruit	<0.01 0.02 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 0.10 0.38 0.02 0.04 0.03 0.01 <0.01 <0.01 <0.01 0.02 0.09 <0.01 <0.01	91353 ⁸

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
				219 252 272 305 378		<0.01 <0.01 <0.01 <0.01 <0.01	
USA (California), 1986, (Valencia)	360 SC	22.4	1	20 62 112 151 232 300 20 62 112 151 232 300 20 62 112 151 232 300	pulp peel whole fruit	<0.01 <0.01 0.01 <0.01 <0.01 <0.01 0.01 <0.01 0.06 0.05 0.06 0.02 <0.01 <0.01 0.02 0.01 0.02 <0.01	91355 ⁹
South Africa (Letsitele), 1976	10 GR	5 g ai/m ²	1 2	123 188 75 123 188	fruit	0.08, <u>0.08</u> 0.07, 0.08 <0.02, <0.02 <u>0.02, 0.03</u> 0.03, 0.03	S. Afr. Bur. Stds. 1976 0311/8947/N333 ¹⁰
Australia 1980, (Navel)	436 g/l	40	1	8	fruit	<u><0.02</u>	Bayer Australia 1980b 20/80 ¹¹
GAP Australia South Africa	400 liq. 10 GR	30 12 (4 g ai/m²)	1	NS 150			
USA (for citrus fruit)	400 EC 350 EC	12 (4 g ai/m²) 1.6-8.2	1-2	30			

¹ Single application by soil broadcast.

² Single application by broadcast (within dripline).

³ Single application by soil drench (within dripline).

⁴ Limit of detection = 0.02 mg/kg, recovery at 2 and 10 mg/kg = 89 and 91% respectively 92 days between 1st and 2nd applications.

⁵ Limit of detection = 0.02 mg/kg.

⁶ Single application by broadcast and incorporation into soil at immature to maturing fruit stage; plot size 74.3 m², sandy soil pH 5.5-6.4, <1% C. Limit of detection = 0.01 mg/kg.

⁷ Single application at dormant or blooming or 2.5 or 5 cm diameter fruit; plot size 60 m², sandy loam pH 6.5-7.5, 1-2% C. Limit of detection = 0.01 mg/kg.

⁸ Single broadcast application and incorporation into soil at mature fruit stage; plot size 0.07 ha, loamy sand soil pH >7.5, <1% C. Limit of detection = 0.01 mg/kg.

⁹ Single application by broadcast with incorporation into soil at immature fruit to mature fruit stages; sandy soil, plot size = 74.3 m², pH 5.5-6.5, <1% C. Limit of detection = 0.01 mg/kg.

¹⁰ Single application by broadcast and incorporation into soil at dormant or blooming or 2.5 or 5 cm diameter fruit; plot size 60 m², sandy loam pH 6.5-7.5, 1-2% C. Limit of detection = 0.01 mg/kg.

¹¹ Single application at mature fruit stage by broadcast and incorporation into soil; plot size 0.07 ha, loamy sand pH >7.5, <1% C. Limit of detection = 0.01 mg/kg.

The Australian and South African trials were according to GAP, and the residues were <0.02-0.08 mg/kg in the whole fruit. The US trials were at 2.6 or 4 times the maximum registered rate. The residues were <0.01-0.17 mg/kg in whole fruit, <0.01-0.02 mg/kg in pulp and <0.01-0.71 mg/kg in peel.

Apples. Numerous trials were conducted in the USA. All data were in summary form and the available details are included in footnotes to Table 81. Application timings ranged from pre-bloom to green fruit stages. Recoveries determined at fortification levels of 0.05 and 0.1 mg/kg are shown in Table 61.

Table 81. Residues in apples from trials in the USA.

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.
	Form.	kg ai/ha	No.				
Washington, 1978, (Winesap)	15 GR	22.4	1	114	pulp peel whole fruit	<u><0.01</u> <0.01 <0.01	Mobay, 1978a 65520 ¹
Michigan, 1978, (Golden Delicious)	15 GR	22.4	1	120 130 120 130 120 130	pulp peel whole fruit	<u><0.01</u> <0.01 <0.01 <0.01 <0.01 <0.01	66064 ²
Virginia, 1978, (Rome Beauty)	15 GR	22.4	1	119 141 119 141 119 141	peel pulp whole fruit	<0.01 <0.01 <u><0.01</u> <0.01 <u><0.01</u> <0.01	66074 ²
California, 1978, (Winesap)	360 SC	22.4	1	114 114 114	peel pulp whole fruit	<0.01 <u><0.01</u> <0.01	Mobay, 1978b 65619 ¹
New York, 1978, (Ida Red)	360 SC	22.4	1	121 151 121 151 121 151	peel pulp whole fruit	<0.01 <0.01 <u><0.01</u> <0.01 <u><0.01</u> <0.01	66065 ³
Pennsylvania, 1978, (Golden Delicious)	360 SC	22.4	1	120 130 120 130 120 130	peel pulp whole fruit	<0.01 <0.01 <u><0.01</u> <0.01 <u><0.01</u> <0.01	66075 ⁴
Virginia, 1978, (Rome Beauty)	360 SC	22.4	1	119 141 119 141 119 141	peel pulp whole fruit	<0.01 <0.01 <u><0.01</u> <0.01 <u><0.01</u> <0.01	66092 ⁴
New York, 1982, (Lodi)	15 GR	22.4	1	102	whole fruit	<u><0.01</u>	Mobay 1982a 80769 ⁵
Pennsylvania, 1982, (Lodi)	15 GR	22.4	1	79	whole fruit	<0.01	80770 ⁵
West Virginia, 1982, (Rambo)	15 GR	22.4	1	98	whole fruit	<0.01	80771 ⁵
Virginia, 1982, (Yellow Transparent)	15 GR	22.4	1	72	whole fruit	<0.01	80772 ⁵
California, 1982, (Golden Delicious)	15 GR	22.4	1	143	whole fruit	<0.01	80773 ⁶
Washington, 1982, (Gravenstein)	15 GR	22.4	1	125	whole fruit	<0.01	80774 ⁷
Michigan, 1982, (Jersey Mac)	15 GR	22.4	1	107	whole fruit	<0.01	80775 ⁶
New York, 1982, (McIntosh)	15 GR	22.4	1	162	whole fruit	<0.01	80776 ⁸
West Virginia, 1982, (Rome Beauty)	15 GR	22.4	1	176	whole fruit	<0.01	80777 ⁹
Virginia, 1982, (Golden Delicious)	15 GR	22.4	1	155	whole fruit	<0.01	80778 ¹⁰

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.
	Form.	kg ai/ha	No.				
California, 1982, (Red Delicious)	15 GR	22.4	1	143	whole fruit	<0.01	80779 ¹¹
Washington, 1982, (Golden Delicious)	15 GR	22.4	1	167	whole fruit	<0.01	80780 ⁷
Michigan, 1982, (Jonathan)	15 GR	22.4	1	154	whole fruit	<0.01	80781 ⁶
New York, 1982, (Lodi)	360 SC	22.4	1	102	whole fruit	<0.01	Mobay, 1982b 80756 ¹²
Pennsylvania, 1982, (Lodi)	360 SC	22.4	1	79	whole fruit	<0.01	80757 ¹⁰
West Virginia, 1982, (Rambo)	360 SC	22.4	1	98	whole fruit	<0.01	80758 ⁹
Virginia, 1982, (Yellow Transparent)	360 SC	22.4	1	72	whole fruit	<0.01	80759 ¹⁰
California, 1982, (Golden Delicious)	360 SC	22.4	1	143	whole fruit	<0.01	80760 ⁶
Washington, 1982, (Gravenstein)	360 SC	22.4	1	125	whole fruit	<0.01	80761 ⁷
Michigan, 1982, (Jersey Mac)	360 SC	22.4	1	107	whole fruit	<0.01	80762 ¹³
New York, 1982, (McIntosh)	360 SC	22.4	1	162	whole fruit	<0.01	80763 ⁸
West Virginia, 1982, (Rome Beauty)	360 SC	22.4	1	176	whole fruit	<0.01	80764 ⁹
Virginia, 1982, (Golden Delicious)	360 SC	22.4	1	155	whole fruit	<0.01	80765 ¹⁰
California, 1982, (Red Delicious)	360 SC	22.4	1	143	whole fruit	<0.01	80766 ⁶
Washington, 1982, (Golden Delicious)	360 SC	22.4	1	167	whole fruit	<0.01	80767 ⁷
Michigan, 1982, (Jonathan)	360 SC	22.4	1	154	whole fruit	<0.01	80768 ¹³
GAP USA	350 EC	5.4-8.2		72			

¹ Single application by soil broadcast and incorporation at 3.8-5 cm diameter fruit. No chromatograms.

² Single application by soil broadcast at green fruit stage. No chromatograms.

³ Single application by soil broadcast (within dripline) at pink fruit stage. No chromatograms.

⁴ Single application by soil broadcast and incorporation (within dripline) at green fruit stage. No chromatograms.

⁵ Single application by soil broadcast at pre-bloom stage.

⁶ Single application by broadcast at pink bud stage, plot size 20 m².

⁷ Single application by broadcast at pink bud stage, plot size 30 m².

⁸ Single application by broadcast at pre-bloom stage; plot size 38.6 m².

⁹ Single application by broadcast at pre-bloom stage; plot size 58 m².

¹⁰ Single application by broadcast at pre-bloom stage; plot size 37m².

¹¹ Single application by broadcast at pink bud stage; plot size 21 m².

¹² Single application by broadcast at pre-bloom stage; plot size 12.5 m².

¹³ Single application by broadcast at pink bud stage; plot size 52 m².

The application rates were 2.6 times the maximum registered rate in the USA, but residues in the whole fruit, pulp and peel were below the limit of detection in all the trials. Samples were collected at normal harvest times, so all are considered to be within the GAP PHI.

Cherries. Results of trials in the USA are shown in Table 82. In most cases, single applications were made at flowering or at the immature fruit stages of growth and samples were taken at intervals ranging from 31 to 98 days after application. Fenamiphos residues were <0.01 to 0.18 mg/kg. Recoveries from cherries were determined at fortification levels of 0.01-0.1 mg/kg and are shown in Table 61. Application rates were 2.6 times the maximum rate specified on registered US labels in all but three of the trials, where they were 1.7 times the maximum.

Table 82. Residues in cherries from trials in the USA.

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
Michigan, 1978, (Montmorency)	15 GR	22.4	1	31	fruit	0.05	Mobay 1978c 66072 ¹
Michigan, 1978, (Montmorency)	360 SC	22.4	1	31	fruit	0.01	Mobay 1978d 66418 ¹
California, 1982, (Pollinator)	15 GR	22.4	1	45	fruit	<0.01	Mobay 1982c 80424 ²
California, 1982, (Bing)	15 GR	22.4	1	52	fruit	0.03	80425 ²
Washington, 1982, (Chinook)	15 GR	22.4	1	82	fruit	<0.01	80426 ³
Oregon, 1982, (Chinook)	15 GR	22.4	1	76	fruit	<0.01	80427 ⁴
Michigan, 1982, (Napoleon)	15 GR	22.4	1	52	fruit	0.14, 0.18	80428 ⁵
Washington, 1982, (Bing)	15 GR	22.4	1	89	fruit	<0.01	80429 ³
Oregon, 1982, (Bing)	15 GR	22.4	1	83	fruit	<0.01	80430 ⁶
Washington, 1982, (Rainier)	15 GR	22.4	1	98	fruit	<0.01	80431 ³
California, 1982, (Early Pollinators)	360 SC	22.4	1	52	fruit	0.04, 0.04	Mobay 1982d 80415 ²
California, 1982, (Bing)	360 SC	22.4	1	52	fruit	0.02	80416 ²
Washington, 1982, (Chinook)	360 SC	8 + 14.3	2	75	fruit	<0.01	80417 ⁷
Oregon, 1982, (Chinook)	360 SC	22.4	1	76	fruit	<0.01	80418 ⁴
Michigan, 1982, (Napoleon)	360 SC	22.4	1	52	fruit	0.02, <0.01	80419 ⁵
Washington, 1982, (Bing)	360 SC	8 + 14.3	2	82	fruit	<0.01	80420 ⁷
Oregon, 1982, (Bing)	360 SC	22.4	1	83	fruit	<0.01	80421 ⁶
Washington, 1982, (Rainier)	360 SC	8 + 14.3	2	91	fruit	<0.01	80422 ⁸
Michigan, 1982, (Sweet Giant)	360 SC	22.4	1	66	fruit	0.01, 0.02	80423 ⁵
GAP USA	350 EC	5.4-8.2		45			

¹ Single application by soil broadcast (within dripline) at green fruit, pit hardening stage. Plot size one tree. No chromatogram.

² Single application by soil broadcast at mid-bloom; plot size one tree.

³ Single application by soil broadcast at bud appearance. Plot size one tree.

⁴ Single application by soil broadcast at 85% buds in white bud stage; plot size one tree.

⁵ Single application by soil broadcast at early bloom stage; plot size one tree.

⁶ Single application by soil broadcast at 95% buds in white bud stage; plot size one tree.

⁷ Two applications at 7 days interval by soil broadcast at early bloom and full bloom. Plot size one tree.

⁸ Two applications at 7 days interval by soil broadcast at 50% early bloom and 50% full bloom. Plot size one tree.

Peaches. The results of trials carried out in the USA and Italy are shown in Table 83. In most trials, a single application was made at growth stages ranging from pre-flowering to immature fruit and sampling was at intervals from 40 to 150 days after treatment. The residues in most of the trials were below the limit of detection or determination. Recoveries with fortification at 0.05 and 0.1 mg/kg are shown in Table 61.

Table 83. Residues in peaches from trials in the USA and Italy.

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
USA (Pennsylvania), 1978, (Red Skin)	15 GR	22.4	1	45 60 90	fruit	0.02 <0.01 <0.01	Mobay 1978e 66047 ¹
USA (New Jersey), 1978, (NJ 232)	15 GR	22.4	1	40 55 85	fruit	0.02 0.02 <0.01	66048 ²
USA (Pennsylvania), 1978, (Red Skin)	360 SC	22.4	1	45 60 90	fruit	0.02 <0.01 <0.01	Mobay 1978f 66049 ¹
USA (New Jersey), 1978, (NJ 232)	360 SC	22.4	1	40 55 85	fruit	0.01 0.02 <0.01	66050 ²
USA (Georgia), 1979, (Elberta)	15 GR	22.4	1	45 60	fruit	0.01 0.09	Mobay 1979a 67933 ³
USA (Sth Carolina), 1979, (Sunlight)	15 GR	22.4	1	73 131	fruit	0.16 <0.01	67934 ⁴
USA (New Jersey), 1982, (Garnet Beauty)	15 GR	22.4	1	93	fruit	<0.01	Mobay 1982e 80791 ⁵
USA (California), 1982, (Corona)	15 GR	22.4	1	118	fruit	<0.01	80792 ⁶
USA (California), 1982, (Starns)	15 GR	22.4	1	130	fruit	<0.01	80793 ⁶
USA (California), 1982, (Condor)	15 GR	22.4	1	91	fruit	0.04, 0.05	80794 ⁵
USA (Sth Carolina), 1982, (Condor)	15 GR	22.4	1	83	fruit	<0.01	80795 ⁷
USA (Michigan), 1982, (Sweet Haven)	15 GR	22.4	1	93	fruit	<0.01	80796 ⁸
USA (Georgia), 1982, (Winblo)	15 GR	22.4	1	106	fruit	0.02, 0.01	80797 ⁴
USA (Sth Carolina), 1982, (Rio-Oso-Gem)	15 GR	22.4	1	150	fruit	<0.01	80798 ⁷
USA (Sth Carolina), 1982, (Condor)	15 GR	22.4	1	83	fruit	0.10, 0.13, 0.11	80802 ⁷
USA (Sth Carolina), 1982, (Red Globe)	15 GR	22.4	1	122	fruit	0.01	80803 ⁷
USA (Sth Carolina), 1982, (Rio-Oso-Gem)	15 GR	22.4	1	150	fruit	<0.01	80804 ⁷
USA (New Jersey), 1982, (Garnet Beauty)	360 SC	22.4	1	93	fruit	<0.01	Mobay 1982f 80782 ⁵
USA (California), 1982, (Corona)	360 SC	22.4	1	118	fruit	<0.01	80783 ⁶
USA (California), 1982, (Starns)	360 SC	22.4	1	130	fruit	<0.01	80784 ⁶
USA (California), 1982, (Condor)	360 SC	22.4	1	91	fruit	0.01	80785 ⁵
USA (Sth Carolina), 1982, (Condor)	360 SC	22.4	1	83	fruit	<0.01	80786 ⁷
USA (Michigan), 1982, (Sweet Haven)	360 SC	22.4	1	93	fruit	<0.01	80787 ⁸
USA (Sth Carolina), 1982, (Red Globe)	360 SC	22.4	1	122	fruit	<0.01	80788 ⁷
USA (Georgia), 1982, (Winblo)	360 SC	22.4	1	106	fruit	<0.01	80789 ⁹
USA (Sth Carolina), 1982, (Rio-Oso-Gem)	360 SC	22.4	1	150	fruit	<0.01	80790 ⁷
USA (Sth Carolina), 1982, (Condor)	360 SC	22.4	1	83	fruit	<0.01	80799 ⁷
USA (Sth Carolina), 1982, (Red Globe)	360 SC	22.4	1	122	fruit	0.08, 0.08	80800 ⁷
USA (Sth Carolina), 1982, (Rio-Oso-Gem)	360 SC	22.4	1	150	fruit	<0.01	80801 ⁷

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
Italy (Bagnacavallo), 1986 (May Grand)	5 GR	25	1	85 93	fruit (depitted)	<0.02, <0.02 <0.02, <0.02	Bayer 1986c 5238-86 ¹⁰
GAP							
USA	350 EC	5.4-8.2		45			
Italy	5 GR	10-15	1	120			

¹ Single application to bare ground by broadcast with incorporation; growth stage at application: green fruit. Plot size two trees. No chromatograms provided.

² Single application by broadcast (within dripline); plot size 37.3 m². No chromatograms provided.

³ Single application by broadcast (within dripline); growth stage at application: bloom.

⁴ Single broadcast application (within dripline) at pre-bloom stage.

⁵ Single broadcast application (within dripline) at pre-bloom stage; plot size 3 trees.

⁶ Single application by broadcast and incorporation; plot size one tree; growth stage at application: late post-bloom.

⁷ Single application by broadcast and incorporation at full bloom stage; plot size 9 trees.

⁸ Single application by broadcast at pink stage; plot size 1 tree.

⁸ Single application by soil broadcast at pre-bloom stage; plot size 58 m².

⁹ Single application by spreading by hand at sepal fall; plot size 100 m², clay loam soil, pH 7.5, 2% C. Limit of determination 0.02 mg/kg, recovery at 0.02 mg/kg 87%. No chromatograms.

The rates in the US trials were 2.7 times the maximum registered rate. Residues in whole fruit were <0.01 to 0.16 mg/kg at the earliest pre-harvest intervals. In the Italian trial the application was at 1.7 times the GAP rate.

Grapes. Supervised trials were conducted in the USA, Mexico, South Africa and Chile. Residues were determined in grapes and raisins in some of the US trials. Recoveries from grapes, raisins and raisin trash with fortification at 0.01-0.1 mg/kg are shown in Table 61.

Table 84. Residues in grapes from supervised trials in Chile, Mexico, South Africa and the USA.

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
Mexico, (Valle de Mexicali), 1979, (Thompson Seedless)	15 GR	4.48	1	97	fruit	<u>0.01</u>	Mobay 1979b 68227 ¹
Mexico, (Sonora), 1979, (Thompson Seedless)	15 GR	4.48	1	69	fruit	<u>0.02</u>	68231 ²
Mexico (Sonora), 1979, (Thompson Seedless)	15 GR	6.72	1	69	fruit	<u>0.03</u>	68232 ²
Mexico (Sonora), 1979, (Thompson Seedless)	15 GR	4.48	1	70	fruit	<u>0.02</u>	68234 ³
USA (California), 1981, (Ruby Cabernet)	15 GR	6.72	3	58 72 85	fruit	<u>0.03</u> 0.02 0.02	Mobay 1981c 69658 ⁴
USA (California), 1981, (Zinfandel)	15 GR	6.72	3	56	fruit	<u>0.01</u>	80069 ⁵
USA (California), 1981, (Chenin blanc)	15 GR	6.72	3	58	fruit	<u>0.09</u> , 0.04	80070 ⁴
USA (Oregon), 1981, (White Riesling)	15 GR	6.72	3	56 70	fruit fruit	<u>0.02</u> 0.01	80071 ⁶
USA (California), 1981, (Ruby Cabernet)	15 GR	6.72	3	55 70	fruit	< <u>0.01</u> <0.01	80197 ⁷
USA (California), 1981, (Chenin blanc)	15 GR	6.72	3	50 63	fruit	<u>0.07</u> <0.01	80204 ⁸
USA (California), 1981, (Ruby Cabernet)	360 SC	6.72	3	58 72 85	fruit fruit fruit	< <u>0.01</u> <0.01 <0.01	Mobay 1981d 69659 ⁹
USA (California), 1981, (Thompson Seedless)	360 SC	6.72	3	55 70	fruit fruit	<u>0.07</u> 0.05	69745 ¹⁰
				80	fruit	0.04	
USA (California), 1981,	360 SC	6.72	3	56	fruit	< <u>0.01</u>	80075 ¹¹

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
(Tokay)							
USA (California), 1981, (Chenin Blanc)	360 SC	6.72	3	58 72 85	fruit fruit fruit	<u>0.02</u> <0.01 <0.01	80076 ¹²
USA (California), 1981, (White Riesling)	360 SC	6.72	3	56 70 84	fruit fruit fruit	<u>0.03</u> <0.01 <0.01	80077 ¹³
USA (California), 1981, (Thompson Seedless)	360 SC	6.72	3	56 73	fruit raisin s fruit raisin s	<u>0.02</u> <u>0.03</u> 0.01 0.01	80080 ¹⁴
USA (California), 1981, (Zinfandel)	360 SC	6.72	3	46 60 74	fruit	<0.01 <0.01 <0.01	80199 ¹⁵
USA (California), 1981, (Emperor)	360 SC	6.72	3	55 70	fruit	<0.01 <0.01	80200 ¹⁶
USA (California), 1981, (Chenin Blanc)	360 SC	6.72	3	0 13	fruit	<u>0.01</u> <0.01	80206 ¹⁷
USA (California), 1981, (Thompson Seedless)	360 SC	10	2	0 1 3 7	fruit	<0.01 <0.01 <0.01 <0.01	80081 ¹⁸
USA, (California), 1981, (Emperor)	360 SC	10	2	0 7	fruit	0.01 0.01	80082 ¹⁹
				14		<u>0.03</u>	
USA (California), 1981, (Thompson Seedless)	15 GR	10	2	0 1 3 7 14 21	fruit	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	80083 ²⁰
USA (California), 1981, (Emperor)	15 GR	10	2	0 1 3 7 14 21	fruit	0.03 0.02 <0.01 <u>0.03</u> 0.02 0.03	80084 ²¹
USA (California), 1981, (Emperor)	360 SC	10	2	0 1 3 7 14	fruit	<0.01 <0.01 <0.01 <0.01 <0.01	80207 ²²
USA (California), 1981, (Emperor)	15 GR	10	2	0 1 3 7 14	fruit	<u>0.02</u> <0.01 0.02 0.02 0.02	80208 ²²
South Africa, 1982, (Clairette Blanche)	400 EC	10 20	1 1	178 178	fruit fruit	<0.05, <0.05 <0.05, <0.05	S. Afr. Bur. Stds. 1983 311/88476/W331 ²³
	400 EC	10 20	2 1 1	178 177 177	fruit fruit fruit	<0.05, <0.05 <0.05, <0.05 <0.05, <0.05	
South Africa (Paarl), 1987, (Queen of the vineyard)	400 EC	1 g ai/m ²	1	97 117 129	fruit	<0.05, <0.05 <0.05, <0.05 <0.05, <0.05	S. Afr. Bur. Stds. 1987 311/88097/D34 ²⁴
(Alphonse Lavalee)	400 EC	2 g ai/m ²	1	97 117 129	fruit	<0.05, <0.05 <0.05, <0.05 <0.05, <0.05	
	400 EC	1 g ai/m ²	1	97 117 129	fruit	<0.05, <0.05 <0.05, <0.05 <0.05, <0.05	

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
		2 g ai/m ²	1	97 117 129	fruit	<0.05, <0.05 <0.05, <0.05 <0.05, <0.05	
Chile (Cuarta), 1988, (Thompson Seedless)	400 EC	6	1	73	fruit	<0.01, <u>0.01</u>	Fundacion Chile 1988 CHL-RENE0187-A1 ²⁵
Chile (Cuarta), 1987, (Thompson Seedless)	400 EC	12	1	73	fruit	0.02, 0.02	CHL-RENE0187-A2 ²⁵
Chile (Cuarta), 1987, (Thompson Seedless)	400 EC	6	1	80	fruit	<u><0.01</u> , <0.01	CHL-RENE0187-A3 ²⁵
Chile (Cuarta), 1987, (Thompson Seedless)	400 EC	12	1	80	fruit	0.02, 0.02	CHL-RENE0187-A4 ²⁵
Chile (Metropolitana), 1987, (Thompson Seedless)	400 EC	12	1	117	fruit	<0.01, <0.01	CHL-RENE0187-B1 ²⁶
Chile (Metropolitana), 1987, (Thompson Seedless)	400 EC	24	1	117	fruit	<0.01, <0.01	CHL-RENE0187-B2 ²⁶
Chile (Metropolitana), 1987, (Thompson Seedless)	400 EC	12	1	125	fruit	<0.01, <0.01	CHL-RENE0187-B3 ²⁶
Chile (Metropolitana), 1987, (Thompson Seedless)	400 EC	24	1	125	fruit	<0.01, <0.01	CHL-RENE0187-B4 ²⁶
Chile (Quinta), 1987, (Thompson Seedless)	400 EC	12	1	103	fruit	<0.01, <0.01	CHL-RENE0187-C1 ²⁷
Chile (Quinta), 1987, (Thompson Seedless)	400 EC	24	1	103	fruit	<0.01, <0.01	CHL-RENE0187-C2 ²⁷
Chile (Quinta), 1987, (Thompson Seedless)	400 EC	12	1	110	fruit	<0.01, <0.01	CHL-RENE0187-C3 ²⁷
Chile (Quinta), 1987, (Thompson Seedless)	400 EC	24	1	110	fruit	<0.01, <0.01	CHL-RENE0187-C4 ²⁷
Chile (Quinta), 1987, (Thompson Seedless)	400 EC	24	2	110	fruit	<0.01, <0.01	CHL-RENE0287-A1 ²⁸
Chile (Cuarta), 1987, (Thompson Seedless)	400 EC	5.16	2	84	fruit	<0.01, <u>0.01</u>	CHL-RENE0287-A2 ²⁹
Chile (Cuarta), 1987, (Thompson Seedless)	400 EC	10.32	2	84	fruit	<u>0.02</u> , 0.02	CHL-RENE0287-A3 ²⁹
Chile (Cuarta), 1987, (Thompson Seedless)	400 EC	5.16	2	90	fruit	<u><0.01</u> , <0.01	CHL-RENE0287-A4 ²⁹
Chile (Cuarta), 1987, (Thompson Seedless)	400 EC	10.32	2	90	fruit	<u>0.01</u> , 0.01	CHL-RENE0287-C1 ²⁹
Chile (Quinta), 1987, (Thompson Seedless)	400 EC	12	2	103	fruit	<0.01, <0.01	CHL-RENE0287-C2 ³⁰
Chile (Quinta), 1987, (Thompson Seedless)	400 EC	24	2	103	fruit	<0.01, <0.01	CHL-RENE0287-C3 ³⁰
Chile (Quinta), 1987, (Thompson Seedless)	400 EC	12	2	110	fruit	<0.01, <0.01	CHL-RENE0287-C4 ³⁰
GAP							
Chile	400 EC	6-8		45			
		2.8-4.8		45			
Mexico	400 EC	4-6					
South Africa	10 GR	1g ai/m²	1	100			
USA	360 EC	3.3-6.54	1-4	2			
		(band)					

¹ Single application as double sidedress band shortly before flowering; plot size 19.5 m², 0.3 m row spacing.

² Single application as double sidedress band at flowering; plot size 802 m², 0.3 m row spacing.

³ Single application as double sidedress band at flowering; plot size 535 m², 0.3 m row spacing.

⁴ Three applications (20 and 13 day intervals) by soil broadcast at blooming stage; plot size 89.1 m², 0.3 m row spacing.

⁵ Three applications (41 and 51 day intervals) by soil broadcast at 0.5 m high plants, pre-bloom and post-bloom stages; plot size 60.1 m², 0.22 m row spacing.

- ⁶ Three applications (35 and 39, 34 and 42 day intervals) by soil broadcast and incorporation at full bloom, 0.16-0.6 cm and 1.2 cm diameter fruit; bud stage, post-bloom and 0.9-1.2 cm diameter fruit; plot size 8.9 m².
- ⁷ Three applications (39 and 37 day intervals) by soil broadcast and incorporation at 0.15 m growth, bloom and berry formation; plot size 89.2 m², 0.3 m row spacing.
- ⁸ Three applications (12 and 23 day intervals) by soil broadcast and incorporation at bud break, bloom and berry formation; plot size 89.2 m², 0.3 m row spacing.
- ⁹ Three applications (29 and 13 day intervals) by soil broadcast and incorporation at blooming stage; plot size 89.2 m², 0.3 m row spacing.
- ¹⁰ Processing study. Three applications (14 and 13 day intervals) by soil incorporation at blooming stage; plot size 89.1 m², row spacing 3 m.
- ¹¹ Three applications (31 and 50 day intervals) by soil broadcast at 0.15 m growth, pre-bloom and post-bloom stages; plot size 45.1 m², row spacing 0.23 m. Chromatograms provided in full report.
- ¹² Three applications (23 and 13 day intervals) by soil broadcast at blooming stage, plot size 89.2 m², row spacing 0.3 m.
- ¹³ Three applications (35 and 39, 34 and 42, 19 and 52 day intervals) by soil broadcast and incorporation at full bloom, 0.16-0.6 cm and 1.2 cm diameter fruit; bud stage, post-bloom and 0.9-1.2 cm diameter fruit; plot size 8.9 m².
- ¹⁴ Processing study. Three applications (49 and 36 days intervals) by soil broadcast spray at 5-10 cm shoots, blooming and post-bloom; plot size 38.9 m², row spacing 0.3 m. Chromatograms provided in full report.
- ¹⁵ Three applications (31 and 62 day intervals) by soil broadcast spray at 0.15 m growth, pre-bloom and post-bloom stages; plot size 60.1 m²; row spacing 0.23 m.
- ¹⁶ Three applications (39 and 37 day intervals) at 0.15 m growth, bloom and berry formation; plot size 0.3 m, row spacing 0.3 m.
- ¹⁷ Three applications (57 and 73 days intervals) by soil broadcast spray and incorporation at bud break, bloom and berry formation stages; plot size 89.1 m², row spacing 0.3 m.
- ¹⁸ Two applications at 29 day interval by broadcast soil spray at fruiting and mature fruit stages; plot size 23 m², 0.2 m row spacing.
- ¹⁹ Two applications at 42 days interval by soil broadcast spray at maturing fruit stages; plot size 78 m², 0.3 m row spacing.
- ²⁰ Two applications by soil broadcast with incorporation at 29 days interval at fruiting and mature fruit stages; plot size 23.4 m², 0.2 m row spacing.
- ²¹ Two applications at 42 days interval by soil broadcast and incorporation at maturing fruit stages; plot size 78 m², 0.3 m row spacing.
- ²² Two applications by soil broadcast spray and incorporation at 36 days interval at 'colour changeng' and maturing fruit stages; plot size 78 m², 0.3 m row spacing.
- ²³ Applications to vines by knapsack. Recovery at 0.1 mg/kg of F, FSO, FSO₂ = 95%, 91% and 92% respectively. Limit of detection = 0.05 mg/kg.
- ²⁴ Limit of detection = 0.05 mg/kg, recovery of F, FSO and FSO₂ at 0.1 mg/kg = 106, 86; 90, 74; 110, 87% respectively. Samples stored for 158 days before analysis.
- ²⁵ Single application by drip irrigation 10 days after end of flowering; plot size 768 m². Soil type 'Franco arcillosa', 1% C. Limit of detection = 0.01 mg/kg, recovery at 0.2 mg/kg = 93.6%, at 0.04 mg/kg = 84.3%.
- ²⁶ Single application by drip irrigation at plant growth 50 to 70 cm; plot size 896 m², soil type 'Franca', pH 6.5-7.5, 1.58-2.11% C. Limit of detection = 0.01 mg/kg, recovery at 0.2 mg/kg = 93.6%, at 0.04 mg/kg = 84.3%.
- ²⁷ Single application by drip irrigation at pre-flowering stage; plot size 551 m², soil type 'Franco arcillosa', pH 7.5 0.63% C. Limit of detection = 0.01 mg/kg, recovery at 0.2 mg/kg = 93.6%, at 0.04 mg/kg = 84.3%.
- ²⁸ Two applications by drip irrigation at pre-flowering stages (231 day interval); plot size 551 m², soil type 'Franco arcillosa', pH 7.5 0.63% C. Limit of detection = 0.01 mg/kg, recovery at 0.2 mg/kg = 93.6%, at 0.04 mg/kg = 84.3%.
- ²⁹ Two applications by drip irrigation at pre-flowering and flowering (158 day interval); plot size 1287 m², soil type 'Franco arcillosa', pH 7.8, 1.58% C. Limit of detection = 0.01 mg/kg, recovery at 0.2 mg/kg = 93.6%, at 0.04 mg/kg = 84.3%.
- ³⁰ Two applications by drip irrigation at pre-flowering and flowering stages (231 day interval); plot size 551 m², soil type 'Franco arcillosa', pH 7.5, 0.63% C. Limit of detection = 0.01 mg/kg, recovery at 0.2 mg/kg = 93.6%, at 0.04 mg/kg = 84.3%.

The US and Chilean trials were at rates in excess of the registered use patterns. The residues in fruit from trials considered to be according to GAP were <0.01-0.09 mg/kg at harvest.

Bananas. Residues from supervised trials in Australia, Brazil, Costa Rica, Spain and the Windward Islands are shown in Table 85. Most of the data were provided in summary form, with reported recoveries and details shown as footnotes to the Table. In some cases chromatograms were not provided. Where the field and analytical phases of the trial were reported in accordance with GLP requirements the trial is described in detail below. From 1 to 3 sprays were applied per season and the residues were determined in the whole fruit, or pulp and peel separately. Where residues in pulp and peel were reported separately and were below the limit of detection in both, the residues in the whole fruit are assumed to be below the limit of detection. Recoveries were reported in supplement E019 to method 00024/M 002 at fortification levels of 0.02-0.5 mg/kg (Specht, 1995). Recoveries of fenamiphos sulfone were 64-97%; other recoveries are shown in endnotes to Table 85.

In a Spanish trial in the Canary Islands (Ohs, 1996), Nemacur 400 EC was applied twice to established banana plants at a rate of 5 g ai/plant by drip irrigation at re-treatment intervals of 161 or 166 days; the first application was at a growth stage of 5 to 9 leaves unrolled and the second was about 90 days before harvest. Plot sizes were 225, 471 and 482 m²; the cropping density was estimated as 700 plants/ha. Samples of bananas were taken at 14/15, 30/32, 53/55, 68/70 and 90 days after treatment and residues were determined in the whole fruit, pulp and peel.

In a subsequent trial in the Canary Islands (Heinemann and Ohs, 1997g), Nemacur 240 CS was again applied twice at 5 g ai/plant by drip irrigation, but with an interval of 210 or 212 days between treatments. The first application was at 17 or 25 leaves unrolled and the second at the end of flowering, or approximately 89 days before harvest. Crop densities were 1700 or 2000 plants/ha, which corresponded to application rates of 8.6 or 10 kg ai/ha. Samples were taken at 28/31, 60 and 89 days after the final treatment and residues were determined in whole fruit, pulp and peel.

Table 85. Residues in bananas from trials in Australia, Brazil, Costa Rica, Spain (Canary Islands) and the Windward Islands.

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.	
	Form.	kg ai/ha	No					
Australia (WA), 1971, (Mons Mari)	400 EC	4.48	1	28	whole fruit	<0.01	Bayer Austrl. 1971I 23/71a ¹	
Australia (WA), 1971, (Mons Mari)	400 EC	8.96	1	28	whole fruit	<0.01	23/71b ¹	
Australia (NSW), 1972, (Williams Hybrid)	5 GR	2.8 g ai/stool	2	21	whole fruit	<0.01	Bayer Austrl. 1972 14/72a ²	
Australia (NSW), 1972, (Williams Hybrid)	5 GR	2.8 g ai/stool	2	112	whole fruit	<0.01	14/72b ²	
Costa Rica (Limon), 1970, (Giant Cavendish)	10 GR	6 g ai/plant	1	1	pulp	<0.02	Burrows 1971a 30164 ³	
				3		<0.02 (0.03 c)		
				7		<0.02		
				14		<0.02		
				30		<0.02		
				61		<0.02		
				90		<0.02		
				195		<0.02		
				1		peel		<0.02
				3				<0.02
				7				<0.02
				14				<0.02
				30				<0.02
				61				<0.02
				90				0.03
	195	<0.02						
	1	pulp	<0.02					
	3		<0.02					
	7		<0.02					
	14		<0.02					
	30		<0.02					
61	<0.02							
90	<0.02							
195	<0.02							
1	peel		<0.02					
3			<0.02					
7			<0.02					
14			<0.02					
30			<0.02					
61			<0.02					
90			<0.02					
195		<0.02						
1		pulp	<0.02					
3			<0.02					
7			<0.02					
14			<0.02					
30			<0.02					
61			<0.02					
90		<0.02						
195	<0.02							
12 g ai/plant			1	1	pulp	<0.02		
				3		<0.02		
				7		<0.02		
				14		<0.02		
				30		<0.02		
				61		<0.02		

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.
	Form.	kg ai/ha	No				
				14 30 61 90 195 1 3 7 14 30 61 90 195	peel	<0.02 <0.02 <u><0.02</u> <0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <u><0.02</u> <0.02 <0.02	
Windward Islands, 1970, (Robusta)	10 GR	2.8 g ai/plant	1	1 2 7 14 31 69 96 1 2 7	pulp peel	<0.025 <0.025 <0.025 <0.025 <0.025 <u><0.025</u> <0.025 <0.025 <0.025 <0.025	Burrows, 1971b 30203 ⁴
		5.7	1	14 31 69 96 1 2 7 14 31 69 96 1 2 7 14 31 69 96	pulp peel	<0.025 <0.025 <u><0.025</u> <0.025 <0.025 <0.025 <0.025 <0.025 <0.025 <u><0.025</u> <0.025 <0.025 <0.025 <0.025 <0.025 <0.025 <0.025 <u><0.025</u> <0.025 <0.025 <0.025	
		11.3	1	1 2 7 14 31 69 96 1 2 7 14 31 69 96	pulp peel	<0.025 <0.025 <0.025 <0.025 <0.025 <u><0.025</u> <0.025 <0.025 <0.025 <0.025 <0.025 <0.025 <0.025 <u><0.025</u> <0.025 <0.025 <0.025 <0.025 <u><0.025</u> <0.025 <0.025 <0.025	
Brazil, 1983	10 GR	4 g ai/plant	1	95	whole fruit	<0.1	Fundacao de C. e T.1984 BRA-71133 ⁵
Brazil 1983	10 GR	4 g ai/plant	1	32	whole fruit	<u><0.1</u>	BRA-71134 ⁵
Brazil 1983	10 GR	4 g ai/plant	1	62	whole fruit	<0.1	BRA-71135-B ⁵
Brazil (Juquia), 1988, (Nanicao)	10 GR	3 g ai/plant	1	30 45 60	whole fruit	<u><0.1</u> <0.1 <0.1	U. Sao Paulo 1988a BRA-MUBP A87-1-A ⁶

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.
	Form.	kg ai/ha	No				
Brazil (Juquia), 1988, (Nanicao)	10 GR	6 g ai/plant	3	30	whole fruit	<0.1	BRA-MUBP A87-1-B ⁶
Canary Isl. (Buenavista del Norte), 1994 (Pequena Enana)	400 EC	5 g ai/plant	2	15	whole fruit	<0.02	Ohs 1996 40395-94 ⁷
				30		<0.02	
				55		<0.02	
				70		<0.02	
				90		<0.02, <0.02	
				90	pulp peel	<0.02	
Canary Isl. (Valle de Guerra), 1994 (Pequena Enana)	400 EC	5 g ai/plant	2	14	whole fruit	<0.02	40396-94 ⁸
				32		<0.02	
				53		<0.02	
				68		<0.02	
				90		<0.02, <0.02	
				90	pulp peel	<0.02	
Canary Isl. (Adeje), 1994, (Pequena Enana)	400 EC	5 g ai/plant	2	90	pulp peel whole fruit	<0.02, <0.02	40397-94 ⁹
Canary Isl. (Adeje), 1996, (Pequena Enana)	240 CS	5 g ai/plant	2	31	green fruit ripe fruit	<0.02, <0.02	Heinemann and Ohs 1997g 0274-96 ¹⁰
				60	green fruit ripe fruit	0.02, 0.02	
				89	green fruit ripe fruit	<0.02, <0.02	
				31	pulp	<0.02, <0.02	
				60		<0.02	
				89		<0.02, <0.02	
				31	peel	<0.02, <0.02	
				60		0.021	
Canary Isl. (Buenavista del Norte), 1996, (Pequena Enana)	240 CS	5 g ai/plant	2	28	ripe fruit	<0.02, <0.02	0368-96 ¹¹
				60	green fruit	<0.02, <0.02	
				89	green fruit	<0.02	
				28	pulp	<0.02, <0.02	
				60		<0.02	
				89		<0.02, <0.02	
				28	peel	<0.02, <0.02	
				60		<0.02	
89		<0.02, <0.02					
GAP							
Australia	100 G	2.5 g ai/plant	3				
	400 liq.	9.6-12 kg ai/ha	3				
Brazil	10 GR	2-3 g ai/plant	2	30			
Costa Rica	10 GR	5 kg ai/ha	1	60			
	15 GR	5.1	1	60			
Spain	10 GR	1.5-3 g ai/plant		60			
	400 EC	10-20 kg ai/ha		90			

¹ Application by flood irrigation; plot size 0.018 ha (20 stools). Limit of detection = 0.01 mg/kg, recovery at 0.1 mg/kg = 73%, at 0.5 mg/kg = 60%. No chromatograms provided.

² Two applications by hand in 15 cm band width 231 days apart. Limit of detection 0.01 mg/kg; recovery at 0.1 mg/kg = 94%, at 0.5 mg/kg = 91%.

³ Single application by broadcast around individual plants 3.5 months before harvest. Limit of detection = 0.02 mg/kg, recovery of F at 0.1 mg/kg = 104, 71 and 63% from pulp and 58, 60 and 59% from peel. Recovery of FSO and FSO₂ at 0.1 mg/kg = 65 and 85% respectively from pulp and 53 and 63% from peel. Recovery of F at 0.5 mg/kg = 90% from pulp and peel.

⁴ Single application in a 61 cm band around plant by spreading; heavy clay soil. Limit of detection = 0.025 mg/kg; recovery of FSO from peel at 0.05 mg/kg = 79%, recovery of F and FSO₂ at 0.1 mg/kg = 85 and 90% from pulp and 89 and 85% from

peel. Recovery of FSO = 77% from pulp at 0.4 mg/kg, 85% from peel at 0.1 mg/kg. Recovery of F at 0.4 mg/kg = 70% from peel.

⁵ Single application by spreading and incorporation. Limit of determination = 0.1 mg/kg, recovery at 0.1 mg/kg = 80%. No chromatograms provided.

⁶ Three applications (189 and 235 day intervals) by spreading; stage at last application fruit development; plot size 70 plants; clay sand, pH 5, 1.3% C. Limit of determination = 0.1 mg/kg, recoveries at 0.1 mg/kg 83-91%.

⁷ Two applications by drip irrigation at 166 day interval; plot size 482 m², loamy sand soil, pH 8, 1.7% C, age of crop >10 years.

⁸ Two applications by drip irrigation at 161 day interval; plot size 471 m², clay sand soil, pH 7.1, 1.4% C, age of crop >10 years.

⁹ Two applications by irrigation at 161 day interval; plot size 225 m², clay sand soil, pH 7.6, 1.3% C, age of crop >10 years. Recoveries at 0.02 mg/kg from fruit 64-91% (n = 4), from pulp 77-94% (n = 4), from peel 70-97% (n = 4).

¹⁰ Two applications by drip irrigation at 212 day interval, first application at 25 leaves unrolled and second at end of flowering; crop density 2000 plants/ha, clay sand soil, pH 7.1-7.6, 1.3-1.8% C.

¹¹ Two applications by drip irrigation at 17 leaves unrolled and end of flowering with 210 day interval.

Residues in bananas and banana pulp were below the limit of detection or determination in all trials. The trials in Costa Rica were designed to allow for crop densities of 416-833 plants/ha and those in the Windward Islands to allow for crop densities of 412-1785 plants/ha.

Pineapples. Data were predominantly from trials in Hawaii. Some trials from Australia and Puerto Rico were also reported. All the data were in the form of summary sheets, usually with chromatograms attached. Recoveries were reported from pineapple pulp, bran, foliage, forage and crowns, with fortification concentrations of 0.05-0.1 mg/kg in Table 61. Details of applications and timing are shown as footnotes to Table 86. In the Hawaiian trials residues were determined in by-products which could be fed to livestock such as foliage, crowns, stumps and bran, as well as in the edible portion of the commodity.

Table 86. Residues in pineapples and pineapple by-products.

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.
	Form.	kg ai/ha	No.				
Hawaii, (Libby), 1970, (Smooth Cayenne)	10 GR 3 LC	22.4 + 11.2 + 11.2 P	1 + 1 + 1	270	whole fruit wet bran foliage soil	<0.01 0.01 <0.01 0.34 (0.08 c)	Chemagro 1970b 27703 ¹
Hawaii, (Dole Lani), 1970, (Smooth Cayenne)	10 GR 3 LC	22.4 + 11.2 + 11.2 P	1 + 1 + 1	255	whole fruit wet bran foliage soil	0.03 0.03 8.68 2.17	27704 ²
Hawaii, (Wahiawa), 1970, (Smooth Cayenne)	10 GR 3 LC	22.4 + 11.2 + 11.2 P	1 + 1 + 1	238	whole fruit wet bran foliage soil	<0.01 <0.01 0.14, 0.11 0.44	27705 ³
Hawaii, (Wahiawa), 1970, (Smooth Cayenne)	10 GR 3 LC	22.4 + 11.2 + 11.2 P	1 + 1 + 1	262	whole fruit wet bran foliage soil	<0.01 <0.01 <0.01 0.06	27707 ⁴
Hawaii, (Wahiawa), 1970, (Smooth Cayenne)	10 GR 3 LC	22.4 + 11.2 + 11.2 P	1 + 1 + 1	265	whole fruit wet bran foliage soil	<0.01 0.03 0.94 0.14	27710 ⁴
Hawaii (Wahiawa), 1970, (Smooth Cayenne)	10 GR + 3 LC	22.4+ 11.2 + 11.2 P	1 + 1 + 1	251	whole fruit wet bran foliage soil	<0.01 <0.01 <0.01 0.18	27711 ⁵
Hawaii (Oahu), 1972, (Cayenne)	15 GR + 3 SC	22.4 + 5.6 P	1 + 4	217 224	whole fruit bran foliage	0.02 0.13 0.05	Chemagro 1972h 32018 ⁶
Hawaii (Molokai), 1971	15 GR + 3 SC	22.4 + 5.6 P	1 + 4	192	whole fruit bran	<0.01 <0.01	32019 ⁷

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.
	Form.	kg ai/ha	No.				
				199	foliage	0.06	
Hawaii (Molokai), 1973	15 GR + 360 SC	22.4 + 5.6 P	1 + 6	31	pulp bran dry bran foliage	<u>0.14</u> 1.07 2.30 4.78	Chemagro 1973e 39012 ⁸
Hawaii (Oahu), 1973	15 GR + 360 SC	22.4 + 5.6 P	1 + 6	83	pulp bran dry bran foliage	<u>0.02</u> 0.11 (0.02 c) 0.35 (0.07 c) 0.05 (0.11 c)	39013 ⁹
Australia, 1974, (Smooth Cayenne)	43.6%	1.1	5	330	whole fruit	<0.01	Bayer Australia 1974 2/74a ¹⁰
Australia, 1974, (Smooth Cayenne)	43.6%	2.2	5	330	whole fruit	<0.01	2/74b ¹⁰
Australia, 1974, (Smooth Cayenne)	43.6%	4.5	5	330	whole fruit	<0.01	2/74c ¹⁰
Puerto Rico (Manati), 1974, (Smooth Cayenne)	15 GR + 3 SC	22.4 + 11.2 P	1 + 3	223	pulp bran crowns leaves	<u><0.01</u> <0.01 <0.01 <0.01	Chemagro 1975 44744 ¹¹
Puerto Rico (Manati), 1975, (Smooth Cayenne)	15 GR + 3 SC	22.4 + 5.6 P	1 + 3	223	pulp bran crowns leaves	<u><0.01</u> <0.01 <0.01 <0.01	44745 ¹¹
Hawaii (Maui), 1976, (Smooth Cayenne)	3 SC	5.6 P	6	237	pulp wet bran dry bran crowns leaves stumps	<0.01 <0.01 <0.01 <0.01 <0.01 0.08	Chemagro 1976d 48145 ¹²
Hawaii (Maui), 1976, (Smooth Cayenne)	3 SC	2.8 P	6	237	pulp wet bran dry bran crowns leaves stumps	<0.01 <0.01 <0.01 <0.01 <0.01 0.12	48146 ¹²
Hawaii (Lanai), 1976, (Smooth Cayenne)	3 SC	5.6 R	8	159	pulp wet bran dry bran crowns leaves stumps	<u><0.01</u> <0.01 0.02 <0.01 0.09 0.11	48147 ¹³
Hawaii (Maui), 1976, (Smooth Cayenne)	3 SC	5.6 R	4	237	pulp wet bran dry bran crowns leaves stumps	<u><0.01</u> <0.01 <0.01 <0.01 <0.01 0.13	48148 ¹⁴
Hawaii (Lanai), 1976, (Smooth Cayenne)	3 SC	5.6 R	4	256	pulp wet bran dry bran crowns leaves stumps	<u><0.01</u> <0.01 <0.01 <0.01 <0.01 <0.01	48150 ¹⁵
Hawaii (Lanai), 1976, (Smooth Cayenne)	15 GR + 3 SC	22.4 + 5.6 P	1 + 4	256	pulp wet bran dry bran crowns leaves stumps	<u><0.01</u> <0.01 <0.01 <0.01 <0.01 <0.01	Chemagro 1976e 48149 ¹⁶

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.
	Form.	kg ai/ha	No.				
Hawaii (Lanai), 1982, (Smooth Cayenne)	360 EC	22.4 + 2.24 + 3.36 P	1 + 1 + 7	27	foliage pulp bran	0.01 <u><0.01</u> <0.01	Mobay 1982g 80646 ¹⁷
Hawaii (Maui), 1982, (Cayenne)	360 SC	22.4 + 3.17 + 3.36 P	1 + 6 + 1	30	foliage pulp bran crown	<0.01 <u><0.01</u> <0.01 <0.01	80647 ¹⁸
Hawaii (Lanai), 1982, (Smooth Cayenne)	360 SC	3.36 + 1.12 P	14 + 1	27	foliage pulp bran	0.04 <u><0.01</u> <0.01	80648 ¹⁹
Hawaii (Maui), 1982, (Cayenne)	360 SC	11.2 + 3.36 P	1 + 10	30	foliage pulp bran crown	0.03 <u><0.01</u> <0.01 <0.01	80649 ²⁰
Hawaii (Oahu), 1982, (Smooth Cayenne)	360 SC	22.4 + 2.24 + 3.36 P	1 + 1 + 6	36	foliage pulp bran crown	0.06 <u>0.01</u> <0.01 0.03	80650 ²¹
Hawaii (Lanai), 1982, (Smooth Cayenne)	360 SC	2.24 + 3.36 R	1 + 6	145	foliage pulp bran crown	0.02 <u><0.01</u> <0.01 <0.01	80658 ²²
Hawaii (Oahu), 1982, (Smooth Cayenne)	360 SC	2.24 + 3.36 R	1 + 6	31	foliage pulp bran crown	0.22 <u><0.01</u> <0.01 0.02	80659 ²³
Hawaii (Lanai), 1982, (Smooth Cayenne)	360 SC	2.24 + 3.36 R	1 + 6	145	foliage pulp bran crown	0.01 <u><0.01</u> <0.01 <0.01	80660 ²²
Hawaii (Lanai), 1982, (Smooth Cayenne)	360 SC	2.24 + 3.36 R	1 + 6	145	foliage pulp bran crown	0.03 <u><0.01</u> <0.01 0.02	80661 ²²
Hawaii (Oahu), 1982, (Smooth Cayenne)	360 SC	2.24 + 3.36 R	1 + 6	31	foliage pulp bran crown	0.07 <u><0.01</u> <0.01 0.05	80662 ²³
Hawaii (Oahu), 1982, (Smooth Cayenne)	360 SC	2.8 R	8	31	wet bran pulp crown foliage	0.25 <u>0.02</u> 0.30 1.21	82389 ²⁴
Hawaii (Oahu), 1982, (Smooth Cayenne)	360 SC	3.37 + 4.5 R	4 + 2	32	wet bran pulp dried bran foliage	0.71 <u>0.05</u> 2.60 2.64	82390 ²⁵
Hawaii (Lanai), 1982, (Smooth Cayenne)	360 SC	3.36 + 1.12 P	14 + 1	27	wet bran pulp crowns foliage	0.10 <u>0.01</u> 0.08 0.17 (0.03 c)	82391 ²⁶
Hawaii (Lanai), 1982, (Smooth Cayenne)	360 SC	2.24 + 3.36 R	1 + 6	139	wet bran pulp crowns foliage	<0.01 <u><0.01</u> 2.27 0.05	82392 ²⁷
Hawaii (Maui), 1982, (Cayenne)	360 SC	3.2 + 3.36 P	12 + 1	30	wet bran pulp crowns foliage	0.05 <u><0.01</u> 0.03 0.12	82393 ²⁸
Hawaii (Lanai), 1982, (Smooth Cayenne)	15 GR + 360 SC	22.4 + 2.24 + 3.36 P	1 + 1 + 7	27	foliage pulp bran	0.04 <u><0.01</u> <0.01	Mobay 1982h 80643 ²⁹
Hawaii (Maui), 1982, (Cayenne)	15 GR + 360 SC	22.4 + 3.17 + 3.36 P	1 + 6 + 1	30	foliage pulp bran	<0.01 <u><0.01</u> <0.01	80644 ³⁰

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.
	Form.	kg ai/ha	No.				
					crowns	<0.01	
Hawaii (Oahu), 1982, (Smooth Cayenne)	15 GR + 360 SC	22.4 + 3.36 + 2.24 P	1 + 6 + 1	36	foliage pulp bran crowns dried bran	0.06 <u><0.01</u> 0.01 0.03 0.03	80645 ³¹
Hawaii (Lanai), 1984, (Smooth Cayenne)	360 SC	2.24 + 3.36 R	1 + 6	139	wet bran pulp foliage	<0.01 <u><0.01</u> <0.01	Mobay 1984a 88759 ³²
Hawaii (Lanai), 1984, (Smooth Cayenne)	360 SC	2.24 + 3.36 R	1 + 6	139	wet bran pulp foliage	<0.01 <u><0.01</u> 0.03	88760 ³²
Hawaii (Maui), 1984, (Cayenne)	360 SC	22.4 + 2.1 + 3.36 P	1 + 9 + 1	30	wet bran pulp foliage	0.07 <u><0.01</u> 0.10	88761 ³³
Hawaii (Lanai), 1984, (Smooth Cayenne)	15 GR + 360 SC	22.4 + 2.24 + 3.36 P	1 + 1 + 7	27	wet bran pulp foliage	0.09 <u><0.01</u> 0.08 (0.03 c)	Mobay 1984b 88746 ³⁴
Hawaii (Lanai), 1984, (Smooth Cayenne)	15 GR + 360 SC	22.4 + 2.24 + 3.36 P	1 + 1 + 7	27	wet bran pulp foliage	0.05 <u><0.01</u> 0.18 (0.03 c)	88747 ³⁴
GAP Australia	400 liq.	2.4 (plant + ratoon)	5				
US (Puerto Rico)	15 GR 350 EC	4.8 (ratoon) 10 (pre-plant) 5.4-9.8 (post- plant, 1st ratoon)	2				
				225			
							Total application 20 kg ai/ha to ratoon crop.
US (Hawaii)		9.8 (Pre-plant)					Total application 26.2 kg ai/ha/plant crop
	350 EC	0.5-3.3 (post- plant)		30			Total application 9.8 kg ai/ratoon crop

P: plant crop

R: rotation crop

¹ Three applications: soil broadcast at 22.4 kg ai/ha with 10 GR 1 day before planting and two broadcast foliar sprays 156 and 308 days after planting. Limit of detection = 0.01 mg/kg.

² Three applications: soil broadcast at 22.4 kg ai/ha with 10 GR 8 days before planting and two broadcast foliar sprays 149 and 308 days after planting. Limit of detection = 0.01 mg/kg.

³ Three applications: soil broadcast at 22.4 kg ai/ha with 10 GR 5 days before planting and two broadcast foliar sprays 150 and 305 days after planting. Limit of detection = 0.01 mg/kg.

⁴ Three applications: soil broadcast at 22.4 kg ai/ha with 10 GR and two broadcast foliar sprays 187 and 376 days after planting. Limit of detection = 0.01 mg/kg.

⁵ Three applications: one soil broadcast with 10 GR 1 day before planting and two broadcast foliar sprays with 3 LC 153 and 309 days after treatment. Limit of detection = 0.01 mg/kg.

⁶ Five applications: one soil broadcast with 15 GR (pre-planting) and 4 foliar sprays with 3 SC 107, 213, 302 and 376 days after planting. Limit of detection = 0.01 mg/kg.

⁷ Five applications: one soil broadcast with 15 GR (pre-planting) and 4 foliar sprays with 3 SC 107, 202, 287 and 381 days after planting. Limit of detection = 0.01 mg/kg.

⁸ 7 applications: one soil broadcast with 15 GR (pre-planting) and 6 foliar sprays with 360 EC 96, 191, 276, 370, 495 and 559 days after planting. Limit of detection = 0.01 mg/kg.

⁹ Seven applications: one soil broadcast with 15 GR (pre-planting) and 6 foliar sprays with 360 EC 98, 204, 294, 368, 462 and 546 days after planting. Limit of detection = 0.01 mg/kg.

¹⁰ 5 sprays applied by knapsack 1, 92, 192, 271 and 377 days after planting. Limit of detection = 0.01 mg/kg, recovery at 0.05 mg/kg = 97%. No chromatograms provided.

¹¹ 4 applications: one pre-plant soil broadcast and incorporation with 15 GR and 3 foliar sprays at 92 day intervals. Limit of detection = 0.01 mg/kg.

¹² 6 foliar sprays 2 days before planting and 26, 92, 154, 216 and 288 days after planting. Limit of detection = 0.01 mg/kg.

¹³ 8 foliar sprays to plant crop and ratoon crop; ratoon crop harvested for analysis. First application 111 days after planting; remaining applications 82, 195, 250, 343, 551, 641 and 735 days after the first application. Plant crop harvested between sprays 5 and 6. Plot size 32.5m². Limit of detection = 0.01 mg/kg.

¹⁴ 4 sprays, first 88 days after planting, then 182, 276 and 348 days after planting. Limit of detection = 0.01 mg/kg.

¹⁵ 4 sprays, first 85 days after planting, then 184, 269 and 354 days after planting. Limit of detection = 0.01 mg/kg. Plot size 32.5 m².

¹⁶ 5 applications: one broadcast application at planting then foliar sprays 85, 184, 269 and 354 days after planting. Limit of detection = 0.01 mg/kg. Plot size 32.5 m².

¹⁷ 9 applications. Initial pre-plant broadcast 15 days before planting, after applications by drip irrigation at vegetative stages (105, 181, 251, 328, 392, 462 days after planting), mid-cone stages, (532 days after planting) and last application at immature fruit stage (637 days after planting). Plot size 30.2 m². Limit of detection = 0.01 mg/kg.

¹⁸ 8 applications. Initial broadcast spray with incorporation at planting, after applications by drip irrigation at vegetative stages (95, 180, 273, 376, days after planting), post-force stage (462 days), red cone stage (546 days) and immature fruit stage (625 days after planting). Plot size 30.2 m². Limit of detection = 0.01 mg/kg.

¹⁹ 15 applications by drip irrigation. Initial application 73 days after planting, after applications at vegetative stages (105, 136, 181, 218, 251, 289, 329, 361, 393, 442, 463 days), early bud stage (498 days), mid cone (533 days) and immature fruit (638 days after planting). Plot size 30.2 m². Limit of detection = 0.01 mg/kg.

²⁰ 10 applications by drip irrigation. Initial application 29 days after planting, after applications at vegetative stages (63, 117, 178, 245, 301, 374, 419), post-force (478 days), early petal (565 days), immature fruit (624 days after planting). Plot size 30.2 m². Limit of detection = 0.01 mg/kg.

²¹ 8 applications by drip irrigation. Initial application 10 days before planting, after applications at vegetative stages (84, 96, 195, 280 days after planting), developing bud stage (353, 413 days) and maturing fruit stage (503 days after planting). Plot size 251 m². Limit of detection = 0.01 mg/kg.

²² 7 applications by drip irrigation to ratoon crop. Initial application 709 days after planting; re-treatment at 30-41 day intervals at vegetative stages, post-force and early bud. Plot size 30.2 m². Limit of detection = 0.01 mg/kg.

²³ 7 applications by drip irrigation to ratoon crop at 25-79 day intervals at vegetative stages, force, developing bud and immature fruit stages. Plot size 251 m². Limit of detection = 0.01 mg/kg.

²⁴ 8 applications by drip irrigation to 1st ratoon crop at 20-71 day intervals at vegetative stages, developing bud stage and maturing fruit stages. Plot size 227 m². Limit of detection = 0.01 mg/kg.

²⁵ 6 applications by drip irrigation to 2nd ratoon crop at 13-47 day intervals at developing bud and maturing fruit stages. Plot size 227 m². Limit of detection = 0.01 mg/kg.

²⁶ 15 applications by broadcast foliar spray to plant crop. Initial application 58 days after planting then at 35-53 day intervals at vegetative, early bud, mid-cone, flowering and immature fruit stages. Plot size 30.2 m². Limit of detection = 0.01 mg/kg.

²⁷ 7 applications by broadcast foliar spray to ratoon crop. Applications at 34-79 day intervals at vegetative, post-force and early bud stages. Plot size 30.2 m². Limit of detection = 0.01 mg/kg.

²⁸ 12 applications by broadcast foliar spray. Initial application 27 days after planting, then at vegetative stages (63, 92, 119, 155, 180, 210, 245, 274 and 303 days), post-force 376 days, early petal 412 days and immature fruit, 626 days after planting. Plot size 30.2 m². Limit of detection = 0.01 mg/kg.

²⁹ 9 applications. Initial broadcast and incorporation with 15 GR 15 days before planting, then application by drip irrigation at vegetative stages (105, 181, 251, 328, 392, 462 days), mid-cone 532 days, and immature fruit 637 days after planting. Plot size 30.2 m². Limit of detection = 0.01 mg/kg.

³⁰ 8 applications. Initial application by broadcast and incorporation at planting with 15 GR, then application by drip irrigation with 360 SC at vegetative stages (95, 180, 274, 376 days post-force 462, red cone 547, immature fruit 626 days after planting). Plot size 30.2 m². Limit of detection = 0.01 mg/kg.

³¹ 8 applications. Initial application by broadcast and incorporation with 15 GR 7 days before planting, then application by drip irrigation with 360 SC at vegetative stages (95, 194, 279 days), developing bud 352 and 412 days and maturing fruit 471 and 592 days after planting. Plot size 251 m². Limit of detection = 0.01 mg/kg.

³² 7 applications by drip irrigation at intervals of 34-41 days. Plot size 30.2 m². Limit of detection = 0.01 mg/kg.

³³ 11 applications. Initial broadcast spray and incorporation at planting, then at vegetative stages (63, 117, 178, 243, 301, 374, 419 days), post-force 478, early petal 575, immature fruit 624 days after planting. Plot size 30.2 m². Limit of detection = 0.01 mg/kg.

³⁴ 9 applications. Initial broadcast and incorporation 11 days before planting, then at pre-bud stage 58, 163, 233, 303, 373 days, early bud stage 443 days, flowering 513 days, 596 days late immature fruit stages. Plot size 30.2 m². Limit of detection = 0.01 mg/kg.

The residues in whole fruit were <0.01 mg/kg in the three Australian trials, but only one trial was in accordance with GAP. In the trials in Puerto Rico the residues were <0.01 mg/kg in the pulp. All of the Hawaiian trials were at excessive rates of 1.2-2.3 times the total plant crop application rate and up to 2.3 times the total ratoon crop rate. Pre-planting applications were at 2.3 times the prescribed rate in all of the Hawaiian trials. The residues were <0.01-0.03 mg/kg in whole fruit and <0.01-0.05 mg/kg in pulp from these exaggerated treatments.

The residues in products used for animal feed were <0.01-8.68 mg/kg in foliage, <0.01-0.71 mg/kg in wet bran, <0.01-2.27 mg/kg in crowns, <0.02-0.02 mg/kg in dry bran and <0.01-0.13 mg/kg in stumps. Additional results are shown in Table 99 in pineapple processing trials. In a pineapple metabolism study (Flint, 1973) the total radioactive residues in fruit 30 days after soil treatment at 22.4 kg ai/ha were less than 10 ng/g (ppb) fenamiphos equivalents or fenamiphos phenol equivalents.

Peanuts. In trials in the USA and South Africa (Table 87) fenamiphos was applied at planting and samples were taken at normal harvest. The residues were determined in animal feed commodities such as vines, foliage and shells, in addition to the edible kernels. The dry matter contents of vines, foliage and dry foliage were not reported. Chromatograms were provided unless otherwise stated. Recoveries were determined at 0.1 mg/kg in peanut hulls and kernels and at 0.5 mg/kg in vines (Table 61).

Table 87. Residues in peanuts, peanut shells and vines from trials in South Africa and the USA.

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
USA (Texas), 1970, (Starr)	10 GR	13.4	1	125	nuts shells vines	<0.01 0.04 0.50	Chemagro 1970c 27020 ¹
USA (Nth Carolina), 1970, (NC-5)	10 GR	13.4	1	152 94	nuts shells vines	<0.01 <0.01 <0.01	27021 ¹
USA (Virginia), 1970, (Flori Giant)	10 GR	13.4	1	139	nuts shells vines	<0.01 0.05 0.94	27022 ¹
USA (Texas), 1970, (Starr)	10 GR	20.16	1	125	nuts shells vines	<0.01 0.02 2.05	27026 ¹
USA (Nth Carolina), 1970, (NC-5)	10 GR	13.4	1	153	nuts shells vines	<0.01 0.04 0.23	27028 ¹
USA (Mississippi), 1970, (Tennessee Red)	10 GR	13.4	1	140	nuts shells vines	<0.01 <0.01 0.41	27029 ¹
USA (Mississippi), 1970, (Tennessee Red)	10 GR	20.16	1	140	nuts shells vines	<0.01 0.02 0.21	27031 ¹
USA (Texas), 1970, (Starr)	3 SC	13.4	1	125	nuts shells vines	<0.01 0.01 1.89	Chemagro 1970d 27016 ¹
USA (Mississippi), 1970, (Tennessee Red)	3 SC	13.4	1	140	nuts shells vines	<0.01 <0.01 0.02	27017 ¹
USA (Mississippi), 1970, (Tennessee Red)	3 SC	20.16	1	140	nuts shells vines	<0.01 <0.01 0.04	27018 ¹
USA (Virginia), 1970, (Flori Giant)	3 SC	13.4	1	139	nuts shells vines	<0.01 <0.01 0.18	27019 ¹
USA (Nth Carolina), 1970, (NC-5)	3 SC	13.4	1	154	nuts shells vines	<0.01 0.31 0.20	27032 ²
USA (Nth Carolina), 1970, (NC-5)	3 SC	13.4	1	148	nuts shells vines	<0.01 <0.01 0.02	27033 ¹
USA (Texas), 1970, (Starr)	3 SC	13.4	1	125	nuts shells vines	<0.01 <0.01 3.19	27035 ¹
South Africa (Nth Cape), 1990, (Nordan)	400 EC	1.6 2.4 3.2	1 1 1	68 68 68	nuts foliage nuts foliage nuts foliage	<0.04, <0.04 <0.04, <0.04 <0.04, <0.04 <0.04, <0.04 <0.04, <0.04 0.04, 0.05	S. Afr. Bur. Stds.1990b 311/88899/G361 ³
South Africa (Transvaal), 1992, (Selly)	400 EC	1.6	1	63 76 90	whole plant foliage nuts foliage nuts	<0.04, <0.04 <0.04, <0.04 <0.04, <0.04 <0.04, <0.04 <0.04, <0.04	S. Afr. Bur. Stds.1994 311/88529/K102 ⁴

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
		3.2	1	104	foliage	<0.04, <0.04	
				116	nuts	<0.04, <0.04	
					dry	<0.04, <0.04	
					foliage		
					nuts	<0.04, <0.04	
				63	shells	<0.04, <0.04	
					whole	<0.04, <0.04	
					plant		
				76	foliage	<0.04, <0.04	
					nuts	<0.04, <0.04	
				90	foliage	<0.04, <0.04	
					nuts	<0.04, <0.04	
				104	foliage	<0.04, <0.04	
					nuts	<0.04, <0.04	
				116	dry	<0.04, <0.04	
					foliage	<0.04, <0.04	
					nuts	<0.04, <0.04	
					shells	<0.04, <0.04	
GAP	400 EC	1.6-	1	63			
South Africa		3.2					
USA	15 GR	1.68-	1				
		2.85					
	400 EC	1.63-	1				
		2.70					

¹ Single broadcast application with incorporation in soil. Limit of detection = 0.01 mg/kg.

² Single broadcast application with incorporation in soil. Limit of detection = 0.01 mg/kg. Recovery at 0.1 mg/kg = 92%.

³ Single application at planting by tractor mounted applicator. Plot size 2800 m², sandy loam soil, pH 6. Foliage samples included leaves and pods. Recovery at 0.1 mg/kg of F, FSO and FSO₂ = 84, 60 and 92% respectively from nuts, 67, 61 and 100% from foliage or whole plant. No chromatograms provided.

⁴ Single application by high volume spray. Plot size 815 m², soil composition: 24% clay, 7% silt and 69% sand; pH 7.6, 0.43% C. Whole plant samples included foliage and pods. Limit of detection = 0.04 mg/kg. Recovery of F, FSO and FSO₂ 63, 66 and 90% from dry foliage and 77, 74 and 90% from shells at 0.04 mg/kg, 81, 100 and 88% from foliage at 0.05 mg/kg and 75, 61 and 72% at 0.1 mg/kg, 67, 95 and 76% from dry foliage and 76, 76 and 69% from nuts at 0.2 mg/kg, and 82, 73 and 67% from nuts at 0.08 mg/kg.

Although up to 7 times the registered rate was applied in the US trials the residues in nuts were <0.01 mg/kg at the earliest harvest intervals. The residues in vines were <0.01-3.19 mg/kg at 4.7-7 times the registered rate after 94-154 days.

Cotton. The results of supervised trials in South Africa, Brazil and the USA are shown in Table 88. In all the trials fenamiphos was applied either before or shortly after planting and cotton seed, gin trash and foliage samples were collected at normal harvest. Recoveries from cotton seed fortified at 0.05 mg/kg were recorded in Table 61.

Table 88. Results of supervised field trials on cotton in the USA, Brazil and South Africa.

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.
	Form.	kg ai/ha	No.				
USA (Oklahoma), 1971, (Lankburn)	15 GR	4.06	1	161	seed gin trash	<0.01 <0.01	Chemagro 1971a 29981 ¹
USA (Arkansas), 1971, (Stoneville 213)	15 GR	3.66	1	163	seed	<0.01	30027 ²
				148	gin trash foliage	<0.01 0.10	
USA (Louisiana), 1971, (D.P.L. Smooth Leaf)	15 GR	3.66	1	196	seed gin trash	<0.01 <0.01	30028 ²
USA (California), 1971, (Delta Pine)	15 GR	3.85	1	157	seed foliage	<0.01 <0.01	30031 ³
USA (Louisiana), 1971, (D.P.L. Smooth Leaf)	15 GR	5.50	1	196	seed gin trash	<0.01 <0.01	30032 ¹

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference.
	Form.	kg ai/ha	No.				
USA (California), 1971, (Delta Pine)	3 SC	3.85	1	157 142	seed foliage	<0.01 <0.01	Chemagro 1971b 30025 ⁵
USA (Louisiana), 1971, (D.P.L. Smooth Leaf)	3 SC	3.66	1	196	seed gin trash	<0.01 <0.01	30026 ²
USA (Louisiana), 1971, (D.P.L. Smooth Leaf)	3 SC	5.5	1	196	seed gin trash	<0.01 <0.01	30029 ⁴
USA (Arkansas), 1971, (Stoneville)	3 SC	3.65	1	163 148	seed gin trash foliage	<0.01 <0.01 0.04	30030 ²
USA (Oklahoma), 1971, (Lankburn)	3 SC	4.06	1	161	seed gin trash	<0.01 <0.01	30033 ¹
USA (Arizona), 1977, (D & PL 16)	15 GR	1.83	1	155	seed	<0.01	Chemagro 1977b 52992 ⁵
USA (Texas), 1977, (SP 37)	15 GR	5.59	1	131	seed	<0.01	52993 ⁶
USA (Arizona), 1977, (DPL 16)	15 GR	3.65	1	155	seed	<0.01	52994 ⁷
USA (Sth Carolina), 1977, (Coker 201)	15 GR	2.04	1	145	seed	0.05 (0.03 c)	53118 ⁸
USA (Texas), 1977, (SP 37)	3 SC	3.68	1	131	seed	<0.01	Chemagro 1977c 52995 ⁹
USA (Texas), 1977, (Lockett 4789A)	3 SC	3.68	1	183	seed	<0.01	52996 ¹⁰
USA (Arizona), 1977, (DPL 16)	3 SC	3.65	1	155	seed	<0.01	52997 ⁷
USA (Texas), 1977, (SP 37)	3 SC	5.59	1	130	seed	<0.01	52998 ⁶
USA (Arizona), 1977, (DPL 16)	3 SC	5.5	1	155	seed	<0.01	52999 ⁴
USA (Sth Carolina), 1977, (Coker 201)	3 SC	2.04	1	145	seed	0.04 (0.03 c)	53117 ¹¹
USA (Mississippi), 1979, (Stoneville 213)	15 GR	1.93	1	147	seed	<0.01	Mobay 1979c 66865 ¹²
USA (Mississippi), 1979, (Stoneville 213)	15 GR	2.89	1	147	seed	<0.01	66866 ¹³
USA (Mississippi), 1979, (Stoneville 213)	3 SC	1.92	1	147	seed	<0.01	Mobay 1979d 66867 ¹¹
USA (Mississippi), 1979, (Stoneville 213)	3 SC	2.89	1	147	seed	<0.01	66868 ¹³
Brazil, (Rolandia), 1987, (IAC-20)	10 GR	4 g ai/plant	1	92 120 147	boll fuzzy seed fuzzy seed	<0.2 <0.2 <0.2	U. de Sao Paulo 1988b BRA-1004-88-A ¹⁴
Brazil (Rolandia), 1987, (IAC-20)	10 GR	8 g ai/plant	1	120	fuzzy seed	<0.2	BRA-1004-88-B ¹⁵
South Africa (Nth Cape), 1989, (Acala)	400 EC	4	1	126 195 210	delinted seed	<0.05, <0.05 <0.05, <0.05 <0.05, <0.05	S. Afr. Bur. Stds. 1990c 311/88944/G528 ¹⁶
South Africa (Nth Cape), 1989, (Acala)	400 EC	4	1	126 195 210	delinted seed	<0.05, <0.05 <0.05, <0.05 <0.05, <0.05	311/88900/G362 ¹⁷
GAP Brazil South Africa	10 GR 10 GR	3-5 15 g ai/100 m row	1	98			
USA	15 GR 350 EC	0.84- 1.65 0.82- 3.27					

¹ Application in 30.5 cm band with incorporation at planting (0.375 g/m row, 91 cm row spacing). Limit of detection = 0.01 mg/kg.

- ² Application in 30.5 cm band with incorporation at planting (0.375 g/m row, 102 cm row spacing). Limit of detection = 0.01 mg/kg.
- ³ Application in 30.5 cm band with incorporation at planting (0.375 g/m row, 96.5 cm row spacing). Limit of detection = 0.01 mg/kg.
- ⁴ Application as a band in-furrow at planting (0.56 g/m row, 102 cm row spacing). Limit of detection = 0.01 mg/kg.
- ⁵ Application in 15.2 cm band at planting (0.19 g/m row, 102 cm row spacing). Limit of detection = 0.01 mg/kg. No chromatograms provided.
- ⁶ Application in 15.2 cm band at planting (0.28 g/m row, 51 cm row spacing). Limit of detection = 0.01 mg/kg. No chromatograms provided.
- ⁷ Application in 15.2 cm band at planting (0.375 g/m row, 102 cm row spacing). Limit of detection = 0.01 mg/kg. No chromatograms provided.
- ⁸ Application in 15.2 cm band at planting (0.19 g/m row, 102 cm row spacing). Limit of detection = 0.01 mg/kg. No chromatograms provided.
- ⁹ Application in 15.2 cm band at planting (0.19 g/m row, 51 cm row spacing). Limit of detection = 0.01 mg/kg. No chromatograms provided.
- ¹⁰ Application in 10.2 cm band at planting (0.19 g/m row, 51 cm row spacing). Limit of detection = 0.01 mg/kg. No chromatograms provided.
- ¹¹ Application in 15.2 cm band at planting (0.19 g/m row, 91 cm row spacing). Limit of detection = 0.01 mg/kg. No chromatograms provided.
- ¹² Applied in 10 to 15.2 cm band at planting (0.19 g/m row, 96.5 cm row spacing). Plot size 1486 m². Limit of detection = 0.01 mg/kg. No chromatograms provided.
- ¹³ Applied in 10 to 15.2 cm band at planting (0.28 g/m row, 96.5 cm row spacing). Plot size 1486 m². Limit of detection = 0.01 mg/kg. No chromatograms provided.
- ¹⁴ Application by spreading at planting. Plot size 120 m², clay soil, pH 6.2, 4% C. Recovery at 0.2 mg/kg 74-80%.
- ¹⁵ Application by spreading at planting. Plot size 80 m², clay soil, pH 6.2, 4% C. Recovery at 0.2 mg/kg 74-80%.
- ¹⁶ Applied by pivot irrigation at planting. Plot size 2.5 ha, sandy loam soil, pH 5.8. Limit of detection = 0.05 mg/kg. Recovery at 0.1 mg/kg = 89, 100 and 83% of F, FSO and FSO₂ respectively. No chromatograms provided.
- ¹⁷ Applied by pivot irrigation at planting. Plot size 2 ha, sandy loam soil, pH 5.5. Limit of detection = 0.05 mg/kg. Recovery at 0.1 mg/kg = 89, 100 and 83% of F, FSO and FSO₂ respectively. No chromatograms provided.

The residues in cotton seed were below the limit of detection or determination in many of the trials. Residues above the limit of detection were found in 2 cotton seed samples but also in the corresponding controls. Residues were present in foliage in some trials where the foliage was sampled before harvest of the seed.

Coffee. Trials were conducted in Guatemala, Brazil and Mexico (Table 89). A single soil application of fenamiphos was made to mature trees at pre-bloom or fruit formation. Residues were determined in the berries and beans. The MRLs apply to the seed (bean) only.

Table 89. Residues in coffee berries and beans from trials in Guatemala, Brazil and Mexico.

Location, year, (variety)	Application			PHI, days	Sample	Residues, mg/kg	Reference
	Form.	kg ai/ha	No.				
Guatemala, (Escuintla), 1978, (Bourbon)	10 GR	2.5 g ai/plant	1	30	bean	<0.1	Inst. Nut. Cent. Amer.y Panama 1978 GUA-78-2-13-A ¹
Guatemala (Escuintla), 1978, (Bourbon)	10 GR	2.5 g ai/plant	1	45	bean	<0.1	GUA-78-2-13-B ¹
Guatemala (Escuintla), 1978, (Bourbon)	10 GR	2.5 g ai/plant	1	60	bean	<0.1	GUA-78-2-13-C ¹
Guatemala (Escuintla), 1978, (Bourbon)	10 GR	2.5 g ai/plant	1	90	bean	<0.1	GUA-78-2-13-D ¹
Brazil (Sao Paulo), 1988, (Catuai)	10 GR	7 g ai/plant	2	30 45 60	bean	<0.2 <0.2 <0.2	U. de Sao Paulo 1988c BRA-1002-88-A ²
Brazil (Sao Paulo), 1988, (Catuai)	10 GR	14 g ai/plant	2	45	bean	<0.2	BRA-1002-88-B ³
Guatemala (Mazatenango) 1986, (Caturra)	12 GR	4	1	60	berry	<0.05	Bayer 1986d
		2	1	60	berry	0.04	0021-86-A ⁴
Guatemala (Mazatenango) 1986, (Caturra)	12 GR	4	1	45	berry	<0.05	0021-86-B ⁴
		2	1	45	berry	0.03	
Guatemala (Mazatenango) 1986, (Caturra)	12 GR	4	1	30	berry	<0.05	0021-86-C ⁴
		2	1	30	berry	0.01	

Location, year, (variety)	Application			PHI, days	Sampl e	Resid ues, mg/kg	Reference
	Form.	kg ai/ha	No.				
Guatemala (Mazatenango) 1986, (Caturra)	12 GR	4	1	15	berry berry	<u><0.05</u>	0021-86-D ⁴
		2	1	15		<u>0.02</u>	
Guatemala (Mazatenango) 1986, (Caturra)	12 GR	8	1	60	berry berry	<0.05	Bayer 1986e 0035-86-A ⁴
		4	1	60		<u>0.01</u>	
Guatemala (Mazatenango) 1986, (Caturra)	12 GR	8	1	45	berry berry	<0.05	0035-86-B ⁴
		4	1	45		<u>0.11</u>	
Guatemala (Mazatenango) 1986, (Caturra)	12 GR	8	1	30	berry berry	<0.05	0035-86-C ⁴
		4	1	30		<u>0.06</u>	
Guatemala (Mazatenango) 1986, (Caturra)	12 GR	8	1	15	berry berry	<0.05	0035-86-D ⁴
		4	1	15		<u>0.01</u>	
Mexico (Coatepec), 1988, (Mundo Nuevo)	15 GR	1.5 g ai/m tree height	1	123	bean	<0.01	Leslie 1988b 96780 MEX-3900-81H ⁵
			154	154		0.09	
Mexico (Coatepec), 1988, (Garnica)	15 GR	1.5 g ai/m tree height	1	123	bean	<0.01	MEX-3901-81H ⁵
Mexico (Xico), 1984, (Garnica)	15 GR	1.5 g ai/m tree height	1	122	bean	<0.01	MEX-3902-81H ⁵
Mexico (Xico), 1984, (Mundo Nuevo)	15 GR	1.5 g ai/m tree height	1	123	bean	0.08	MEX-3903-81H ⁵
				154		0.10	
GAP							
Brazil	10 GR	1– 1.5 g ai/plant	3	45			
Guatemala	10 GR	5	1	60			
	12 GR	2.4-3.6					
	15 GR	5.1					
Mexico	10 GR	1-1.5 g ai/plant	1	45			

¹ Single application by spreading around trunks at fruit development. Plot size 15 plants. Limit of determination = 0.1 mg/kg. No chromatograms provided.

² Two applications at 49 day interval with final application at the beginning of fruit ripening. Applied by spreading. Plot size 40 plants, clay soil, pH 5.7, 1.68% C. Limit of determination = 0.2 mg/kg, recoveries at 0.2 mg/kg 77-87%. No chromatograms provided.

³ Two applications at 49 day interval with final application at the beginning of fruit ripening. Applied by spreading. Plot size 20 plants, clay soil, pH 5.7, 1.68% C. Limit of determination = 0.2 mg/kg, recoveries at 0.2 mg/kg 77-87%. No chromatograms provided.

⁴ Single application by spreading. Plot 132 m², loamy soil, pH 5.6, 6.8% C. Limit of determination = 0.05 mg/kg., recovery at 0.05 mg/kg = 72%. No chromatograms provided.

⁵ Single in-furrow application at pre-bloom stage around tree at drip line. Plot size 54 m², silty clay soil, pH 5.5-6.4, 3-4% C. Recoveries of F, FSO and FOS₂ at 0.05 mg/kg = 86, 70 and 78% respectively. Recoveries of F at 0.1 mg/kg = 79 and 86%, and at 0.5 mg/kg = 77%.

The residues in the Guatemala trials were <0.01-<0.02 mg/kg in the beans from the 10 GR and 0.01-0.11 mg/kg in the berries from the 12 GR product. The Mexican application rates were expressed in terms of g ai/tree height.

Animal feeding studies

Alfalfa pellets containing fenamiphos sulfoxide were fed to dairy cattle for 28 days at levels equivalent to 2, 6 and 20 ppm in the diet (Wargo, 1978). The sulfoxide was used as it was considered to be the major component of the residues found in treated crops which may be used as animal feed items. The feed levels equated to 44, 151 and 493 mg/kg bw/day for an average feed intake of 15 kg/animal/day and the average body weight of each group. There were 3 cows in each treatment group and one control animal. Blood samples were taken on days 0, 7, 14, 21 and 28 of the trial and cholinesterase activity was compared with that in blood taken at 3 intervals in the 7 days before dosing.

The cows were milked in the morning and evening and samples of milk from the cows in each group for each day were composited for analysis. On day 29 the animals were killed and samples of

liver, kidney, muscle (flank and loin), and fat (omental, subcutaneous and renal) were extracted for analysis less than 24 hours after death. The limit of detection was reported as 0.001 mg/kg in the milk and 0.01 mg/kg in the tissues.

Cholinesterase activity was not reduced after feeding at the 2 and 6 ppm levels, but significant depression of activity was noted by day 21 at 20 ppm.

The residues were <0.001 mg/kg in all milk samples taken from the highest dose group. Residues in the liver were ≤ 0.01 mg/kg in the 6 ppm group and <0.01 to 0.012 mg/kg in the 20 ppm group (levels of 0.011-0.012 mg/kg were found in 1 of the 3 animals). In the kidneys, composite fat and muscle taken from the 20 ppm group, residues were all <0.01 mg/kg.

Groups of 4 laying hens were fed for 14 consecutive days with [U-*phenyl*-¹⁴C]fenamiphos at 0, 0.06, 0.18 and 0.65 ppm incorporated into the feed each morning (Gronberg *et al.*, 1973). The feed concentrations corresponded to average intakes of 3.42, 10.18 and 37.96 μg fenamiphos/kg bw/day. Eggs were collected each morning and the hens were killed after 14 days for the analysis of blood, brain, skin, muscle, heart, liver, gizzard, kidney and fat.

The radioactive residues in eggs reached maximum levels after 7 days feeding in all groups. The limit of detection in eggs was reported as 0.003 mg/kg. Residues in the tissues of the high-dose group are shown in Table 90.

Table 90. Total radioactive residues in hen tissues and blood after feeding at 0.65 ppm (Gronberg *et al.*, 1973).

Sample	TRR, $\mu\text{g}/\text{kg}$ as fenamiphos
Brain	2.58
Heart	5.32
Liver	4.51
Kidney	4.70
Muscle	2.76
Fat	2.96
Gizzard	4.22
Skin	2.46
Whole blood	3.90

The highest radioactive residues were found in heart, kidney, liver and gizzard.

In a subsequent trial (Bell *et al.*, 1974), groups of four laying hens were fed for 14 consecutive days with [U-*phenyl*-¹⁴C]fenamiphos at 2, 4 and 100 ppm in the feed, corresponding to average concentrations of 0.12, 0.28 and 0.76 mg fenamiphos/kg bw/day. Eggs were collected each morning and samples of brain, liver, kidney, fat, gizzard, heart, and white and dark muscle were collected at death.

The total radioactivity in eggs reached maximum levels after 6 days feeding in all groups, similar to the 7 days found in the Gronberg study. The maximum residues in the tissues were below the minimum quantifiable limits (7-20 $\mu\text{g}/\text{kg}$) in the 2 and 4 ppm groups. At the 10 ppm feeding level, residues of 47, 27 and 18 $\mu\text{g}/\text{kg}$ were present in the gizzard, kidneys and liver respectively.

Estimation of dietary burden for livestock

Estimates of the exposure of cattle and hens to fenamiphos residues in treated feed items are shown in Tables 91 and 92, together with the estimated maximum and median residues in animal feed commodities. All median residues have been estimated from the results of trials which were considered to be according to GAP. Items for which intake figures were not available but which may be used in animal feed have also been included. The main contributions to the intake are shown bold. For cattle an intake of 15 kg dry matter/day is assumed, with an average body weight of 500 kg.

Table 91. Estimated dietary burden for dairy cattle.

Commodity	Maximum residue, mg/kg	Median residue, mg/kg	% in the feed	% DM	Intake, mg/animal/day
Citrus peel	0.71	0.06	20		0.18
Citrus pulp		0.01	20		0.03
Apple pomace, dry		0.18	40		1.08
Grape pomace, dry		0.1	20		0.3
Raisin trash		0.15	20		0.45
Peanut meal		0.01	15		0.02
Peanut vines	0.05	0.04	25	85	0.15
Cotton seed	<0.01	<0.01	25	88	<0.038
Cotton hulls		0.01	20	89	0.03
Cotton meal		0.01	15		0.02
Gin by-products		<0.01	20		0.03
Tomato pulp, dry		0.13	10		0.195
Tomato pomace		0.12	10		0.18
Pineapple wet bran	1.07	<0.01			
Pineapple dry bran	2.60	<0.01	30		0.045
Pineapple foliage	8.68	0.04			
Pineapple crowns	2.27	<0.01			
Cabbage trash	0.19				

The total exposure from a diet composed of the items providing the highest median intake (dry apple pomace, raisin trash, peanut vines and dry tomato pulp) is 1.875 mg/animal/day or 0.125 ppm in the feed (0.004 mg/kg bw/day). The lowest feed level in the cattle trial reported by Wargo (1978) was 2 ppm fenamiphos sulfoxide, or about 15 times the estimated intake.

The intake for hens was estimated assuming an average body weight of 2 kg and an intake of 150 g dry matter/day.

Table 92. Estimated dietary burden for laying hens.

Commodity	Maximum residue, mg/kg	Median residue, mg/kg	% in the feed	% DM	Intake, mg/animal/day
Cotton meal		0.01	20		0.0003
Peanut meal		0.01	25		0.0003

Assuming 100% intake of peanut meal, hens would be exposed to 0.01 ppm in the feed. The lowest feed level in the reported hen feeding studies was 0.06 ppm of [U-*phenyl*-¹⁴C]fenamiphos (Gronberg *et al.*, 1973). The total radioactive residues in the tissues and eggs were reported without characterization of the radioactivity. In a subsequent feeding study (Bell *et al.*, 1974) the maximum residues in the tissues were below 20 µg/kg after feeding at the 2 ppm level.

FATE OF RESIDUES IN STORAGE AND PROCESSING

Processing studies with tomatoes, oranges, apples, grapes and pineapples were reported.

Tomatoes. Whole tomatoes containing 0.5 mg/kg fenamiphos were subjected to commercial processing into canned tomatoes, pasteurised juice and ketchup (Morris, 1975). The residues in the processed fractions and the corresponding processing factors are shown in Table 93.

Table 93. Residues and processing factors from a tomato processing trial (Morris, 1975).

Sample	Residue, mg/kg	Processing factor
Whole tomatoes	0.50	NA
Pasteurised tomato juice	0.44	0.88
Sterilized tomatoes (canned)	0.36	0.72
Tomato ketchup	0.29	0.58
Tomato juice	0.37	0.74

Sample	Residue, mg/kg	Processing factor
Tomato pulp solids	0.52	1.04s
Dry tomato seeds and fibres	0.79	1.58
Dry tomato peels and cores	1.89	3.78
Dry tomato pulp solids	3.12	6.24
Dry tomato pomace	1.25	2.5

The recoveries of fenamiphos, fenamiphos sulfoxide and fenamiphos sulfone at 0.1 mg/kg were 100, 100 and 101% respectively in whole tomatoes, 91, 93 and 92% in ketchup, and 107, 84 and 84% in dried tomato pulp.

In another study (Simmons and Thornton, 1975), raw tomatoes were fortified with [U-*phenyl*-¹⁴C]fenamiphos sulfoxide at a concentration of 0.8 mg/kg. The tomatoes were allowed to stand at room temperature for 24 hours, then blanched, peeled, cored and cooked in canning jars for 40 minutes. The jars were cooled and the fractions analysed for radioactive residues.

The residues were extracted into organic solvents and the organosoluble radioactivity was distributed among the fractions as shown in Table 94.

Table 94. Distribution of radioactive residues in tomatoes fortified with [¹⁴C]fenamiphos sulfoxide and their processed fractions (Simmons and Thornton, 1975).

Sample	Total ¹⁴ C*	FSO**	FSO ₂ **	FPSO**	FPSO ₂ **
Blanching water	3.7 (0.03)	96.7 (0.03)	2.3 (<0.001)	0.9 (0.0003)	0.15 (<0.001)
Cooking water	23.4 (0.19)	95.3 (0.143)	2.6 (0.004)	0.7 (0.001)	0.09
Peels and cores	1.5 (0.01)	83.6 (0.009)	6.4 (<0.001)	6.5 (<0.001)	3.5 (<0.001)
Tomatoes	54.3 (0.44)	97 (0.41)		0.9 (0.004)	2.0 (0.008)
Filter paper	0.2 (0.002)				

* % of total applied ¹⁴C and (mg/kg as FSO)

** % of ¹⁴C in organic extract and (mg/kg as FSO).

The results indicate that the organosoluble radioactivity was largely unchanged fenamiphos sulfoxide and that the cooking process leads to little chemical change. Blanching and cooking reduced the residues in the tomatoes by almost 50%, with 27% present in the blanching and cooking water. There was negligible loss by removing peels and cores.

Oranges. Oranges trees were treated with Namacur 15% granular at a rate equivalent to 100 kg ai/ha (Thornton, 1976). Leaves were taken at monthly intervals to determine when peak residues had moved systemically into the upper parts of the trees. When residues had reached plateau levels in the leaves, fruit were harvested and processed. The commercial processing procedure commenced with a pre-rinse wash, scrubbing with soap and water and an after-wash rinse.

The separate fractions analysed included the rinse waters, juice, peel, pulp, orange oil, dried peel and molasses. The residue data and corresponding processing factors are shown in Table 95. The residues in whole fruit were <0.01 to 0.13 mg/kg (average 0.07 mg/kg). Recoveries were determined in several fractions and are shown in Table 61.

Samples of oranges were held in frozen storage for 578 or 579 days with 15% degradation of fenamiphos and 13% degradation of fenamiphos sulfoxide. There was no observed degradation of fenamiphos sulfone.

Table 95. Residues in processed fractions of oranges and processing factors (Thornton, 1976).

Sample	Residue, mg/kg	Processing factor
Whole fruit	0.07	-
Peel (unwashed)	0.47	6.71
Pulp (unwashed)	<0.01	0.14

Sample	Residue, mg/kg	Processing factor
Peel (washed)	0.60	8.57
Pulp (washed)	0.01	0.14
Juice	0.02	0.28
Finisher pulp	0.02	0.28
Peel bits	0.23	3.28
Clear oil	4.48	64
Chopped peel	0.13	1.86
Pressed dry peel	0.40	5.71
Press liquor	0.20	2.85
Molasses	0.49	7

The concentration of residues was greatest in clear oil. Residues were also concentrated in peel and dried peel, both of which are used as cattle feed. Drying peel reduced the total residues by approximately 20%, but the loss of water was greater. Residues in the pulp and juice were negligible.

Apples. Apple trees were treated with a single soil broadcast application of Nema-cur 3 SC at a rate equivalent to 33.6 kg ai/ha (Chemagro, 1976e). Fruit were harvested 66 days after treatment and processed into juice and pomace.

Table 96. Effect of processing on residues in apples.

Sample	Residue, mg/kg	Processing factor*
Whole fruit	0.14	
Wet pomace	0.80, 0.57; average 0.68	4.85
Dry pomace	2.66, 2.31; average 2.48	17.7
Juice	0.11, 0.11	0.78

* Calculated from average residues in processed fractions.

Residues were concentrated in wet and dry pomace, both of which are used as animal feed. The residues in apple juice were lower than those in the whole apples. Chromatograms were provided but recoveries were not reported.

Grapes. Processing studies were conducted in the USA. The results are shown in Tables 97 and 98.

The soil around grape vines was treated with three sprays of Nema-cur 360 EC at a rate equivalent to 6.72 kg ai/ha (Mobay, 1981d, reports 69745 and 80080), the first at blooming, with 13-14 day intervals. The sprays were incorporated into the soil after application by hand sprayer.

In a processing trial in California (Grace, 1989, report 99611) Nema-cur 3 EC was applied twice at a 6-week interval at a rate equivalent to 25.2 kg ai/ha (5 times the normal rate) as band sprays with incorporation. Samples of grapes, grape juice and wet pomace were collected 7 days after the second spray.

Table 97. Effect of processing on residues in Thompson Seedless grapes, California, USA.

Year	Application			PHI, days	Sample	Residues, mg/kg	Report no.
	Form.	kg ai/ha	No.				
1981	360 EC	6.72	3	55	whole fruit	0.07	69745 ¹
				70		0.05	
				80		0.04	
				55	Raisins, sun-dried	0.07	
				70		0.01	
				80		0.02	
				55	raisin trash, sun-dried	0.77	
				70		0.11	
				80		0.06	

Year	Application			PHI, days	Sample	Residues, mg/kg	Report no.
	Form.	kg ai/ha	No.				
				70	raisin trash, oven-dried	<0.01	
			80	<0.01			
			55	0.12			
			70	0.12			
			80	0.10			
1981	360 EC	6.72	3	56	whole fruit	0.02	80080 ²
				73		0.01	
				56	raisins, sun-dried	0.03	
				73		0.01	
				56	raisin trash*	0.22	
				73		0.09	
				56	raisins, oven-dried	0.03	
				73		0.02	
				56	raisin trash, oven-dried	0.19	
				73		0.13	
1988	360 EC	25.2	2	7	whole fruit	0.02	99611 ³
					wet pomace	0.02	
					dry pomace	0.10	
					juice	0.02	

¹ 3 applications (14-day intervals) starting at blooming, by hand sprayer followed by incorporation. Plot size 74.2 m².

² 3 applications (49 and 36 days intervals) by broadcast spray to soil at 5-10 cm shoots, blooming and post-bloom. Plot size 39.9 m².

³ Two band sprays to soil near vines with incorporation at 42-day interval, 1st spray near maturity and 2nd spray at mature ripe fruit stage; plot size 17.8 m², sandy loam soil, pH 6.5-7.5, <1% C. Limit of determination = 0.01 mg/kg. Recoveries from fruit were F 102, 91%, FSO 76, 83% and FSO₂ 81, 102% at 0.01 mg/kg; F 82 %, FSO 75%, and FSO₂ 95% at 0.02 mg/kg; F 72%, FSO 76%, FSO₂ 94% at 0.1 mg/kg. Recoveries from juice were F 99%, FSO 86% and FSO₂ 91% at 0.1 mg/kg, from wet pomace F 74, 101%, FSO 75, 84%, FSO₂ 83, 90% at 0.05 mg/kg, and from dry pomace F 75% at 0.1 mg/kg.

Table 98. Effect of processing on residues in grapes and mean processing factors for processed fractions.

Sample	Residue, mg/kg	Processing factor ¹	Report no.
Whole fruit (PHI 55/56 days)	0.07, 0.02 (average 0.045)		69745, 80080
Raisins	0.07, 0.09, 0.03, 0.03 (average 0.055)	1.22	
Raisin trash	0.77, 0.12, 0.22, 0.19 (average 0.33)	7.33	
Whole fruit (PHI 7 days)	0.02		99611
Wet pomace	0.02	1	
Dry pomace	0.10	5	
Juice	0.02	1	

¹ (Mean residue in processed fraction) ÷ (mean residue in whole fruit)

The residues were concentrated in raisins and raisin waste, and in dry pomace after juicing and drying. The residues in juice and wet pomace did not differ from those in the whole fruit. Raisin waste and pomace are used as animal feed.

Pineapples. A processing trial was conducted in Wahiwa, Hawaii (Leslie, 1989a). One year-old pineapple plants were treated with 7 sprays of Namacur 3 EC at a rate equivalent to 16.8 kg ai/ha (5 times the recommended rate) at 14- to 30-day intervals. Samples of mature pineapples were collected 14 days after the last application and the fruit were processed into raw and dried bran and raw and canned juice. Crowns were separated from the whole fruit and analysed separately.

The processing involved removal of the crowns and washing the remaining fruit, followed by removal of the tops and butts. The fruit were then peeled and cored and the peel, tops and butts cut up and dried to form one component of dried bran. The cores, shells and fruit pulp were disintegrated, leaving raw juice and pomace. The juice was heated to 93°C and canned; the pomace was dried and

added to the dried tops and butts to form dried bran. Crowns are normally used for re-planting, but occasionally may also be used as an animal feed item, as is pineapple foliage. Pineapple bran (wet and dry) is a major animal feed commodity.

When pineapple fruit were held in frozen storage (0-23°C) for 635-826 days the periods of degradation ranges were 4-24% for fenamiphos, 0-29% for fenamiphos sulfoxide and 0-4% for fenamiphos sulfone. The samples in the processing trial were stored for a maximum period of 218 days before analysis. The results are shown in Tables 99 and 100.

Table 99. Residues in Cayenne pineapples and their processed fractions, Hawaii, 1988 (Leslie, 1989a).

Application			PHI, days	Sample	Residues, mg/kg	Processing factor	Report no.
Form.	kg ai/ha	No.					
360 EC	16.79	7	14	whole fruit	0.67		99609 ¹
				raw crowns	8.32	12.4	
				dried bran	1.68	2.51	
				raw bran	1.44	2.15	
				canned juice	0.80	1.19	
				raw juice	0.40	0.60	

¹ 7 sprays applied at 14-34 day intervals at early flowering, post-flowering, fruit formation, fruiting, flat eye and maturing fruit stages. Plot size 7.9 m², silty clay soil, pH 2, 5% C. Limit of determination = 0.2 mg/kg. Recoveries of F, FSO and FSO₂ from fruit = 110, 67 and 75% at 0.2 mg/kg, 110, 76 and 93% at 0.4 mg/kg, 83, 70 and 71% at 1 mg/kg, and from raw crowns 90 and 73%, 89 and 74%, and 78 and 106% at 2 mg/kg, 86, 120 and 116% at 5 mg/kg, 106, 92 and 73% at 10 mg/kg.

The residues were higher in the pineapple crowns and bran than in the whole fruit, although in trials at rates corresponding to GAP the residues in the crowns and bran were not consistently higher than those found in the whole fruit.

Peanuts. Nema-cur 15 GR was applied once to peanut crops, at a fivefold rate equivalent to 11.2 kg ai/ha (Leslie, 1989b). The band-over-row treatment was applied at the mid to late pegging stage, 60 days before final digging, by tractor-mounted equipment to a plot of 55.7 m². Inverted peanut plants were allowed to field-dry for 11 days before sampling threshed mature nuts in shell 71 days after treatment.

Processing involved drying and hulling to leave hulls, kernels and trash. The kernels were pressed to release the crude peanut oil, and the presscake yielded more crude oil by solvent extraction. The crude oil was separated into refined oil and soapstock, and the refined oil was hydrogenated and deodorised as for commercial use. The results are shown in Table 100.

Table 100. Residues in peanuts and their processed fractions, Georgia, USA, 1988 (Leslie, 1989b).

Application			PHI, days	Sample	Residues, mg/kg	Processing factor	Report no.
Form.	kg ai/ha	No.					
15 GR	11.2	1	71	kernels	0.01		99639 ¹
				meal	0.01	1	
				soapstock	<0.01	<1	
				crude oil	0.02	2	
				refined oil	<0.01	<1	

¹ Single band over row application at mid to late pegging. Plot size 55.7 m², sandy loam soil, pH 7, 0.7% C. Limit of determination = 0.01 mg/kg. Recoveries at 0.01, 0.02 and 0.05 mg/kg: fenamiphos 70-91% in kernels, 79-125% in meal, 78-99% in soapstock and 94-102% in crude oil; fenamiphos sulfoxide 82-110% in kernels, 72-106% in meal, 84-89% in soapstock and 77-114% in crude oil; fenamiphos sulfone 99-111% in kernels, 74-125% in meal, 77-84% in soapstock and 81-100% in crude oil.

Concentration of fenamiphos residues was only apparent in crude peanut oil, with no detectable residues in refined oil after treatment at an exaggerated rate.

Samples of peanut kernels were held in frozen storage (-23°C) for 223 days and the recovery of fenamiphos, fenamiphos sulfoxide and fenamiphos sulfone was measured. Fenamiphos sulfoxide levels showed a 3% reduction and there was no measurable loss of fenamiphos or fenamiphos sulfone. When vines were stored at -23°C for 155 days there was no apparent loss of fenamiphos.

Cotton. Nema-cur 15 GR was applied at planting at an exaggerated rate of 14.45 kg ai/ha with band incorporation to a plot of 470.6 m² (Leslie, 1989c). Cotton bolls were harvested after 153 days at normal maturity. The bolls were ginned and the gin trash and lint separated from the cotton seed. The seed was delinted, hulled and processed into meal, soapstock, hulls, and crude and refined oil. The refined oil was further bleached, hydrogenated and deodorised for commercial use. The results are shown in Table 101.

Samples of cotton seed fortified with fenamiphos, fenamiphos sulfoxide and fenamiphos sulfone at 0.1 mg/kg were held in frozen storage (-23°C) for up to 692 days. The average percentage decomposition after 687-692 days ranged from 19 to 26%. The samples in the processing trial were stored for a maximum period of 276 days before analysis.

Table 101. Residues in Delta Pine 50 cotton and its processed fractions, Mississippi, USA, 1988 (Leslie, 1989c).

Form.	Application kg ai/ha	No.	PHI, days	Sample	Residues, mg/kg	Processing factor	Reference
15 GR	14.45	1	153	cotton seed	0.01		Leslie 1989c ¹
				meal	<0.01	<1	
				soapstock	<0.01	<1	
				hulls	0.01	1	
				crude oil	0.02	2	
				refined oil	0.01	1	

¹ Plot size 471 m², sandy loam soil, pH 6, 1-2% C. Limit of determination = 0.01 mg/kg. Recoveries of F, FSO and FSO₂ at 0.01 mg/kg 98, 89%, 86, 108% and 97, 104% from seed; 102, 114%, 81, 111%, and 86; 89 and 90% from meal; 103, 108%, 90, 93, 106%, and 107 and 117% from hulls; 99%, 101% and 78% from soapstock; 67, 70, 71 and 82%, 110 and 76%, 77 and 77% from crude oil. Recoveries at 0.05 mg/kg were 71%, 90% and 103% from hulls, 82%, 73% and 84% from soapstock.

Residues were concentrated in crude oil, but reduced to their original level in the refining step.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

National monitoring data were reported by the governments of Australia and The Netherlands.

Table 102. Monitoring data for fenamiphos in The Netherlands (1994-1996).

Commodity	Samples	Samples below LOD (0.05 mg/kg)	Samples with residues <MRL	Samples with residues >MRL	Mean ¹ mg/kg	MRL, mg/kg
Fruiting vegetables	390	389	–	1 (0.06)	<0.05	0.05*
Brassica vegetables	3306	3306	–	–	<0.05	0.05*
Leafy vegetables and fresh herbs	368	367	–	1 (1.4)	<0.05	0.05*

¹ For samples with residues below the LOD, half the LOD is taken for the calculation of the mean.

Table 103. Monitoring data for fenamiphos in Australia (Qld, 1996-1998 and NSW 1989-1997).

Commodity	No. of analyses	No. of samples with fenamiphos residues
Qld (1996-1998)		
Mandarins, oranges	7	0
Apricots, nectarines, peaches	16	0
Grapes	14	0
Carambola	1	0
Bananas, longans, mango, pawpaw, passion fruit	33	0
Eschallot	1	0
Broccoli, Brussels sprouts, cabbage, cauliflower, red cabbage, cabbage sugarloaf	22	0
Cucumber, rockmelon, zucchini	20	0
Chickpeas	2	0
Butter lettuce, Chinese cabbage, long Chinese cabbage, lettuce, silver beet	54	0
Capsicum, cherry tomato, chillies, egg plant, egg tomatoes, sweet corn, tomatoes	98	0
Carrot	6	0
Celery	1	0
NSW (1989-1997)		
Citrus	152	0
Apples, pears	253	0
Cherry, nectarine, peach	282	0
Strawberry, grapes, blueberry	292	0
Avocado, banana, kiwifruit, lychee, mango, pawpaw	361	0
Onion	178	0
Broccoli, cabbage, cauliflower	367	0
Cucumber (Lebanese), pumpkin, rockmelon, zucchini	330	0
Bok choy, Chinese cabbage, lettuce, lettuce (hydroponic), silver beet	463	1 (lettuce, <1/2 MRL)
Capsicum, mushroom, sweet corn, tomato	479	0
Beans	151	0
Carrot, potato	519	0
Asparagus, celery	159	0

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported. The residue is defined as “sum of fenamiphos, its sulfoxide and sulfone, expressed as fenamiphos”, unless otherwise stated.

Country	MRL, mg/kg	Commodity
Argentina	0.05	Kiwifruit, pineapple, sugar beet
	0.1	Banana, beans, coffee, coffee bean (roasted), cucumber, grapes, melon, pepper (sweet), potato, tobacco, tomato
	0.2	Grapefruit, onion, orange
Australia	0.005*	Milk
	0.05*	Banana, brassica vegetables, celery, citrus fruit, edible offal (mammalian), eggs, cucurbits, ginger, grapes, leafy vegetables (except lettuce), meat (mammalian), onion, peanut, pineapple, poultry (edible offal of), poultry meat, sugar cane
	0.1	Mushrooms,
	0.2	Lettuce (head), root and tuber vegetables, strawberry
	0.3	Lettuce (leaf)
	0.5	Tomato
	1	Aloe vera
Belgium ¹	0.05*	Other plant commodities
	0.1	Banana, coffee, grapes, potato
	0.5	Citrus fruit
Brazil ¹	0.1	Cacao, coffee, potato, tomato
Chile	0.05*	Carrot, sugar beet, citrus fruit (except orange), other fruit
	0.1	Grapes, orange pulp,
	0.2	Potato, tomato
Cyprus	0.05	Beets, melon, nuts, watermelon
	0.1	Citrus fruit (without peel), grapes

Country	MRL, mg/kg	Commodity
	0.2	Potato, tomato
	0.5	Banana, citrus fruit
Germany	0.05	Other plant commodities
	0.1	Banana, coffee, grapes
	0.2	Potato, tomato
Israel	0.05	Corn/maize, melon, onion, peanut, wheat
	0.1	Banana,
	0.2	Cucumber, potato, summer squash, tomato
Italy	0.05	Orange, peach, strawberry,
	0.1	Aubergine, bean (without pods), melon, onion, potato, sugar beet, tobacco, tomato
Malaysia	0.005	Milk, milk products
	0.05	Citrus fruit, cucumber, ginger, grapes, meat, onion, peanut, pineapple, tomato
	0.1	Banana, coffee, mushroom, pepper (black), sweet potato
Mexico	0.02	Asparagus, peanut
	0.05	Cotton seed, soya
	0.1	Banana, Brussels sprouts, cabbage, coffee
	0.2	Cacao, potato
	0.25	Apple, peach
	0.3	Okra, pineapple,
	0.5	Garlic
	0.6	Grapefruit, lemon, lime (sour), orange, tangerine
	1	Grapes
Netherlands	0.05*	Other plant commodities
	0.1	Banana, coffee, coffee (infusion), grapes, potato, sweet potato
	0.2	Orange
New Zealand	0.2	Root and tuber vegetables, tomato
Paraguay ¹	0.2	Tomato
Portugal	0.2	Potato
South Africa	0.05	Banana, citrus fruit, cotton seed, grapes, guava, litchi, nectarine, onion, papaya, pea (garden), peach, pecan nut (shelled),
	0.05 E	Grapes, nectarine, peach
	0.1	Ginger, pineapple, tomato
	0.2	Potato
	10	Tobacco
Spain	0.02	Berry (wild), brassica vegetables, cacao, cereals, coffee, cola, forage crops and straw, fruit and vegetables (dried), herbs, hops, leafy vegetables, mushrooms, nuts, oilseeds, other berries and small fruit, other bulb vegetables, other citrus fruit, other fruiting vegetables, other legume vegetables, other tropical/subtropical fruit, pome fruit, potato, pulses, root and tuber vegetables, rubus fruit, spices, stem vegetables, stone fruit, strawberry, sugar cane, sweet corn, tea, tobacco
	0.05	Cucumber, garlic, melon, sugar beet
	0.1	Banana, bean (pods and/or immature), grapes, pepper (sweet), tomato
	0.2	Orange
Switzerland	0.5	Orange
Turkey	0.01	Milk
	0.3	Citrus fruit
Uruguay ¹	0.2	Potato, tomato
USA ¹	0.02	Cacao, peanut,
	0.02 R	Asparagus [Connecticut, Delaware, Minnesota, Massachusetts, Maine, New Hampshire, New Jersey, New York, Pennsylvania, Rhode Island]
	0.05	Cotton seed, soya, cattle fat, cattle meat, cattle meat by-products, goat fat, goat meat, goat meat by-products, horse fat, horse meat, horse meat by-products, milk, pig fat, pig meat, pig meat by-products, sheep fat, sheep meat, sheep meat by-products
	0.1	Aubergine, banana, Brussels sprouts, cabbage, grapes, raspberry
	0.1 R	Kiwifruit [California]
	0.25	Apple, cherry, peach
	0.3	Okra, pineapple
	0.3 F	Grapes, raisins
	0.4	Peanut hulls
	0.5	Garlic
	0.5 R	Chinese cabbage [California]
	0.6	Citrus fruit, strawberry

Country	MRL, mg/kg	Commodity
	0.6 R	Peppers (non-bell) [Georgia, Puerto Rico, California]
	1 R	Garden beet tops [New York, Pennsylvania, Illinois, Indiana, Michigan, Ohio]
	1 F	Grape pomace
	1.5 R	Garden beet root [New York, Pennsylvania, Illinois, Indiana, Michigan, Ohio]
	2.5	Citrus fruit molasses
	2.5 F	Citrus fruit pulp (dry)
	10	Pineapple bran
	25 F	Citrus fruit oil, refined

¹ Residue defined as sum of fenamiphos, its sulfoxide and sulfone.

E = export tolerance. F = Food additive tolerance. R = Regional tolerance.

APPRAISAL

Fenamiphos was first reviewed by the JMPR in 1974, with subsequent residues evaluations in 1977, 1978 and 1980. The compound was scheduled for periodic review at the 27th Session of the CCPR (ALINORM 95/24A Appendix IV). At the 30th (1998) Session it was noted that the TMDI based on existing CXLs slightly exceeded the revised ADI of 0.0008 mg/kg body weight allocated by the 1997 Meeting.

The manufacturer submitted a comprehensive data package in support of the existing CXLs for bananas, Brussels sprouts, cabbages, coffee beans, cotton seed, grapes, melons, oranges, peanuts, pineapples and tomatoes. Additional data were reported to estimate new maximum residue levels for apples, cherries, lemons, limes, grapefruit, onions, peaches and peppers.

Physicochemical data for fenamiphos indicate that the compound is moderately soluble in organic solvents (10-20 g/l) and highly soluble in toluene and 2-propanol (>200 g/l at 20° C).

Animal metabolism

The metabolism of fenamiphos was investigated in rats, a lactating goat, a dairy cow and laying hens.

In a series of four experiments, rats were dosed with [*phenyl*-1-^{13,14}C]fenamiphos as a single low i.v. dose, a single low oral dose, repeated low oral doses and a single oral dose at 10 times the low dose rate. Similar patterns of elimination and transformation were observed in all cases. Urine was the main route of elimination, with 93-100% of the radioactivity recovered within 48 hours after administration. In the faeces 1.5-3.8% of the dose was eliminated during the first 48 hours. The total radioactivity in the tissues, including the GI tract, was 0.045-0.23% of the administered dose; the radioactivity in the tissues of all the animals was below the limit of quantification and was not examined further.

The identified radioactivity in the excreta accounted for more than 93% of the total recovered radioactivity. The main metabolites in the urine and faeces were fenamiphos sulfoxide phenol (FSOP) accounting for 4-22% and its sulfate conjugate (FSOP-sulfate), 40-54%. The presence of these compounds indicates that a major transformation pathway in rats involves oxidation of the methylthio group, with cleavage of the isopropyl chain on the amine and of the phosphate ester function.

The most recent toxicological review of fenamiphos by the JMPR was in 1997. The findings in the rat studies were identical, *i.e.* fenamiphos was rapidly excreted, with >96% of the radioactivity excreted renally by 48 hours after dosing. At 48 hours, most of the residues in the tissues were below the limits of quantification. The main urinary metabolites were fenamiphos sulfoxide phenol sulfate, fenamiphos sulfoxide phenol, fenamiphos phenol sulfate and fenamiphos sulfone phenol sulfate.

In a dairy cow study, [U-*phenyl*-¹⁴C]fenamiphos sulfoxide was administered in a single dose of 0.8 mg/kg body weight. Blood, milk and urine samples were taken at hourly intervals and faeces were collected upon elimination. Four hours after administration, the animal was slaughtered and tissue samples including GI tract were taken for analysis. Approximately 88% of the administered dose was recovered, with 47% in the rumen contents, 39% in the urine, and 1.4% in the tissues.

Peak radioactivity in the blood of 0.24 mg/kg fenamiphos sulfoxide equivalents was observed 1 hour after dosing, with a steady decrease to 0.09 mg/kg 4 hours after administration. The main source of the recovered radioactivity in the blood was fenamiphos sulfoxide phenol at levels of 55 to 74%.

The radioactivity in the milk peaked in the 4 hour samples at a level of 0.061 mg/kg fenamiphos sulfoxide equivalents. The predominant radioactive components were fenamiphos sulfoxide phenol (37-40%) and fenamiphos phenol (<21%); 27-46% of the radioactivity remained unidentified.

In the urine, fenamiphos sulfoxide phenol was the main component of the total radioactive residue, at levels of 60-70% of the recovered radioactivity over the 4-hour period of the study.

Metabolites were identified in specific tissue samples, including muscle, fat, liver, kidney and heart. A large proportion of the radioactivity remained unidentified. Of the identified compounds, unchanged fenamiphos sulfoxide and fenamiphos sulfoxide phenol were the predominant compounds; fenamiphos was detected in the liver, fat, kidneys and heart.

[*Phenyl*-¹⁴C]fenamiphos was administered to a lactating goat at a dose of 1 mg/kg body weight for 3 days. Samples of blood, urine, faeces and milk were taken at regular intervals. Peak plasma radioactivity equivalent to 0.6 µg/ml was observed 0.25 hours after the first dose and decreased steadily to 0.12 µg/ml at 6 hours after administration. A half-life of 4.5 hours was calculated for the elimination of the radioactivity from plasma during the 1-6 hours after administration of the first dose.

The total recovered radioactivity was 65.5%, with urine accounting for 61% of the dose. Additional elimination in the faeces and milk accounted for 3.6% and 0.06% of the dose respectively. Radioactivity in edible tissues and organs totalled 0.3% of the dose.

The main radioactive metabolites in milk were fenamiphos phenol sulfate, fenamiphos sulfoxide phenol sulfate and fenamiphos sulfone phenol sulfate. Conjugate formation increases the water-solubility of the metabolites, so similar metabolite patterns are found in urine and in milk.

In edible tissues, the highest radioactivity was present in the liver and kidneys, at levels of 0.13 and 0.04 mg/kg fenamiphos equivalents respectively, or 0.09 and 0.04% of the recovered radioactivity. The main radioactive components in liver were fenamiphos sulfoxide and fenamiphos sulfoxide phenol. In kidney however, the main metabolites were fenamiphos sulfoxide phenol sulfate and fenamiphos sulfone phenol sulfate, again indicating that conjugation is a necessary transformation before the elimination of the metabolites. The tissues were re-analysed 8 and 24 months after the initial extractions. At 8 months similar results were found in both liver and kidney, but re-analysis of the liver samples at 24 months showed that the metabolites initially present had undergone some reductive transformation to fenamiphos and fenamiphos phenol sulfate.

Three types of muscle samples were analysed for metabolite composition: loin, flank and round. In round and loin muscle, the compounds present were fenamiphos sulfoxide, fenamiphos sulfoxide phenol sulfate and fenamiphos sulfone phenol sulfate. In flank muscle however, they were desisopropyl-fenamiphos sulfone, fenamiphos sulfoxide phenol and fenamiphos sulfone phenol sulfate, indicating incomplete transformation from fenamiphos sulfoxide to fenamiphos sulfoxide

phenol sulfate in flank muscle, as cleavage of the isopropyl and phosphate ester groups are two of the main processes of metabolic degradation of fenamiphos.

Laying hens were dosed orally with [*phenyl*-1-^{13,14}C]fenamiphos at 1 mg/kg body weight for 3 days. Blood, excreta and eggs were collected at regular intervals. The birds were killed 0.5 hours after the third dose.

Peak radioactive levels of 0.44 µg/ml were found in plasma 0.5 hours after the third dose, and decreased to 0.03 µg/ml at 24 hours. An elimination half-life of 4.3 hours was calculated from samples taken over a 24-hour period after administration. The total recovered radioactivity in individual birds ranged from 64 to 73% of the administered dose with excreta contributing to 60-70% of the total radioactivity. The TRR in eggs amounted to 0.03% and in tissues from 1.74 to 4.85%. The highest levels of radioactivity, 0.23, 0.61 and 2.2 µg/g fenamiphos equivalents, were present in the kidneys, liver and gizzard respectively.

The predominant metabolites in the tissues and eggs were fenamiphos phenol, fenamiphos sulfoxide phenol, fenamiphos sulfone phenol and/or their sulfate conjugates. Unchanged fenamiphos was also present in all tissues and eggs. The main pathways of transformation in hens include oxidation of the methylthio group and cleavage of the phosphate ester group, followed by conjugation of the resulting phenols.

In summary, the primary processes of metabolism in rats, goats, cows and hens involve oxidation of the methylthio sulfur, cleavage of the isopropyl group leaving a primary amine, cleavage of the phosphate ester group and conjugation of the resulting phenols leading to ease of elimination. Evidence of cleavage of the isopropyl group was found only in the goat and hen studies, where desisopropyl-fenamiphos sulfoxide was identified as an additional metabolite.

Plant metabolism

Studies on beans, tomatoes, carrots, cabbage and pineapples were reported. Application methods included spray, stem injection, soil treatment and uptake from solution. Snap beans were treated with [*ethyl*-¹⁴C] and [*methylthio*-³H]fenamiphos by stem injection (1 mg/plant) or soil treatment (6.7 kg ai/ha). After 4 weeks the plants were sampled and extracted. After soil treatment most of the radioactivity was recovered from the soil, with the remainder present in plant solids and as volatiles caught in acid and base traps. Less than 1% of the applied ¹⁴C was present in extracted plant material. After stem injection most of the radioactivity was trapped as volatile compounds or remained as unextracted plant material; 11% of the applied ¹⁴C was extracted. The main extractable ¹⁴C metabolites from both treatments were fenamiphos sulfoxide and fenamiphos sulfone.

In a subsequent study bean plants were treated by uptake from solution and stem injection. Uptake from solution resulted in a slower incorporation of the radiolabel into the plants, so the predominant extracted radioactive compound was parent fenamiphos instead of fenamiphos sulfoxide during the first 14 days.

[*Ethyl*-¹⁴C] and [*U-phenyl*-¹⁴C]fenamiphos were applied as soil treatments to tomatoes at a rate equivalent to 6.7 kg ai/ha, 20-30 days before fruit maturity. The distribution of radioactivity in the fruit was investigated up to 74 days after treatment. Most of the radioactivity was extracted, with less than 3% of the ¹⁴C present in insoluble fractions from the ring label but up to 36% from the ethyl label. This difference is presumably because different fragments of fenamiphos are incorporated after cleavage into plant components. The predominant radioactive components of the residue in the organic extracts were fenamiphos sulfoxide and fenamiphos sulfone, confirming that oxidation of the methylthio group followed by cleavage of the phosphate ester function are the main transformation pathways in plant metabolism.

Carrots were transplanted into soil treated with [*ethyl*-¹⁴C]fenamiphos at a rate equivalent to 10 kg ai/ha. Whole plants were harvested 53, 67 and 86 days after treatment. A large proportion of the radioactivity in both roots and foliage (34-66%) was present in unextracted solids and similar proportions of the radioactivity were extracted into aqueous and organic phases. Hydrolysis of the aqueous extracts showed that most of the water-soluble radioactivity was due to the phenol sulfoxide and phenol sulfone conjugates.

[*Ethyl*-¹⁴C] or [U-*phenyl*-¹⁴C]fenamiphos was applied as a soil treatment at a rate equivalent to 13.4 or 33.6 kg ai/ha before transplanting cabbage seedlings. Cabbage heads were harvested at intervals up to 90 days after treatment and samples of whole head, outer and inner leaves were analysed for radioactivity. The results indicated that as the crop matures the aqueous extractable radioactivity increases and the organic extractable radioactivity decreases. The total radioactivity and its distribution in outer and inner leaves after treatment with [*ethyl*-¹⁴C]fenamiphos were similar at the same sampling intervals. The main identified radioactive components in the organic extractable fractions were generally fenamiphos sulfoxide and fenamiphos sulfone. Enzymic hydrolysis of the aqueous fractions indicated that the water-soluble metabolites were glucoside conjugates of phenol derivatives of fenamiphos. Acid digestion of the insoluble fraction yielded the metabolites found in the organic phase, *i.e.* fenamiphos sulfoxide and fenamiphos sulfone.

In a series of five experiments, pineapple plants were treated with [*ethyl*-¹⁴C] or [U-*phenyl*-¹⁴C]fenamiphos either as a soil treatment, spray or stem injection. Most of the radiolabel from the soil treatment was present in the soil, with a gradual increase in the radioactivity in the pineapple foliage up to 90 days after treatment. As in cabbages and carrots, there was an increase in the radioactivity in the aqueous and insoluble fractions with time compared to the proportion of organosoluble radioactivity, irrespective of the application method. The predominant radioactive components were fenamiphos sulfoxide and sulfone, with lower levels of the corresponding phenols. Similar metabolite patterns were observed after spray treatment and stem injection. Enzymatic hydrolysis of the aqueous fractions yielded 14-34% of the applied radioactivity as fenamiphos sulfoxide phenol and 6-14% as fenamiphos sulfone phenol after stem injections and spray applications.

Crop rotation studies were conducted with various crops including cereals, a root crop, an oilseed crop and leafy vegetables after treatment of the soil at 7.6 kg ai/ha. The results with the different crops were similar and in agreement with the plant metabolism studies, *i.e.* the radioactivity extracted into aqueous fractions and remaining in the plant solids increased with time owing to conjugation of fenamiphos sulfoxide and sulfone phenols. The soil radioactivity was measured at each cropping interval and the patterns of degradation observed in the soil and rotational crops were similar.

The maximum residues of fenamiphos sulfoxide and fenamiphos sulfone (as fenamiphos equivalents) were 0.08-6.55 mg/kg and 0.04-5.41 mg/kg respectively in the crops and crop fractions investigated after the first rotation (30 days). In the second rotation (120 days), fenamiphos sulfoxide and fenamiphos sulfone residues ranged from 0.02 to 2.82 mg/kg and 0.01 to 2.90 mg/kg respectively, and in the third rotation (269 days) the corresponding residues were <0.01-0.39 mg/kg and <0.01-0.62 mg/kg.

The additional metabolites desisopropyl-fenamiphos sulfoxide and desamino-fenamiphos sulfoxide were both identified in the rotational studies and provide evidence that cleavage of the isopropyl group and the resulting amino group are among the metabolic transformations that occur in plants.

In another crop rotation study, unlabelled fenamiphos was applied to the soil at a rate equivalent to 6.72 kg ai/ha and incorporated after application. Rotational crops of wheat, sorghum, turnips, spinach and mustard greens were planted 1, 4 and 8 months after the soil treatment. Soil samples were taken immediately after treatment and at planting and harvest of the rotational crops. The residues were <0.01 mg/kg at 4 months plant back in all samples except spinach leaves and

sorghum forage and straw. The residues in sorghum forage were 0.44 and 0.68 mg/kg and in straw 0.02 mg/kg at one site, but <0.01 mg/kg at another site. After 8 months plant back the residues were <0.01 mg/kg in all sorghum plant fractions. The residues in spinach leaves were 0.03 mg/kg at 4 months plant back.

In summary, the conclusions from the plant metabolism studies were that fenamiphos sulfoxide and fenamiphos sulfone are the main metabolites formed after the application of fenamiphos by various methods. In crops with a substantial period from treatment to harvest fenamiphos sulfoxide phenol and fenamiphos sulfone phenol are also formed, as is apparent by the change in the extraction characteristics of the radioactivity with time. Overall, the metabolites of fenamiphos in plants and animals are similar and the existing definition of the residue as “sum of fenamiphos, its sulfoxide and sulfone, expressed as fenamiphos” is appropriate.

Environmental fate

The photodegradation of [U-*phenyl*-¹⁴C]fenamiphos on soil proceeds via first order kinetics with measured half-life values of 1.6 hours under laboratory conditions (Hg vapour lamp) and 2.7 hours in natural sunlight. The main photolytic products are fenamiphos sulfoxide and fenamiphos sulfone.

The adsorption/desorption properties of fenamiphos were investigated in four types of soil ranging from sand to clay loam. Measured K_{oc} values for fenamiphos indicated low mobility in the sandy soil and medium mobility in sandy loam, silt loam and clay loam (K_{oc} 150-500 medium mobility; 500-2000 low mobility). In another study, the adsorption / desorption characteristics of fenamiphos were investigated in 16 soils from different geographic locations, ranging from cool/moderate to sub-tropical climates. The highest adsorption capacity was measured in a soil with high clay and silt contents. In all the soils higher adsorption constants were found for fenamiphos than for fenamiphos sulfoxide phenol or fenamiphos sulfone phenol. Silt loam adsorbed a higher proportion than clay loam of both fenamiphos sulfoxide and fenamiphos sulfone.

Degradation half-lives of <30 days were reported for fenamiphos in aerobic conditions, with fenamiphos sulfoxide and fenamiphos sulfone the main degradation products. The half-life in anaerobic conditions was 87.9 days, with fenamiphos sulfoxide the main degradation product. In aerobic soil degradation studies conducted in 16 soils from different geographic locations, half-lives were reported as less than 15 days at 22° C. In an outdoor degradation study, the half-life of fenamiphos was reported as 19.9 and 18.2 days in predominantly sandy soils at two sites in California.

In leaching experiments with aged residues, [¹⁴C]fenamiphos was applied to soil and the mixture aged for 63 days. The treated soil was applied to columns containing a sandy loam and a silt loam, which were eluted for 48 hours with water. Less than 1% of the applied radioactivity was found in the eluates; up to 16% was collected as volatiles. Fenamiphos was mainly converted to fenamiphos sulfoxide in the aged soil.

In two field dissipation studies degradation half-lives of 15 and of 15.9 days were reported for fenamiphos. Soil core samples were taken after 1 and 2 sprays of fenamiphos at 12.3 kg ai/ha. The maximum residues of fenamiphos were 1.5 and 2.7 mg/kg and 2.0 and 2.5 mg/kg after the 1st and 2nd sprays respectively, and of fenamiphos sulfoxide 3.3 and 4.1 mg/kg after the first applications and 2.0 and 2.5 mg/kg after the second; they decreased to <0.01 mg/kg at 90, 93 and 254, 361 days after the 1st and 2nd sprays, at all soil depths examined (down to 61 cm). The results indicate that fenamiphos is degraded rapidly, whereas fenamiphos sulfoxide dissipates at a relatively rapid rate after 1 spray and more slowly after 2 sprays.

Calculated half-lives for the hydrolysis of fenamiphos in buffer solutions at pH 3 and 9 were 3-10 and 22-230 days respectively. In pH 3 buffer solution, the main hydrolysis product was deaminated fenamiphos, with fenamiphos phenol and deaminated fenamiphos phenol present below 10% of the applied concentration. At pH 9 however, the main hydrolysis products were fenamiphos

phenol, fenamiphos sulfoxide phenol and fenamiphos sulfoxide, presumably owing to base hydrolysis of the phosphate ester group. At elevated temperatures of 60, 70 and 80° C the half-lives ranged from 1.7-9.8 days, 14-67 days and 5-70 hours at pH 4, 7 and 9 respectively.

In sterile solutions at pH 5, 7 and 9 which were kept in the dark, the calculated half-lives for hydrolysis ranged from 235-301 days, with the longest at pH 7.

Aqueous solutions of fenamiphos in phosphate buffer were irradiated under laboratory conditions (Hg lamp) and samples were analysed at regular intervals up to 24 hours. The half-life was calculated as 3.6 hours and the main photolytic products at 24 hours were fenamiphos sulfoxide and fenamiphos sulfonic acid phenol.

In summary, the degradation of fenamiphos in soil and water proceeds via oxidation of fenamiphos to fenamiphos sulfoxide and fenamiphos sulfone, so the products formed are similar to the metabolites formed in plants and animals. An additional product detected was fenamiphos phenol sulfonic acid, formed by oxidative demethylation of the methylthio group and hydrolysis of the phosphate ester.

Methods of residue analysis

Analytical methods for the determination of fenamiphos and its metabolites in various plant substrates, animal tissues, soil and water are based on the published method of Thornton (1971). The method was originally validated for citrus peel and pulp, pineapple fruit, bran and forage, peanut kernels, hulls and vines, and tobacco. Several modifications were subsequently reported. The basic procedure involves homogenization of the sample, filtering, and partitioning the solution with methyl chloride or other organic solvents. The organic extract is evaporated to dryness, and the residue is redissolved in acetone and oxidised with KMnO_4 solution. The oxidised residues are partitioned again into methyl chloride, which is evaporated before dissolution in acetone for quantification by GLC with an FPD in the phosphorus mode. Total residues of fenamiphos, fenamiphos sulfoxide and fenamiphos sulfone are quantified in a single sulfone peak. The limits of determination in various crops are reported as 0.01-0.1 mg/kg

Fenamiphos is included in multi-residue enforcement methods which were provided by the governments of Australia and The Netherlands. In the *Official Methods of Analysis in The Netherlands*, fenamiphos is quantified at a limit of 0.05 mg/kg in various crops; recoveries were reported for lettuce. In fatty foods the limit of determination is also 0.05 mg/kg. Inclusion of the sulfoxide and sulfone metabolites was not mentioned. Fenamiphos is quantified at a limit of 0.02 mg/kg in crops in the method provided by the Australian government.

Analyses of animal tissues involve quantification of total residues including fenamiphos sulfoxide, sulfone and sulfoxide phenol (FSO, FSO_2 , and FSOP). The metabolites desisopropyl-fenamiphos and its sulfoxide and sulfone (DIF, DIFSO and DIFSO_2) are quantified in an additional peak containing the methylated residues. The work-up procedures for animal tissues and milk are similar to those for crops, but CH_3CN is used in the partitioning steps before oxidation. Reported limits of quantification in milk and tissues are 0.005 and 0.01 mg/kg respectively.

The limits of quantification in soil and water are 0.01 mg/kg and 0.1 $\mu\text{g/l}$ respectively. An electrospray MS method was developed for the determination of fenamiphos and its degradation products in soil. Deuterated fenamiphos is introduced into the soil sample as an internal standard before work-up, followed by extraction in CH_3CN and analysis by LC-MS-MS. Aliquots are then analysed by HPLC/MS to determine fenamiphos and its sulfoxide and sulfone. The limit of determination for the individual compounds is 0.01 mg/kg.

Recoveries were determined by fortification with fenamiphos alone or a mixture of fenamiphos and its sulfoxide and sulfone.

The stability of residues was determined in stored samples of a number of crops including asparagus, banana, cotton seed (seed, meal, hulls and oil), garlic, and grapes (berries, juice, wet and dry pomace and raisins). Samples were fortified with a mixed standard composed of fenamiphos, FSO and FSO₂ at 1 mg/kg each (3 mg/kg total) and held in frozen storage ($\leq -5^{\circ}\text{C}$) for up to 18 months. Some decrease of total residues (<10%) was observed in garlic and grape pomace after 12 months. At 18 months <10% decrease was found in most commodities and crop fractions except raisins and cotton seed hulls, which had decreased by <20%. The Meeting agreed that a decrease of <20% should not be considered significant, and that residues in the commodities examined were stable when stored frozen for 18 months.

In a study of the storage stability of residues in animal tissues, extracts of cattle fat, kidney, liver and muscle were fortified separately with 1 mg/kg fenamiphos, DIF, FSO, DIFSO, FSO₂ or DIFSO₂, and milk with 1 mg/kg fenamiphos, FSO or FSO₂. Tissues and milk were stored at -25°C for up to 2 and 3 months respectively. The results showed that fenamiphos, FSO and FSO₂ were stable in milk for 61 days, but fenamiphos was unstable in fat, liver, kidney and muscle, and was degraded within 83 days. As fenamiphos would have been converted to its sulfoxide and sulfone and the analytical method determines total residues as fenamiphos sulfone, the total fenamiphos residues in tissues are considered to be stable.

Use pattern

Fenamiphos is registered in many countries as a nematicide. Numerous labels from registered products were submitted by the manufacturer. For many crop uses, fenamiphos is applied pre- or post-planting as a soil treatment, in-furrow spray or by drip irrigation. To established crops, it is applied as a spray to individual plants or trees, with repeat treatments if necessary. Both fenamiphos sulfoxide and fenamiphos sulfone also exhibit nematocidal activity.

Supervised trials

Data were provided in support of the existing CXLs for bananas, Brussels sprouts, cabbages, carrots, coffee beans, cotton seed, grapes, melons, pineapples, potatoes and tomatoes. New data were reported on apples, cherries, lemons, limes, grapefruit, onions, peaches, peppers and zucchini.

The residues in the supervised crop trials were determined as the sum of fenamiphos, its sulfoxide and sulfone, expressed as fenamiphos.

Root, tuber and bulb vegetables. There are existing CXLs for fenamiphos in carrots, potatoes and sweet potatoes.

Trials on carrots were conducted in Australia, Italy and Spain. Fenamiphos was applied to soil before sowing in all the trials. The registered use pattern in Australia is for a single application at a maximum rate of 9-9.6 kg ai/ha and a PHI of 84 days. In Italy, a single application of 15 kg ai/ha is registered with a PHI of 90 days. The Spanish trials were not directly comparable to a registered use in Spain and were evaluated against GAP in Italy. Where higher rates of application gave residues below the limit of detection the results were also used in the estimation of the maximum residue level. The residues from trials according to GAP ranged from <0.02 to 0.08 mg/kg. The residues in rank order were <0.02 (8), 0.02, 0.024, 0.027, 0.05, 0.06 (2), 0.07 and 0.08 mg/kg. The Meeting estimated a maximum residue level of 0.2 mg/kg (the existing CXL) and an STMR of 0.02 mg/kg. (HR = 0.08 mg/kg).

Supervised trials on potatoes were conducted in Australia and Spain. Registered uses in Australia allow single applications at a maximum rate of 10 kg ai/ha with a PHI of 84 days. In Spain single applications of 8-10 kg ai/ha are allowed with a PHI of 120 days. The treatments were applied pre-planting. The residues in the tubers were <0.01-0.17 mg/kg in six trials. The Meeting considered

that there were insufficient data to confirm the existing CXL of 0.2 mg/kg and recommended its withdrawal.

As no data were provided for sweet potatoes, the Meeting recommended withdrawal of the existing CXL of 0.1 mg/kg.

Trials on onions were conducted in Australia and South Africa. The product was applied to soil 4 or 5 days before sowing. The registered use in Australia is for a single application at 9.7 kg ai/ha with a PHI of 84 days. In South Africa the maximum rate is 3 kg ai/ha with a PHI of 80 days. Some samples in one of the South African trials were not fully mature although the reported PHIs in that trial were longer than in the other trials. The residues in onion bulbs ranged from <0.01 to 0.05 mg/kg in trials which complied with GAP. The Meeting considered that there were insufficient data to estimate a maximum residue level.

Brassica vegetables. There are existing CXLs for broccoli, Brussels sprouts, cabbages and cauliflower.

Data from 7 trials in the USA were provided for Brussels sprouts. The registered use pattern allows single applications of 6.7 kg ai/ha at planting with no specified PHI. The residues in 6 trials were <0.01 mg/kg; in the other trial a higher rate of 10 kg ai/ha was employed which resulted in residues of 0.02 mg/kg. The Meeting estimated a maximum residue level of 0.05 mg/kg and an STMR of 0.01 mg/kg, and recommended the withdrawal of the existing CXL of 0.05* mg/kg (HR = 0.01 mg/kg).

Trials on cabbages were conducted in Australia and the USA. The registered use pattern in Australia for crucifers is a single application up to 7 days before planting at a rate of 9-11 kg ai/ha and no specified PHI. GAP in the USA allows single applications at 6.7 kg ai/ha with no specified PHI. The residues in cabbage heads were <0.01-0.05 mg/kg in 12 US trials which complied with GAP. The residues in all the trials according to GAP in rank order were <0.01 (10), 0.01, <0.02, 0.02 (3) and 0.05 mg/kg. The Meeting recommended the withdrawal of the existing CXL of 0.05* mg/kg and estimated a maximum residue level of 0.05 mg/kg and an STMR of 0.01 mg/kg (HR = 0.05 mg/kg).

In the US trials, wrapper leaves and field trash were also analysed for fenamiphos residues. The residues in the wrapper leaves were slightly higher than those levels found in the cabbage heads.

No data in support of the existing CXLs for broccoli and cauliflower were submitted. No information on specific GAP for cauliflower or broccoli was provided, although there are registered uses for brassicas in Costa Rica and crucifers in Australia. The Meeting agreed to recommend withdrawal of the existing CXLs for broccoli and cauliflower.

Fruiting vegetables. Supervised trials on peppers were conducted in Italy, Spain and Portugal in glasshouses and under field conditions. Applications ranged from several days before planting in the field trials to drip irrigation at flowering or early stages of fruiting in the glasshouse trials. Data from trials with an encapsulated formulation (CS) have been recorded in the Tables, but registration of the product is only pending in Spain and Portugal and the data were therefore not used in the estimation of the maximum residue level and STMR. Current registered uses in Spain are single applications at planting at rates of 5-10 kg ai/ha with a PHI of 90 days.

The residues in peppers ranged from <0.02 to 0.35 mg/kg after treatment with GR and EC formulations, and from <0.02 to 0.31 mg/kg after treatment with the CS formulation. The range of residues was similar from treatment with the registered formulations and the CS product. The residues in rank order were <0.02, <0.05, 0.05 (2), 0.06 (2), 0.26 and 0.35 mg/kg. A maximum residue level of 0.5 mg/kg and an STMR of 0.055 mg/kg were estimated. (HR = 0.35 mg/kg).

Sixteen field and 11 glasshouse trials on tomatoes were conducted in Australia, Brazil, Italy, Portugal, South Africa and Spain with GR, EC, EW and CS formulations. In the field trials fenamiphos was applied at or shortly after planting, and in the glasshouse trials application was by drip irrigation pre-flowering or at early fruit formation. The registered use in Australia is for single applications at a maximum rate of 11 kg ai/ha with no specified PHI. In Brazil a single application at 3-4 kg ai/ha with a PHI of 90 days is registered. The Italian use pattern is for single applications at 10-15 kg ai/ha with a PHI of 20 days. In Portugal, a maximum of 3.4 kg ai/ha may be applied in a single treatment with a PHI of 20 days. On South African labels, application rates are expressed in g ai/m, allowing a maximum of 1 g ai/m and unspecified PHI. Spanish labels for the GR and EC products specify single applications at rates of 5-10 kg ai/ha with a PHI of 90 days.

The residues in tomatoes were <0.02-0.30 mg/kg after treatment with the GR and EC formulations and <0.02-0.15 mg/kg after treatment with the CS formulation. Although the CS product is a pending registration, the residues from the CS trials were within the range found for the currently registered formulations. The residues from the CS formulation were not considered in the estimation of the maximum residue level or STMR. The residues in rank order were <0.02 (4), <0.05 (5), <0.1, 0.15, 0.17, 0.27 and 0.30 mg/kg. The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.05 mg/kg, and recommended withdrawal of the existing CXL of 0.2 mg/kg (HR = 0.30 mg/kg).

Four glasshouse trials on zucchini were conducted in Italy in which the CS formulation was applied at the 3 to 5 leaf stage of growth. The proposed GAP for the CS product in Spain allows single applications at 10 kg ai/ha with a PHI of 90 days. The residues were <0.02 mg/kg in all the trials. As registration of the CS product is pending and there were no trials with registered formulations, a maximum residue level for zucchini could not be estimated.

Thirteen field and 9 glasshouse trials on melons were conducted in Australia, Brazil, Guatemala, Mexico and Italy with GR, EC and CS formulations. Application timings ranged from 14 days before sowing to flowering. In many trials residues were determined in the whole fruit and pulp; in some the residues in peel were reported separately. Registered use patterns in Australia are for single applications at a maximum rate of 9.6 kg ai/ha and no specified PHI. In Brazil, labels recommend single applications at 4 kg ai/ha with no specified PHI. The 10, 12 and 15 GR products are registered in Guatemala with single applications at a maximum rate of 5.1 kg ai/ha and a PHI of 60 days. In Italy the 5 GR product may be applied at rates of 5-10 kg ai/ha with a PHI of 20 days. In Spain GR and EC products are registered with a maximum rate of 10 kg ai/ha and a PHI of 90 days. Registration of the CS formulation is pending. The trials in Mexico were evaluated against the registered uses in Guatemala.

The residues in the whole fruit after treatment with the GR and EC formulations were <0.01- <0.05 mg/kg. In trials with the CS formulation residues in the whole fruit were <0.02-0.03 mg/kg. The residues in the pulp were below the reported limit of detection or determination in all the trials. The residues in the whole fruit in rank order were <0.01 (6), <0.02 (3) and <0.05 (4) mg/kg. The Meeting estimated a maximum residue level of 0.05* mg/kg, the same as the existing CXL. The residues in the pulp in rank order were <0.01 (4) and <0.02 mg/kg. The Meeting estimated an STMR of 0.02 mg/kg for melon pulp (HR = 0.02 mg/kg).

Two trials on watermelons were carried out in Italy, where the registered use pattern is a single application of a GR product at 5-10 kg ai/ha with a PHI of 20 days. Sampling in the trials was at fruit maturity, 85-109 days after treatment, so the PHI was not observed although the application was made up to 20 days before planting. The residues in the whole fruit and pulp were below the limit of determination of 0.02 mg/kg. The Meeting agreed that as there were few trials for watermelons and Italian GAP for melons and watermelons is identical, the trials on melons could be used to support a recommended MRL for watermelon. A maximum residue level of 0.05* mg/kg was therefore estimated for watermelon, with an STMR of 0.02 mg/kg (HR = 0.02 mg/kg).

Citrus fruits. Many of the trials on citrus fruit were conducted in the USA at excessive treatment rates. In trials on grapefruit, lemons, limes and oranges, soil applications were made at rates of 2.4 and 4 times the registered label rates. The registered use patterns for citrus fruits in the USA prescribe single applications at a maximum rate of 8.4 kg ai/ha with a PHI of 30 days. In many trials, residues were reported in the whole fruit, pulp and peel.

Supervised trials on grapefruit in the USA were at rates equivalent to 4 times the maximum label rate. The residues were <0.01-0.29 mg/kg and <0.01-0.08 mg/kg in the whole fruit and pulp respectively, in samples taken at PHIs of 30-243 days. As the trials did not reflect GAP in the USA and application at excessive rates resulted in detectable residues, the data could not be used to estimate a maximum residue level for grapefruit.

Seven trials on lemons were conducted in Australia, South Africa and the USA. Again in the 5 US trials, the rates were equivalent to 2.6 or 4 times the maximum registered rate. The registered use pattern in Australia is single applications at 30 kg ai/ha with no specified PHI. In South Africa, GAP allows a maximum rate of 12 kg ai/ha or 2 g ai/m² to be applied with a PHI of 150 days. The residues from the Australian and South African trials were <0.05 mg/kg in the whole fruit, pulp and peel. The residues in the US trials were <0.01-0.44 mg/kg in the whole fruit and <0.01 mg/kg in the pulp. The Meeting considered that 5 of the 7 trials were not according to GAP and could not be used to estimate a maximum residue level for lemons.

In two US trials on limes the rates of application were 4 times the maximum GAP rate. The residues in the whole fruit 147 days after treatment were below the limit of detection of 0.01 mg/kg.

Trials were conducted in Australia, South Africa and the USA in support of the existing CXL of 0.5 mg/kg for oranges. All the trials in the USA were at rates of 2.6 or 4 times the maximum registered rate. GAP in Australia and South Africa is identical to that for lemons. The residues in the whole fruit were <0.02-0.08 mg/kg in the trials in Australia and South Africa. In the US trials the residues were <0.01-0.17 mg/kg in the whole fruit and <0.01-0.02 mg/kg in the pulp. As most of the trials were in the USA at excessive rates they could not be used to estimate a maximum residue level. The Meeting therefore recommended the withdrawal of the existing CXL for oranges.

In summary, the Meeting concluded that as exaggerated treatments were applied in most of the trials on citrus fruits and residues above the limit of determination were found at varying intervals after treatment, no maximum residue levels could be estimated.

Pome fruits. Numerous trials on apples were conducted in the USA where the registered use rates are 5.4-8.2 kg ai/ha with a PHI of 72 days. The trials were all at a rate of 22.4 kg ai/ha, 2.6 times the maximum rate. The residues in the whole fruit in all 33 trials were below the limit of detection of 0.01 mg/kg; the reported limit of determination was 0.05 mg/kg. Although the trials were not in accord with US GAP, the Meeting considered that as there were no detectable residues in any of the trials after exaggerated treatments, the data could be used to estimate a maximum residue level of 0.05* mg/kg and an STMR of 0.01 mg/kg (HR = 0.01 mg/kg).

Stone fruits. Nineteen supervised trials on cherries were conducted in the USA, 16 at 2.7 and 3 at 1.7 times the maximum registered rate (8.2 kg ai/ha with a PHI of 45 days). The residues in the whole fruit ranged from <0.01 to 0.18 mg/kg at PHIs of 45-52 days after treatment. The trials were not in accord with GAP in the USA or other countries, so no maximum residue level could be estimated.

Twenty nine trials on peaches were conducted in the USA and one in Italy at rates equivalent to 2.7 and 1.7 times the maximum national registered rates respectively. The residues in the whole fruit were <0.01-0.16 mg/kg at the earliest sampling intervals, after treatment at stages from pre-flowering to immature fruit. As the trials did not comply with GAP in either Italy or the USA, the Meeting could not estimate a maximum residue level.

Berries and other small fruits. Supervised trials on grapes were conducted in Chile, Mexico, South Africa and the USA. In the US trials, residues were determined in raisins and raisin trash as well as grapes. GAP in Chile specifies rates of 6-8 kg ai/ha (in-furrow) or 2.8-4.8 kg ai/ha (drip irrigation), with a PHI of 45 days. Registered use patterns in Mexico are for 4-6 kg ai/ha and no specified PHI. In South Africa, a maximum rate of 1 g ai/m² is registered with a PHI of 100 days, and in the USA 3.3-6.5 kg ai/ha with a PHI of 2 days.

The Chilean and US trials were at GAP rates and above, up to 3 and 1.5 times the maximum rates in Chile and the USA respectively. Sampling was at longer intervals than the GAP PHI in many of the US trials, but the earliest sampling is considered to accord with GAP if mature fruit were collected. The residues in the grapes were <0.01-0.09 mg/kg from a total of 49 trials. The residues from trials which were considered to comply with GAP in rank order were <0.01 (11), 0.01 (6), 0.02 (7), 0.03 (5), <0.05 (4), 0.07 (2) and 0.09 mg/kg. Although analytical recoveries of fenamiphos were determined at concentrations of 0.01, 0.02, 0.03 and 0.05 mg/kg, recoveries of all components of the defined residue (fenamiphos, FSO and FSO₂) were determined only at concentrations of 0.05 and 0.1 mg/kg, so the validated limit of determination in grapes is 0.05 mg/kg. The Meeting estimated a maximum residue level of 0.1 mg/kg, confirming the existing CXL, and an STMR of 0.02 mg/kg (HR = 0.09 mg/kg).

Tropical fruits - inedible peel. Trials on bananas were conducted in Australia, Brazil, the Canary Islands (Spain), Costa Rica and the Windward Islands. Trials in the Canary Islands and the Windward Islands were evaluated against GAP for Spain and Costa Rica respectively. Rates at or above the registered use patterns were applied in the trials. In some cases rates of application were expressed both as kg ai/ha and as g ai/plant to allow for different cropping densities in different regions. For example the trials in Costa Rica allowed for cropping densities ranging from 416 to 833 plants/ha, and those in the Windward Islands for 412-1785 plants/ha. Although trials with the CS product were reported, the results were not used in the estimation of the maximum residue level or STMR because registration of the product was pending. The residues were determined separately in the pulp and peel or on a whole fruit basis; where residues in the pulp and peel were below the limit of detection or determination, the residues in the whole fruit were considered to have the same value.

The residues in the whole fruit in rank order were <0.01 (7) and <0.02 (3) mg/kg. The Meeting estimated a maximum residue level of 0.05* mg/kg, based on the routine limit of quantification of fenamiphos, and recommended withdrawal of the existing CXL of 0.1 mg/kg. The residues in banana pulp in rank order were <0.02 (6) and <0.025 (3) mg/kg. An STMR of 0.02 mg/kg was estimated (HR = 0.025 mg/kg).

Trials on pineapples were mainly in Hawaii, with a few trials in Australia and Puerto Rico. Registered use patterns in Australia allow a maximum of 5 applications at 2.4 kg ai/ha to the main plant crop and ratoon crop and a maximum of 2 applications at 4.8 kg ai/ha to the ratoon crop alone; PHIs are not specified. Registered labels in the USA specify two use patterns, one for Puerto Rico and the other for Hawaii. For Puerto Rico, a pre-plant application of 10 kg ai/ha with additional applications at 5.4-9.8 kg ai/ha post-planting and to the first ratoon crop, with total applications of 20 kg ai/ha per ratoon crop are recommended, with a PHI of 225 days for the post-planting applications. In Hawaii the total applications are 26.2 kg ai/ha per plant crop and 9.8 kg ai/ha per ratoon crop, made up of a pre-planting application of 9.8 kg ai/ha for the plant crop and post-planting sprays at 0.5-3.3 kg ai/ha. A PHI of 30 days is recommended for the post-planting applications.

In the Hawaiian trials the rates were equivalent to 2.3 times the pre-planting application, 1.2-2.3 times the total plant crop treatment and up to 2.3 times the ratoon crop treatment. The residues were <0.01-0.03 mg/kg in the whole fruit and <0.01-0.05 mg/kg in the pulp. Residues were determined in bran, foliage, forage, crowns and stumps in addition to whole fruit and pulp.

In two Puerto Rican trials, twice the pre-plant rate and up to 1.1 times the post-plant rates were applied to the plant crop.

The residues in the whole fruit in the 3 Australian trials were <0.01 mg/kg, but only 1 trial complied with GAP. The residues in the pulp were <0.01 mg/kg in the Puerto Rican trials. Although the residues in the Hawaiian trials were from exaggerated treatments, the number of post-planting applications to the plant and ratoon crops are not always specified on the label, and multiple applications may be required at these stages depending upon pest pressure. Total application rates per plant crop and ratoon crop are indicated however. The Meeting considered that although treatments were exaggerated the residues in the whole fruit and pulp were below the limit of detection in most of the trials, so the results were acceptable for estimating a maximum residue level and an STMR. The residues in the whole fruit in rank order were <0.01 (8), 0.02 and 0.03 mg/kg, and in the pulp <0.01 (26), 0.01 (2), 0.02 (2), 0.05 and 0.14 mg/kg. The Meeting included the figure of 0.14 mg/kg in the data set, as the conditions of the trial (PHI, application rate and application timing) did not differ from other instances where residues in the pulp were <0.01 mg/kg. The validated limit of determination in pineapple pulp, bran, foliage and crowns was 0.05 mg/kg. The Meeting estimated a maximum residue level of 0.05* mg/kg, confirming the existing CXL for pineapples, and an STMR of 0.01 mg/kg for pineapple pulp (HR = 0.14 mg/kg).

The residues in wet bran were <0.01-0.25 mg/kg, in dry bran <0.01-2.3 mg/kg and in unspecified bran <0.01-0.13 mg/kg.

Oilseeds. Supervised trials on peanuts were conducted in the USA and South Africa. The residues were determined in nuts, shells, foliage and vines. In the US trials, up to 7 times the maximum registered rate was applied and nuts were sampled at normal harvest. Residues in all samples of nuts were below the limit of detection or determination. The residues in vines were <0.01-3.19 mg/kg at rates of 4.7-7 times the label rate and PHIs of 94-154 days after planting. GAP in the USA allows single applications at rates of 1.6-2.9 kg ai/ha, with no specified PHI. In South Africa, the registered use pattern is a single application at 1.6-3.2 kg ai/ha and a PHI of 63 days. Although exaggerated treatments were applied, no residues were detectable in the nuts and the Meeting therefore concluded that the existing CXL of 0.05* mg/kg could be supported. The Meeting estimated an STMR of 0, as no residues were detectable in any samples. It was considered that owing to the high oil content of peanuts residues might accumulate in the nuts, but this was not found (HR = 0.01 mg/kg for peanut).

In trials on cotton seed in Brazil, South Africa and the USA fenamiphos was applied before or shortly after planting. In the US trials the rates were 0.6-1.7 times the maximum registered rate. GAP in the USA requires application at 0.82-3.27 kg ai/ha with no specified PHI, in Brazil 3-5 kg ai/ha with a PHI of 98 days, and in South Africa 15 g ai/100m of row with no specified PHI. Residues were determined in seed (delinted and fuzzy) and ginned trash. The residues in the cotton seed in all the relevant trials (25) were <0.01 mg/kg. The Meeting estimated a maximum residue level of 0.05* mg/kg, confirming the existing CXL, as this was the validated limit of determination. An STMR of 0.01 mg/kg was estimated (HR = 0.01 mg/kg).

The residues in the cotton gin trash were <0.01 mg/kg in 7 trials which were considered to conform to US GAP.

Coffee. Supervised trials were conducted in Brazil, Guatemala and Mexico. In all the trials a single soil application was made to mature trees at the pre-bloom or fruit formation stage. Residues were determined in the fruit (berries) and the beans. The MRL applies to the seed only; the bean and other parts of the fruits are not included.

GAP in Brazil is 1-7 g ai/plant with a PHI of 45 days, in Guatemala 2.4-5 kg ai/ha and a PHI of 60 days, and in Mexico 1-1.5 g ai/plant with a PHI of 45 days. The Mexican trials were not considered, as the prescribed GAP could not be compared with the application rate as expressed in the supervised trials. The residues in the two relevant samples of beans were <0.1 and <0.2 mg/kg. The residues in the berries in rank order were 0.01 (3), 0.02, 0.03, 0.04, <0.05 (4), 0.06 and 0.11 mg/kg. The Meeting did not estimate a maximum residue level as there were insufficient data for beans, and recommended withdrawal of the existing CXLs of 0.1 mg/kg for coffee beans and coffee beans, roasted.

Dietary burden of livestock and animal feeding studies

Tables of dietary burden were compiled for dairy cattle and hens, in which maximum and median residues in various feed items were listed together with an indication of the percentage dry matter, percentage of the item in the diet, and the intake expressed as mg/animal/day. Commodities in which the dry matter was above 85% as received were not corrected for dry matter.

For dairy cattle, a dry matter intake of 15 kg/day for a 500 kg animal was assumed. An exposure of 0.13 ppm in the feed (1.88 mg/animal/day; 0.004 mg/kg body weight/day) was estimated on the basis of the consumption of dry apple pomace, raisin trash, peanut vines and dry tomato pulp, which provided the highest median intake. As a typical diet would not consist only of these items the estimate is probably exaggerated. The lowest level in the diet in the cattle feeding study was 2 ppm fenamiphos sulfoxide or about 15 times the calculated exposure. After feeding at 2 ppm for 28 days residues in the milk, liver, kidney, muscle (flank and loin) and fat (omental, subcutaneous and renal) were below the limits of detection of 0.001 mg/kg in milk and 0.01 mg/kg in tissues. The limits of determination were reported as 0.005 mg/kg in milk and 0.01 mg/kg in tissues. On the basis of these limits the Meeting estimated maximum residue levels of 0.005* mg/kg in milk and 0.01* mg/kg in the tissues. STMRs of 0 were estimated for milk, meat and edible offal, since no residues were detectable in any tissues after feeding at 15 times the calculated exposure level for a dairy animal.

For hens, an intake of 150 g dry matter/day and 2 kg body weight were assumed. An estimated maximum exposure of 0.01 ppm in the feed was based on 100% peanut meal as a worst-case situation, as only peanut meal and cotton meal were included in the dietary burden table. The lowest feeding levels in two studies with labelled fenamiphos were 0.06 and 2 ppm for 14 consecutive days. The total radioactivity from the 0.06 ppm level in eggs or tissues was not reported. With feeding at 2 ppm the maximum radioactivities in the tissues were below the minimum quantifiable limits of 7 to 20 ng/g (ppb) as fenamiphos. On the assumption that the limit of determination reported in cattle tissues is also applicable to poultry tissues and eggs, the Meeting estimated maximum residue levels of 0.01* mg/kg for poultry meat, poultry offal and eggs. It should be noted that the calculated exposure of 0.002 ppm in the hen diet is probably exaggerated as feeding 100% peanut or cotton meal would not be considered typical. STMRs of 0 were estimated for eggs, poultry meat and poultry offal.

Processing

Processing studies on tomatoes, oranges, apples, grapes and pineapples were reported.

Tomatoes containing residues of 0.5 mg/kg fenamiphos were subjected to commercial processing into canned tomatoes, juice and ketchup. Total fenamiphos residues were concentrated in tomato pulp solids (wet and dry) and tomato pomace, as well as other commodities that may be used as animal feed items. Calculated processing factors for tomato juice, pasteurised tomato juice, ketchup and canned tomatoes were 0.74, 0.88, 0.58 and 0.72 respectively. As an STMR of 0.05 mg/kg was estimated for whole tomatoes, an STMR of 0.05 mg/kg was also estimated for tomato juice (HR-P = 0.27 for tomato juice).

Tomatoes fortified with [U-*phenyl*-¹⁴C]fenamiphos at 0.8 mg/kg were allowed to stand at room temperature for 24 hours then blanched, peeled, cored, and cooked for 40 minutes. Blanching and cooking led to a reduction of residues by almost 50%, with 27% of the radioactivity present in the cooking water. There was negligible loss of radioactivity (1.5%) by peeling and coring.

Orange trees were treated at a rate equivalent to 100 kg ai/ha and fruit were harvested and processed when residues had reached maximum levels in the leaves, which were sampled at monthly intervals after treatment. The residues in the whole fruit ranged from <0.01 to 0.13 mg/kg, with average residues of 0.07 mg/kg. The residues were concentrated in unwashed and washed peel, peel bits, clear oil (produced from the peel), chopped peel, pressed dry peel, press liquor and molasses, with processing factors of 6.71, 8.57, 3.28, 64, 1.86, 5.71, 2.86 and 7.0 respectively. Processing

factors for juice and pulp were 0.28 and 0.14 respectively. STMRs were not calculated for the processed fractions as no maximum residue level was estimated for oranges or citrus fruits.

Apple trees were treated with a soil application at 33.6 kg ai/ha. Fruit were harvested 66 days after treatment and processed into juice and pomace. The residues in the apples were 0.14 mg/kg and were concentrated in wet and dry pomace with processing factors of 4.86 and 17.7 respectively. A processing factor of 0.78 was calculated for apple juice. As an STMR of 0.01 mg/kg was estimated for apple an STMR of 0.0078 mg/kg was calculated for juice.

Grapes were processed after treatment at rates of one and 5 times the maximum registered rate in the USA. The residues in fruit were 0.07, 0.02 and 0.02 mg/kg at 55, 56 and 7 days after treatment respectively at 1, 1 and 5 times rates. Fenamiphos residues were concentrated in raisins, raisin trash and dry pomace, with processing factors of 1.57, 8.3 and 5 respectively. Processing factors for juice and wet pomace were <1. An STMR of 0.009 mg/kg was calculated for juice (HR-P for raisins = 0.14 mg/kg; grape juice = 0.04 mg/kg).

Pineapples were processed into raw juice, canned juice, raw bran and dried bran. The residues in the whole fruit were 0.67 mg/kg after 7 applications at 5 times the registered US rate for Hawaii. The residues were concentrated in canned juice, raw bran and dried bran with calculated processing factors of 1.2, 2.1 and 2.5 respectively. STMRs of 0.006 and 0.012 mg/kg were calculated for raw and canned juice (HR-P = 0.17 mg/kg for pineapple juice, canned).

Peanuts were treated with 5 times the maximum registered US rate at the mid to late pegging stage. The residues in the kernels were 0.01 mg/kg. The peanuts were processed into meal, soapstock, crude oil and refined oil. The residues were ≤ 0.01 mg/kg in all the fractions except crude oil which contained 0.02 mg/kg. This was not considered to be a concentration effect as the refined oil contained < 0.01 mg/kg. An STMR of 0 was estimated for peanuts, so the STMR for peanut oil is the same. The Meeting estimated a maximum residue level of 0.05* mg/kg for peanut oil, crude.

The residues in cotton seed were 0.01 mg/kg after a single exaggerated application at planting. Cotton bolls were harvested at maturity 153 days after treatment. The seed was processed into meal, hulls, soapstock, crude oil and refined oil. The residues in all the processed fractions were ≤ 0.01 mg/kg, except in crude oil which contained 0.02 mg/kg. This was not considered to be a concentration effect. The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.01 mg/kg for cotton seed oil, crude.

The Meeting recommended withdrawal of the following existing CXLs which were not supported by data: broccoli, cauliflower, kiwifruit, soya beans, sugar beet and sweet potato.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting estimated the maximum residue and STMR levels shown below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue for compliance with the MRL and for estimation of dietary intake: "sum of fenamiphos, its sulfoxide and sulfone, expressed as fenamiphos".

Commodity		MRL, mg/kg		STMR,	HR/HR-P
CCN	Name	New	Previous	mg/kg	mg/kg
FP 0226	Apple	0.05*		0.01	0.01
JF 0226	Apple juice			0.0078	0.0078
FI 0327	Banana ¹	0.05*	0.1	0.02	0.025
VB 0400	Broccoli	W	0.05*		
VB 0402	Brussels sprouts	0.05	0.05*	0.01	0.01
VB 0041	Cabbages, Head ^{1,2}	0.05	0.05*	0.01	0.05
VR 0577	Carrot ^{1,2}	0.2	0.2	0.02	0.08

Commodity		MRL, mg/kg		STMR,	HR/HR-P
CCN	Name	New	Previous	mg/kg	mg/kg
VB 0404	Cauliflower	W	0.05*		
SB 0716	Coffee beans	W	0.1		
SM 0716	Coffee beans, roasted	W	0.1		
SO 0691	Cotton seed	0.05*	0.05*	0.01	0.01
OC 0691	Cotton seed oil, crude	0.05*		0.01	
MO 0105	Edible offal (Mammalian)	0.01*		0	
PE 0112	Eggs	0.01*		0	
FB 0269	Grapes ^{1,2}	0.1	0.1	0.02	0.09
JF 0269	Grape juice			0.009	0.04
FI 0341	Kiwifruit	W	0.05*		
MM 0095	Meat (Mammalian)	0.01*		0	
VC 0046	Melons, except Watermelon ^{1,2}	0.05*	0.05*	0.02	0.02
ML 0106	Milks	0.005*		0	
FC 0004	Oranges, Sweet, Sour	W	0.5		
SO 0697	Peanut	0.05*	0.05*	0	0.01
OC 0697	Peanut oil, crude	0.05*		0	
VO 0051	Peppers ^{1,2}	0.5		0.055	0.35
FI 0353	Pineapple ^{1,2}	0.05*	0.05*	0.01	0.14
JF 0341	Pineapple juice, canned			0.012	0.17
	Pineapple juice, raw			0.006	
VR 0589	Potato	W	0.2		
PO 0111	Poultry, Edible offal of	0.01*		0	
PM 0110	Poultry meat	0.01*		0	
VD 0541	Soya bean (dry)	W	0.05*		
VR 0596	Sugar beet	W	0.05*		
VR 0508	Sweet potato	W	0.1		
VO 0448	Tomato ^{1,2}	0.5	0.2	0.05	0.30
JF 0448	Tomato juice			0.05	0.27
VC 0432	Watermelon ^{1,2}	0.05*		0.02	0.02

HR: highest residue in edible portion of raw commodity from supervised trials

HR-P: highest residue in processed commodity, calculated from the HR and the processing factor

¹ The information provided to the JMPR precludes an estimate that the acute dietary intake for children would be below the acute reference dose.

² The information provided to the JMPR precludes an estimate that the acute dietary intake for the general population would be below the acute reference dose.

DIETARY RISK ASSESSMENT

Chronic intake

STMRs were estimated for all commodities included in the dietary intake assessment. International Estimated Daily Intakes for the five GEMS/Food regional diets were in the range of 3-14% of the ADI (Annex III).

The Meeting concluded that the intake of residues of fenamiphos resulting from its uses that have been considered by the JMPR is unlikely to present a public health concern.

Acute intake

The acute reference dose for fenamiphos is 0.0008 mg/kg bw as the available data did not permit the 1997 Meeting to establish an acute reference dose different from the ADI. The calculated short-term intakes ranged from 15 to 2900% of the acute reference dose for children and 8 to 863% of the acute reference dose for the general population (Annex IV). It should be noted that for commodities such as apples, bananas, melons, peanuts and watermelons, residues in the edible portion in all supervised trials were below the limit of detection of the method used, but for the purposes of the short-term risk assessment figures at the limit of detection were used in the calculation. The current method does not allow any further refinement of the acute intake assessment.

The Meeting concluded that all commodities should be considered further when an acute reference dose is established from new data or when new data on unit weight, variability factor etc. become available.

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