FENPROPATHRIN (185)

The first draft was prepared by professor Árpád Ambrus, temporary adviser, Hungary

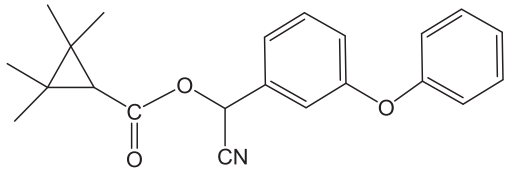
EXPLANATION

Fenpropathrin is a type II pyrethroid insecticide and acaricide used for the control of a variety of arthropods including aphids, worms, moths, beetles, mites, spiders, thrips, flies, fleas and other pests in agriculture. It acts by contact and ingestion. It is also a repellent. It interferes with nerve impulse transmission by acting on sodium channels.

Fenpropathrin was first evaluated by JMPR in 1993 when an ADI of 0–0.03 mg/kg bw was established and a number of MRLs recommended. In 2006 MRL for tea was recommended. The compound was re-evaluated for toxicology within the periodic review programme in 2012 when the Meeting reaffirmed the ADI of 0–0.03 mg/kg bw and established an ARfD of 0.03 mg/kg bw.

Fenpropathrin is scheduled for periodic re-evaluation of residues in 2014. Data to support proposed Codex MRLs on a number of commodities and on animal products were submitted for review.

IDENTITY

ISO common name:	Fenpropathrin
IUPAC name:	(*RS*)-α-cyano-3-phenoxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate
Chemical Abstract name:	cyano(3-phenoxyphenyl)methyl 2,2,3,3-tetramethylcyclopropanecarboxylate
CAS No.:	64257-84-7 (racemate); 39515-41-8 (unstated stereochemistry)
Molecular Formula:	$C_{22}H_{23}NO_3$
Structural Formula:	
Molecular Weight:	349.43

Specifications

Specifications for fenpropathrin have not yet developed by FAO.

Physical and Chemical Properties

Technical material (90%) (Reference: JMPR, 1993)

Chemical/physical property	Results	
Physical state	Liquid	
Colour	Yellow to brown	
Odour	Faint characteristic odour	
Density	1.105	
Vapour pressure	2.15×10^{-6} Pa	
Melting range	45-50 °C	
Flammability	Flash point : 205 ºC Ignition point: 325 ºC	
Solubility in organic solvents (g/L, 23ºC)	Acetone	> 500

Chemical/physical property	Results	
	Acetonitrile	> 500
	Cyclohexanone	> 500
	Ethyl acetate	> 500
	Methanol	216
	Xylene	> 500
Solubility in water	36.3 µg/L at 25.1 ºC	
Octanol/water partition coefficient	Log $_{Po/w}$ = 6.0± 0.20	
Hydrolytic half lives	pH 5 and pH: 7 200 days pH 9: 17.1 days (calculated based on hydrolysis of cyclopropyl-1-^{14}C)-fenpropathrin.	
Stability	No significant breakdown after 20 weeks storage at 60 ºC	

Hydrolytic degradation

The hydrolytic degradation of fenpropathrin was examined at pH 5, 7, and 9 with [phenoxyphenyl-^{14}C]-fenpropathrin or [cyclopropyl-1-^{14}C]-fenpropathrin in sterile aqueous solution containing 1% acetonitrile as co-solvent (Takahashi, *et.al.*, 1983 and 1985, References FM-30-0010 and FM-50-0053; Concha, *et al*, 1992; Reference FM-21-0060).

The predominant hydrolysis reactions were cleavage of the ester linkage and hydration of the cyano group. Major hydrolysis products from cyclopropyl labelled fenpropathrin were TMPA, TMPA-carboxamide and CONH$_2$-fenpropathrin, while from phenoxyphenyl labelled fenpropathrin, were PBacid and CONH$_2$-fenpropathrin

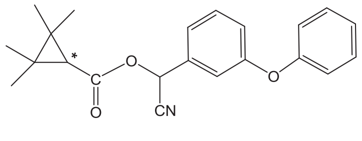
Formulation

The emulsifiable concentrate formulation containing 300 g/L fenpropathrin was used in the residue trials submitted for evaluation.

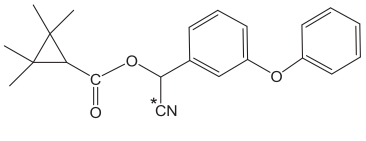
METABOLISM AND ENVIRONMENTAL FATE

The metabolism of fenpropathrin has been investigated in apple, tomato, beans, cotton, cabbage, lactating goat and laying hens. The crops selected represent those for which supervised trials have been provided.

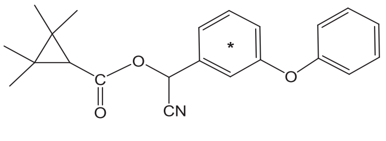
The fate and behaviour of fenpropathrin in plants, animals and soil were investigated using either [cyclopropyl-1-^{14}C]-fenpropathrin, [benzyl-^{14}C]-fenpropathrin, [phenoxyphenyl-^{14}C]-fenpropathrin or [cyano-^{14}C]-fenpropathrin (all with radiochemical purity >98%).



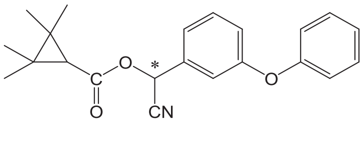
[cyclopropyl-1-^{14}C]-fenpropathrin



[cyano-^{14}C]-fenpropathrin



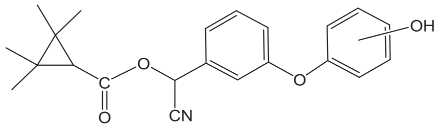
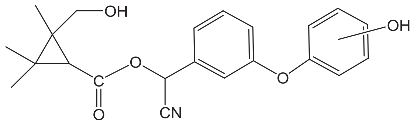
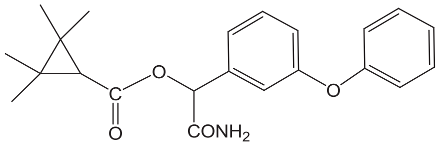
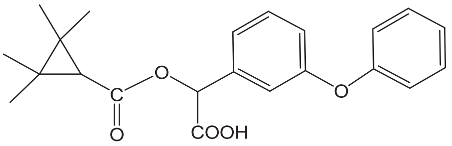
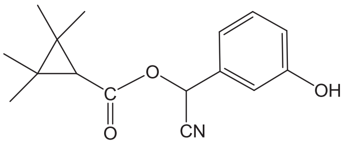
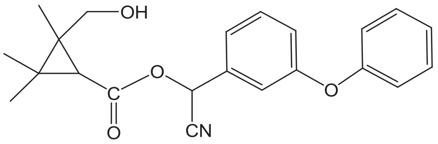
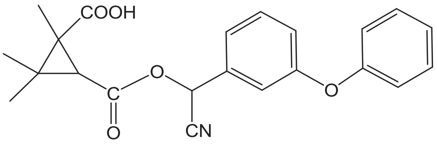
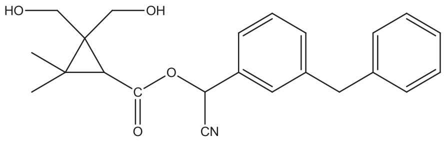
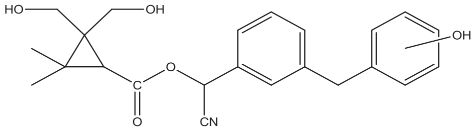
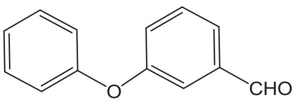
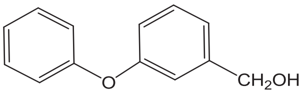
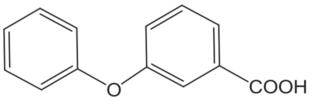
[phenoxyphenyl-^{14}C]-fenpropathrin

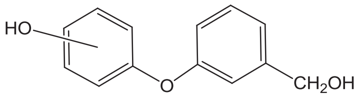
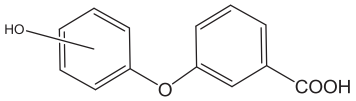
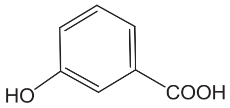
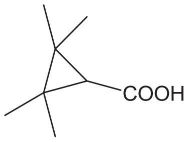
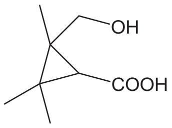
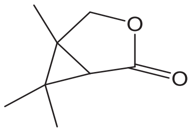
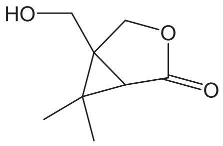
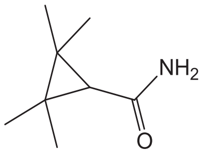
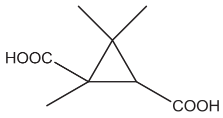


[benzyl-^{14}C]-fenpropathrin

The chemical structures and code names of metabolites and the major degradation products of fenpropathrin are shown in Table 1.

Table 1 Degradation compounds from the metabolism of fenpropathrin

Compound Name	Structure	Found in:
2'-or 4'-OH-Fenpropathrin [α-cyano-3-(2'- or 4'-hydroxyphenoxy)benzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Plant, animal, soil
2'- or 4'-OH-Fenpropathrin-CH_2OH [α-cyano-3-(2'- or 4'-hydroxyphenoxy) benzyl-2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylate]		Plant, animal
CONH$_2$-Fenpropathrin [α-carbamoyl-3-phenoxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Soil, water plant
COOH-Fenpropathrin [α-carboxy-3-phenoxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Soil, plant
Desphenyl-Fenpropathrin [α-cyano-3-hydroxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Animal, soil, plant
Fenpropathrin-CH_2OH [α-cyano-3-phenoxybenzyl 2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylate]		Plant, animal
Fenpropathrin-COOH [α-cyano-3-phenoxybenzyl 2-carboxy-2,3,3-trimethylcyclopropanecarboxylate]		Animal
Fenpropathrin-$(CH_2OH)_2$ [α-cyano-3-phenoxybenzyl 2,2-dihydroxymethyl-3,3-dimethylcyclopropanecarboxylate]		Plant
2'- or 4'-OH-Fenpropathrin-$(CH_2OH)_2$ [α-cyano-3-(2'- or 4'-hydroxyphenoxy)benzyl 2,2-dihydroxymethyl-3,3-dimethylcyclopropanecarboxylate]		Plant
PB aldehyde (PBald) [3-phenoxybenzaldehyde]		Plant, animal, soil
PB alcohol (PBalc) [3-phenoxybenzyl alcohol]		Plant, soil
PBacid [3-phenoxybenzoic acid]		Plant, animal, soil, water

Compound Name	Structure	Found in:
2'- or 4'-OH-PBalc [3-(2'- or 4'-hydroxyphenoxy)benzyl alcohol]		Plant
2'- or 4'-OH-PBacid [3-(2'- or 4'-hydroxyphenoxy)benzoic acid]		Plant, animal, soil
3-OH-Bacid [3-hydroxy-benzoic acid]		Animal, soil
TMPA [2,2,3,3-tetramethylcyclopropane-carboxylic acid]		Plant, animal, soil, water
TMPA-CH_2OH [2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylic acid]		Plant, animal
TMPA-lactone [5,6,6-trimethyl-3-oxabicyclohexan-2-one]		Plant
TMPA-CH_2OH lactone [5-hydroxymethyl-6,6-dimethyl-3-oxabicyclohexan-2-one]		Plant, animal
TMPA carboxamide [2,2,3,3-tetramethylcyclopropane-carboxamide]		Water
TMPA-COOH [2-carboxy-2,3,3-trimethylcyclopropanecarboxylic acid]		Plant, animal

Animal metabolism

Laboratory animals

Absorption by rats was rapid and excretion almost complete (97%) within 48 hours. About 56% of the administered dose was found in urine and 40% in faeces at 48 hours. The amount of radioactivity excreted via expired air was 0.005%. The low residues found in blood, liver, kidney, fat, muscle and brain 24 hours after dosing depleted rapidly over the following 7 days to barely detectable levels, and less than 1.5% of the administered dose remained in the body 8 days after treatment. The highest residue was found in the fat. About 29–53% of the parent compound was detected in the faeces and no parent compound was detected in the urine. The predominant urinary metabolites derived from the acid moiety were identified as TMPA–glucuronide and TMPA-CH_2OH (*trans*). Other metabolites identified were TMPA-COOH (*trans*) and TMPA-CH_2OH-lactone in free form or as the glucuronide conjugate. The major urinary metabolites derived from the alcohol moiety were PBacid in free form and as the glycine conjugate, 4′-OH-PBacid–sulfate and 2′-OH-PBacid–sulfate. The urinary metabolites from the alcohol moiety were similar to those from other pyrethroids. The major faecal metabolite was identified as CH_2OH *trans*-fenpropathrin, followed by COOH *trans*-fenpropathrin, 4′-

OH-fenpropathrin and 4′-OH,CH_2OH *trans*-fenpropathrin. Depending on the dose administered, 30–50% of the applied radioactivity was excreted in faeces as parent compound. Fenpropathrin and TMPA were the major components of ^{14}C in tissues. No sex-related differences in tissue distribution were observed.

Lactating goats

The adsorption and deposition of radioactivity was studied in lactating goats (Ku and Doran, 1990, Reference FM-01-0041). Two animals per group were dosed for five consecutive days via capsules with either [phenoxyphenyl-^{14}C]-fenpropathrin or [cyclopropyl-1-^{14}C]-fenpropathrin at a rate equivalent to 50 ppm (based on a 2 kg diet, 100 mg per dose of test material). A single control animal was dosed, using a placebo capsule, in an identical manner. Sample collection was initiated on study day 1, with the twice daily collection of milk (morning and afternoon) until sacrifice. Urine and faeces were collected daily from study day 1 to day 4 (with cage wash conducted at study termination). Blood samples were collected at specified times during the last day of dosing. At sacrifice, and within 4 hours of the terminal dose, samples of omental and perirenal fat, kidneys, liver, gall bladder contents, heart, loin muscle and rear leg muscle were taken for analysis. Urine, milk and butterfat (obtained from the milk) were quantified directly by LSC of replicate aliquots. Faeces and non-fatty tissues were homogenized and replicate subsamples taken for combustion and radio-assay. Replicate samples of the fatty tissues were solubilised prior to radio-assay.

Milk, tissues and fat (omental and perirenal) were extracted with either acidified acetonitrile/hexane (milk) or acidified acetonitrile (fat and tissues). Extracts were combined, concentrated and partitioned with either ethyl ether or ethyl acetate with further concentration and subsequent quantification by HPLC. Aqueous fractions from the milk, tissues, urine and faeces extractions were subjected to acid hydrolysis, extracted with either ethyl acetate or ethyl ether and the organo-soluble components analysed. Non extractable residues of milk, tissues, fat and faeces were air dried prior to combustion and radio-assay. Selected non-extractable residues were subjected to acid hydrolysis with analysis of the organo-soluble components.

The mean total recovery of radioactivity following dosing with [cyclopropyl-1-^{14}C]-fenpropathrin was 65.2% (63.9–66.5%) with 40.9% (40.0–41.8) recovered in the urine and 23.5% (23.0–24.0) in the faeces. Excretion via milk was a minor route with radioactivity accounting for *ca* 0.15% (0.73 (0.497–0.954)) mg fenpropathrin equivalents for the phenoxyphenyl label and *ca* 0.087% (0.43 (0.416–0.449)) mg fenpropathrin equivalents of total dose 500 mg for the cyclopropyl label. Total radioactive residues in the milk reached a steady state by the evening milking on the third day when average residues in the whole milk were 0.11 mg/L for [cyclopropyl-1-^{14}C]-fenpropathrin treated animals and 0.25 mg/L for the [phenoxyphenyl-^{14}C]-fenpropathrin treated animals. Less than 3% of the radioactivity in the milk was associated with the butterfat. Most of the retained activity was found in the liver, kidney and fat. Levels in these three organs were in the range of 0.4–0.7 mg/kg fenpropathrin equivalent for both labels. Muscle levels were in the range of 0.02–0.04 mg/kg fenpropathrin equivalent.

Between 20% and 40% of the radiocarbon in the milk from animals receiving the phenoxyphenyl label was associated with the parent compound, with nearly all of the remainder being present as the glycine conjugate of PBacid which reached levels of 0.03–0.15 mg/L (Table 3). There were minor amounts of the hydroxylated derivatives of PBacid (0.003–0.01 mg/kg) and also of fenpropathrin itself (0.02–0.12 mg/kg). With the cyclopropyl label, 57–75% of the activity was associated with the parent material with moderate amounts of TMPA (< 0.002–0.005 mg/kg), its hydroxymethyl (< 0.002–0.003 mg/kg) and carboxy (< 0.002–0.003 mg/kg) derivatives and its hydroxymethylated lactone (< 0.002–0.004 mg/kg) (Table 4). In this case, however, the total recovery was only about 70–80% and the concentration of TMPA and all its derivatives did not exceed 0.01 mg/L. The identity of the compounds associated with the radioactivity in the tissues was somewhat similar except that in the liver and kidney there were only traces left as parent material (Table 5). In the case of the phenoxyphenyl label, most of the activity was in the form of PBacid (kidney 0.22–0.34 mg/kg; liver 0.05–0.07 mg/kg) and its glycine conjugate (kidney 0.21–0.38 mg/kg; liver 0.06-0.11 mg/kg) and in the case of the cyclopropyl label TMPA and its derivatives predominated. The hydroxymethyl TMPA-lactone (TMPA-CH_2OH lactone) was practically absent from the fat but was prominent in muscle, liver and kidney, accounting for up to 40% of the activity in the kidney, equivalent to about 0.2 mg/kg.

Table 2 Distribution of radioactive residues in milk and tissues following administration of [phenoxyphenyl-^{14}C]-fenpropathrin or [cyclopropyl-1-^{14}C]-fenpropathrin to lactating goats for 5 consecutive days

Tissue		Radioactive residue (mg/kg)					
		[phenoxyphenyl-^{14}C]-fenpropathrin			[cyclopropyl-1-^{14}C]-fenpropathrin		
		Goat 12	Goat 16	Mean	Goat 10	Goat 13	Mean
Liver		0.329	0.532	0.431	0.495	0.432	0.464
Kidney		0.514	0.863	0.689	0.521	0.441	0.481
Heart		0.0534	0.113	0.0832	0.130	0.110	0.120
Muscle [a]		0.0177	0.0253	0.0215	0.0428	0.0394	0.0411
Fat [a]		0.451	0.852	0.652	0.748	0.556	0.652
Blood [b]		0.0933	0.127	0.110	0.197	0.245	0.221
Blood [c]		0.0618	0.0332	0.0475	0.184	0.244	0.214
Blood [d]		0.0834	0.093	0.0882	0.178	0.228	0.203
Milk:							
Study day	-1 (am)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	-1 (pm)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	1 (am)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
	1 (pm)	0.139	0.282	0.211	0.117	0.0666	0.0918
	2 (am)	0.0682	0.0850	0.0766	0.0609	0.0453	0.0531
	2 (pm)	0.190	0.240	0.215	0.115	0.0877	0.101
	3 (am)	0.0956	0.151	0.123	0.0820	0.0609	0.0715
	3 (pm)	0.186	0.313	0.250	0.141	0.0816	0.111
	4 (am)	0.107	0.0986	0.103	0.0842	0.0624	0.0733
	4 (pm)	0.223	0.278	0.251	0.119	0.0853	0.102
	5 (am)	0.101	0.0958	0.0984	0.103	0.0654	0.0842
	5 (pm)	0.114	0.0989	0.106	0.101	0.0612	0.0811

[a] mean mg/kg value for the tissue samples representing a specific sample matrix

[b] taken at sacrifice,

[c] prior to the final dose,

[d] taken at last milking

LOQ – limit of quantification (0.0004 mg/kg)

Table 3 Summary of the nature of the ^{14}C-fenpropathrin residues in milk following dosing with [phenoxyphenyl-^{14}C]-fenpropathrin

[phenoxyphenyl-^{14}C]-fenpropathrin														
	4’-OH-PBacid		PBacid-glycine		PBacid		Fenpropathrin-CH_2OH		4’-OH-Fenpropathrin		Fenpropathrin		Total[a]	
Milk	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Goat 12														
3 (am)	8.52	0.0083	44.17	0.0431	10.85	0.0106	1.43	< 0.002	0.72	< 0.002	18.98	0.0185	84.67	0.0976
3 (pm)	3.75	0.0073	41.64	0.0815	11.05	0.0216	2.01	0.0039	0.89	< 0.002	24.25	0.0475	83.59	0.1958
4 (pm)	3.83	0.0096	60.66	0.1515	1.27	0.0032	0.73	< 0.002	0.47	< 0.002	27.99	0.0699	94.95	0.2498
5 (am)	5.80	0.0070	56.83	0.0682	2.37	0.0028	0.67	< 0.002	0.34	< 0.002	22.67	0.0272	88.68	0.1200
5 (pm)	4.22	0.0045	37.51	0.0397	5.87	0.0062	2.27	0.0024	2.08	0.0022	28.98	0.0307	80.93	0.1058
Goat 16														
3 (am)	5.53	0.0059	46.98	0.0498	4.68	0.0050	1.28	< 0.002	1.00	< 0.002	28.94	0.0307	88.41	0.1060
3 (pm)	2.93	0.0088	45.06	0.1349	4.96	0.0149	0.86	0.0026	0.54	< 0.002	39.31	0.1177	93.66	0.2994
4 (pm)	3.84	0.0103	48.52	0.1303	2.96	0.0080	0.81	0.0022	0.85	0.0023	36.24	0.0973	93.22	0.2686
5 (am)	8.36	0.0078	32.52	0.0302	5.98	0.0055	1.67	< 0.002	1.09	< 0.002	28.14	0.0261	77.76	0.0928
5 (pm)	3.95	0.0026	41.94	0.0279	3.18	0.0021	6.32	0.0072	2.19	< 0.002	30.31	0.0202	87.89	0.0666

Table 4 Summary of the nature of the ^{14}C-fenpropathrin residues in milk following dosing with [cyclopropyl-1-^{14}C]-fenpropathrin

[cyclopropyl-1-^{14}C]-fenpropathrin																
	TMPA-CH$_2$OH		TMPA-COOH		TMPA-CH$_2$OHlactone		TMPA		Fenpropathrin-CH$_2$OH		4'-OH-Fenpropathrin		Fenpropathrin		Total[a]	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Goat 10																
3 (am)	2.88	0.0024	0.45	< 0.002	2.68	0.0022	5.66	0.0047	0.22	< 0.002	0.00	< 0.002	65.95	0.0547	78.10	0.0830
3 (pm)	1.93	0.0030	1.90	0.0029	2.20	0.0034	2.23	0.0034	0.05	< 0.002	0.50	< 0.002	74.83	0.1154	83.64	0.1542
4 (pm)	2.80	0.0034	0.42	< 0.002	3.17	0.0039	3.10	0.0038	0.66	< 0.002	1.98	0.0024	66.09	0.0804	78.22	0.1217
5 (am)	0.92	< 0.002	1.14	< 0.002	1.20	< 0.002	2.14	0.0021	0.42	< 0.002	1.06	< 0.002	72.21	0.0724	79.09	0.1003
5 (pm)	0.32	< 0.002	1.60	< 0.002	1.43	< 0.002	2.34	0.0025	0.63	< 0.002	0.77	< 0.002	74.99	0.0790	82.08	0.1053
Goat 13																
3 (am)	3.27	0.0020	1.01	< 0.002	1.68	< 0.002	2.41	< 0.002	0.27	< 0.002	1.80	< 0.002	62.19	0.0373	72.63	0.0599
3 (pm)	2.71	0.0024	0.90	< 0.002	3.89	0.0034	1.82	< 0.002	0.47	< 0.002	1.00	< 0.002	64.91	0.0570	75.70	0.0878
4 (pm)	3.15	0.0031	1.01	< 0.002	3.30	0.0032	2.38	0.0023	1.11	< 0.002	3.04	0.0030	57.58	0.0565	71.57	0.0982
5 (am)	3.05	< 0.002	1.68	< 0.002	3.62	0.0021	2.07	< 0.002	0.55	< 0.002	1.01	< 0.002	56.53	0.0325	68.51	0.0575
5 (pm)	2.98	< 0.002	0.29	< 0.002	1.82	< 0.002	1.98	< 0.002	0.34	< 0.002	0.33	< 0.002	67.34	0.0398	75.08	0.0591

[a] ^{14}C- Fenpropathrin equivalents,

[b] Fractions containing TMPA-CH$_2$OH may contain TMPA-COOH,

[c] Fractions containing Fenpropathrin-CH$_2$OH may contain Desphenyl-Fenpropathrin or Fenpropathrin-COOH

Table 5 Summary of the nature of the ^{14}C-fenpropathrin residues in tissues following dosing with [phenoxyphenyl-^{14}C]-fenpropathrin

[phenoxyphenyl-^{14}C]-fenpropathrin														
	4'-OH-PBacid		PBacid-glycine		PBacid		Fenpropathrin-CH_2OH[c]		4'-OH-Fenpropathrin		Fenpropathrin		Total[a]	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Goat 12														
Rear leg muscle	2.19	< 0.002	8.73	< 0.002	9.94	< 0.002	5.12	< 0.002	1.16	< 0.002	50.15	0.0075	77.29	0.0150
Loin muscle	2.18	< 0.002	16.62	0.0048	10.03	0.0029	1.33	< 0.002	1.32	< 0.002	51.67	0.0148	83.15	0.0287
Omental fat	0.75	0.0029	4.95	0.0193	1.83	0.0071	0.87	0.0034	0.29	< 0.002	76.65	0.2986	85.34	0.3895
Perirenal fat	0.69	0.0034	5.07	0.0248	1.97	0.0097	0.83	0.0041	0.57	0.0020	78.29	0.3837	87.42	0.4901
Kidney	9.42	0.0533	36.42	0.2059	39.77	0.2248	0.64	0.0036	0.45	0.0025	0.88	0.0050	87.58	0.5653
Heart	1.91	< 0.002	22.97	0.0126	7.38	0.0041	1.76	< 0.002	1.24	< 0.002	40.40	0.0222	75.66	0.0550
Liver	9.77	0.0327	19.31	0.0645	14.80	0.0494	4.84	0.0162	2.54	0.0085	3.02	0.0101	54.28	0.3342
Goat 16														
Rear leg muscle	4.18	< 0.002	30.19	0.0096	10.33	0.0033	1.69	< 0.002	0.88	< 0.002	42.58	0.0135	89.85	0.0317
Loin muscle	1.71	< 0.002	34.00	0.0089	13.42	0.0035	1.11	< 0.002	0.53	< 0.002	34.88	0.0090	85.65	0.0259
Omental fat	0.31	0.0025	3.71	0.0294	1.20	0.0095	0.86	0.0068	0.26	0.0021	81.28	0.6448	87.62	0.7933
Perirenal fat	0.48	0.0043	5.03	0.0454	2.68	0.0242	0.78	0.0070	0.36	0.0032	76.70	0.6918	86.03	0.9020
Kidney	8.76	0.0813	40.70	0.3778	36.80	0.3416	0.38	0.0035	0.60	0.0056	1.60	0.0149	88.84	0.9280
Heart	3.32	0.0038	12.21	0.0141	3.37	0.0039	1.70	0.0020	0.61	< 0.002	61.28	0.0707	82.49	0.1154
Liver	12.15	0.0638	20.56	0.1079	12.48	0.0655	6.51	0.0342	2.38	0.0125	3.34	0.0175	57.42	0.5250

Table 6 Summary of the nature of the ^{14}C-fenpropathrin residues in tissues following dosing with [cyclopropyl-1-^{14}C]-fenpropathrin

[cyclopropyl-1-^{14}C]-fenpropathrin																
	TMPA-CH$_2$OH[b]		TMPA-COOH		TMPA-CH$_2$OHlactone		TMPA		Fenpropathrin-CH$_2$OH[c]		4'-OH-Fenpropathrin		Fenpropathrin		Total[a]	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Goat 10																
Rear leg muscle	15.29	0.0071	3.88	< 0.002	18.84	0.0088	7.32	0.0034	1.97	< 0.002	1.91	< 0.002	12.58	0.0059	61.79	0.0467
Loin muscle	5.61	0.0022	13.04	0.0052	20.48	0.0082	3.74	< 0.002	1.62	< 0.002	0.97	< 0.002	10.30	0.0041	55.76	0.0401
Omental fat	0.75	0.0059	0.26	0.0020	1.80	0.014	1.00	0.008	0.54	0.004	0.48	0.004	88.10	0.688	92.93	0.781
Perirenal fat	0.43	0.0033	0.50	0.0038	2.61	0.02	0.90	0.0068	0.27	0.0021	0.31	0.0024	84.81	0.6447	89.83	0.7602
Kidney	15.94	0.0861	3.71	0.0200	41.33	0.223	6.67	0.0360	1.25	0.0068	0.62	0.0033	1.68	0.0091	71.20	0.5400
Heart	5.98	0.0083	0.93	0.0013	7.69	0.0106	2.42	0.003	0.73	< 0.002	0.36	< 0.002	52.89	0.0732	71.0	0.1384
Liver	15.59	0.0768	7.13	0.0350	14.62	0.0720	19.19	0.094	4.02	0.02	2.51	0.012	2.61	0.013	65.67	0.493
Goat 13																
Rear leg muscle	16.74	0.0076	4.23	< 0.002	18.15	0.0082	5.89	0.003	1.08	< 0.002	0.78	< 0.002	14.9	0.008	61.80	0.046
Loin muscle	6.78	0.0024	14.00	0.0052	20.32	0.0074	7.25	0.003	3.93	< 0.002	0.95	< 0.002	7.30	0.003	60.53	0.038
Omental fat	1.06	0.0062	0.58	0.0034	2.22	0.0130	1.58	0.009	0.86	0.0051	0.56	0.0033	81.4	0.478	88.27	0.588
Perirenal fat	0.60	0.0033	1.18	0.0064	3.80	0.0206	1.62	0.009	0.95	0.0051	0.54	0.0029	68.4	0.371	77.12	0.542
Kidney	13.05	0.0606	4.13	0.0192	40.22	0.1867	7.99	0.037	1.56	0.0072	0.87	0.0040	1.27	0.006	69.09	0.464
Heart	10.32	0.0121	1.95	0.0023	6.74	0.0079	5.24	0.006	0.69	< 0.002	0.77	< 0.002	42.2	0.049	67.89	0.117
Liver	16.05	0.0693	4.39	0.0190	10.47	0.0452	17.30	0.075	2.27	0.0098	2.08	0.0090	2.28	0.01	54.84	0.432

[a] ^{14}C- Fenpropathrin equivalents,

[b] Fractions containing TMPA-CH$_2$OH may containing TMPA-COOH,

[c] Fractions containing Fenpropathrin-CH$_2$OH may contain Desphenyl-Fenpropathrin or Fenpropathrin-COOH

Laying Hens

Fenpropathrin, labelled in either the cyclopropyl or the benzyl ring was administered in capsules to laying hens daily for 10 days (Ku and Doran, 1990, Reference FM-01-0042). The group of 10 hens were dosed with [phenoxyphenyl-^{14}C]-fenpropathrin (benzyl-label) and [cyclopropyl-1-^{14}C]-fenpropathrin (cyclo-label) at a nominal rate of either 0.5 or 5 mg/kg body weight. Eggs were collected every morning and evening and excreta daily. The hens were all sacrificed within four hours of the last dose and kidneys, liver, heart, gizzard (washed without contents), ovaries and contents, muscle and skin were retained for analysis.

The samples were combusted and the ^{14}C activity was determined with liquid scintillation counter. Liquid samples were directly analysed with LSC. The residues in eggs and tissues were extracted with acetonitrile/1% conc. HCl. The organic phase was evaporated and the remaining aqueous phase was diluted and partitioned into ethyl acetate. The concentrated extract was analysed with HPLC. The remaining aqueous fraction was hydrolysed by refluxing it at 80–90 °C with 6N HCl overnight. The radioactivity was determined with LSC. The remaining radioactivity in PES was also determined with LSC. Portions of egg, meat, fat and liver were also analysed with gas chromatography based on acetone/hexane (1/2 v/v) extraction, silica gel clean-up. The polar metabolites were determined after methylation with diazomethane. The compounds were identified with GC/MS applying authentic analytical standards.

The recovery of total radioactivity from excreta, eggs and tissues was between 75 and 84% of the total applied dose. Between 98.9 and 99.6% of the recovered activity was found in the faeces irrespective of the label. Approximately 0.05% of the applied benzyl label was found in the eggs and 0.2% of the cyclopropyl label. The total radioactive residues expressed as fenpropathrin equivalent are summarised in Table 7.

Table 7 Total radioactive residues [mg/kg] expressed as fenpropathrin and parent fenpropathrin found following treatments with benzyl-labelled and cyclopropyl-labelled fenpropathrin at 5 ppm level

Sample	Benzyl -label		Cyclopropyl -label	
	Total, mg/kg	Parent, mg/kg	Total, mg/kg	Parent, mg/kg
Egg	0.05-0.22	0.016-0.07	0.22-0.49	0.014-0.061
Meat, breast	0.07	0.02	0.53	0.0324
Meat, thigh	0.16	0.0376	0.57	0.033
Fat	0.87	0.4315	0.90	0.5781
Liver	1.46	0.0143	2.91	0.0357
Kidney	4.56	0.0963	4.19	0.2127
Heart	0.37	0.0838	1.35	0.0825
Gizzard	5.08	0.6563	4.86	0.9492
Skin	0.48	0.1653	0.81	0.2164
Excreta	48.8-67.5	2.72-4.82	50.7-63.3	3.69-4.01

The percent proportion and concentration of parent compound and the major metabolites, expressed as parent fenpropathrin, in eggs and tissues after treatments at 5 ppm level are shown in Tables 8–13.

Table 8 Distribution of residues in eggs after treatment with [benzyl-^{14}C]-fenpropathrin at 5 mg/kg bw.

Component	Percent	mg/kg
Fenpropathrin	29.90-32.38	0.0161-0.0698
4'-OH PBacid	4.07-5.88	0.003-0.0129
PBacid-glycine	1.72-5.03	0.0026-0.0093
PBacid	3.23-3.68	0.0017-0.0079
Fenpropathrin-CH_2-OH	3.98-6.08	0.0032-0.0117
4'-OH- Fenpropathrin	1.33-1.7	0.0009-0.0029
3-OH-BAacid	4.53-5.68	0.0023-0.0108

Table 9 Distribution of residues in eggs after treatment with [cyclopropyl-1-^{14}C]-fenpropathrin at 5 mg/kg bw.

Component	Percent	mg/kg
Fenpropathrin	6.05 - 13.34	0.014-0.061
TMPA-CH_2OH	10.24-11.73	0.023-0.053
TMPA-COOH	2.23-3.01	0.005-0.013
TMPA-CH_2-lactone	5.97-6.96	0.016-0.032
TMPA	5.91-9.02	0.02-0.029
Fenpropathrin-CH_2-OH	1.55-2.33	0.004-0.012
4'-OH- Fenpropathrin	1.51-4.29	0.01-0.013

Table 10 Percent distribution of residues in chicken muscle after treatment with [phenoxyphenyl-^{14}C]-fenpropathrin

	Breast muscle		Thigh muscle	
Component	Percent	mg/kg	Percent	mg/kg
Fenpropathrin	14.75	0.0100	23.12	0.0376
4'-OH PBacid	9.01	0.0061	5.23	0.0085
PBacid-glycine	5.11	0.0034	2.48	0.0040
PBacid	23.90	0.161	19.84	0.0323
Fenpropathrin-CH_2-OH	2.33	0.0016	2.65	0.0043
4'-OH- Fenpropathrin	1.73	0.0012	1.95	0.0032
3-OH-BAcid	16.02	0.011	9.34	0.0152

Table 11 Percent distribution of residues in chicken muscle after treatment with [cyclopropyl-1-^{14}C]-fenpropathrin

	Breast muscle		Thigh muscle	
Component	Percent	mg/kg	Percent	mg/kg
Fenpropathrin	6.09	0.0324	5.82	0.0331
TMPA-CH_2OH	14.47	0.0769	18.013	0.1032
TMPA-COOH	7.72	0.0410	4.54	0.0258
TMPA-CH_2-lactone	12.93	0.0687	11.66	0.0664
TMPA	15.14	0.0804	16.27	0.0926
Fenpropathrin-CH_2-OH	0.60	0.0032	1.06	0.006
4'-OH- Fenpropathrin	0.97	0.0052	1.49	0.0085

Table 12 Percent distribution of residues in chicken liver, kidney skin after treatment with [phenoxyphenyl-^{14}C]-fenpropathrin

Component	Liver	Kidney	Skin	Fat	Gizzard
Fenpropathrin	0.98	2.11	34.51	49.63	12.91
4'-OH PBacid	16.10	26.02	12.53	0.65	14.38
PBacid-glycine	5.62	6.23	2.4	0.39	8.67
PBacid	14.65	8.73	7.73	1.48	6.89
Fenpropathrin-CH_2-OH	1.33	0.63	3.13	2.84	1.70
4'-OH- Fenpropathrin	0.84	0.44	1.01	1.24	0.70
3-OH-BAacid	28.89	35.35	12.70	3.77	12.25

Table 13 Percent distribution of residues in chicken liver, kidney, skin after treatment with [cyclopropyl-1-^{14}C]-fenpropathrin at 5 ppm level

Component	Liver	Kidney	Skin	Fat	Gizzard
Fenpropathrin	1.23	5.08	26.73	63.91	19.54
TMPA-CH_2OH	14.71	7.71	13.00	2.20	5.22
TMPA-COOH	11.37	15.28	3.15	0.85	6.89
TMPA-CH_2-lactone	14.81	5.3	6.95	5.64	6.08
TMPA	26.38	47.49	15.37	14.23	17.18
Fenpropathrin-CH_2-OH	1.77	1.02	3.28	4.28	22.99
4'-OH- Fenpropathrin	1.13	1.28	1.15	1.57	2.89

Summary of animal metabolism

The major biotransformation reactions of fenpropathrin in animals consisted of oxidation at the methyl groups of the acid moiety and at the 2'- or 4'-positions of the alcohol moiety, cleavage of the ester and ether linkages and conjugation of the resultant carboxylic acids and alcohols. The proposed metabolic pathway for fenpropathrin in animals is shown in Figure 1.

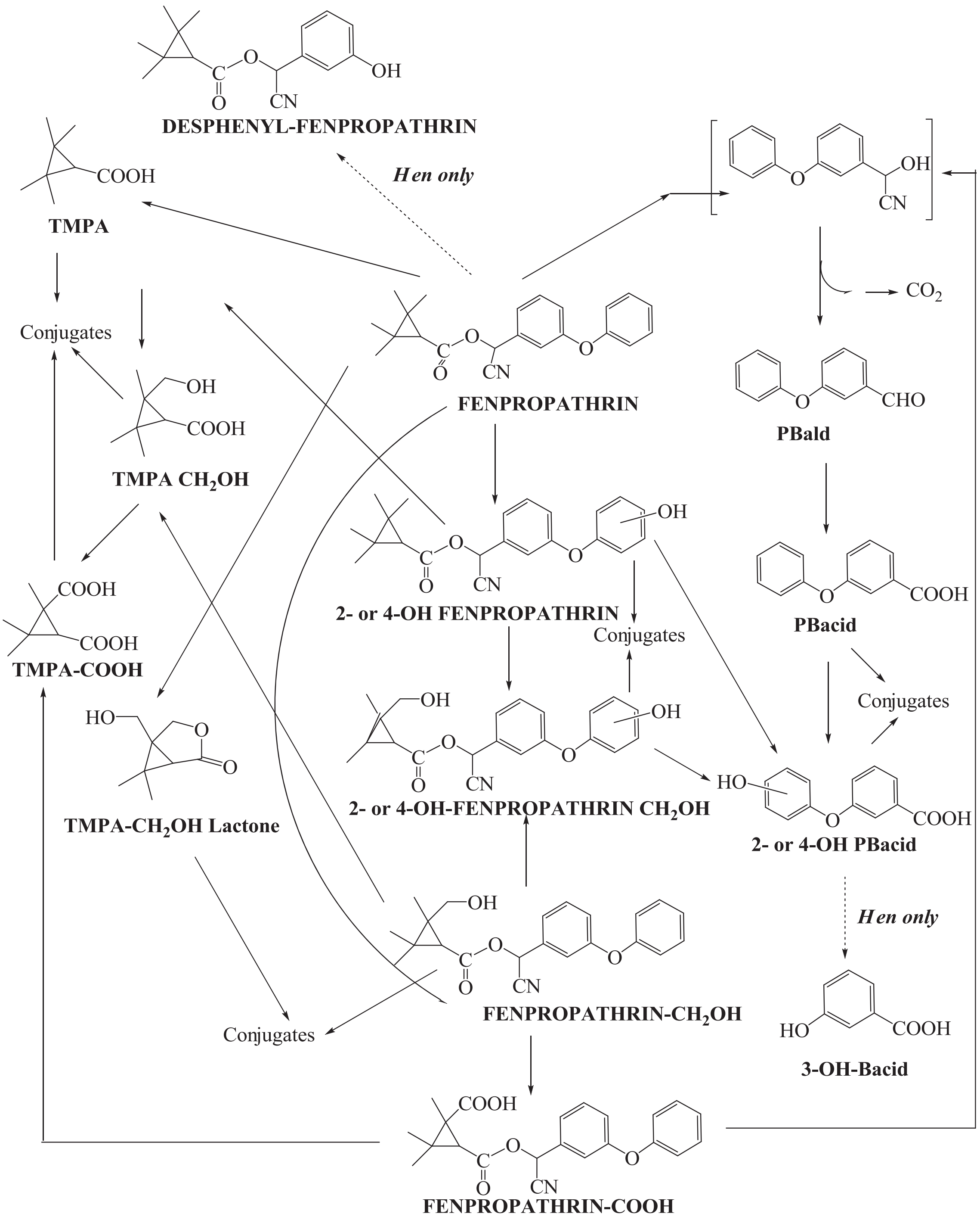


Figure 1 Metabolism and Biotransformation Pathway for Fenpropathrin in Animals

Plants metabolism

Apples

In 1984, one apple tree (cv. Red Delicious) in California, USA was treated 3 times with [cyclopropyl-1-^{14}C]-fenpropathrin and [benzyl-^{14}C]-fenpropathrin (both radiochemical purity > 99%) at a rate equivalent to 0.45 kg ai/ha (Chen and Abell, Report FM-41-0031,1985 and Reference FM-51-0022, 1986). Samples were collected 14 days after the final application. Apple fruit was washed with methanol and separated into peel and pulp. Samples were pulverized with dry ice then extracted with acetone and methanol/water. Analysis was by autoradiography/TLC/LSC. The radioactivity remaining in un-extractable material was determined by combustion/LSC. Free metabolites were separated from conjugated metabolites by solid phase chromatography on a silica column eluted with hexane/acetone, methanol/acetone and methanol. To release aglycones, conjugated metabolites were subjected either to enzyme hydrolysis using cellulase or β-glucosidase then acidified and extracted with diethyl ether, or to chemical hydrolysis with both acid and base. Characterization of metabolites was by radio-TLC.

Table 14 Distribution of ^{14}C-fenpropathrin in apple tree samples

Sample	Benzyl-label		Cyclopropyl-label	
	mg/kg fenpropathrin equivalent	% TRR	mg/kg fenpropathrin equivalent	% TRR
Leaves	12.2	61.0	15.9	72.1
Branches	4.0	13.9	2.5	4.2
Fruit	2.11	25.1	1.40	24.7

Table 15 Extractability of ^{14}C-fenpropathrin radioactivity (%TRR)

	Leaves		Branches	Fruit	
	Benzyl-label	Cyclopropyl-label	Benzyl-label	Benzyl-label	Cyclopropyl-label
Acetone	81	87	89	94	94
Methanol/water	11	8	6	3	3
Un-extractable	8	5	5	3	3

The cyclopropyl-^{14}C-treated branches contained only 2.5% of TRR and therefore no further analysis was carried out.

Table 16 Identity of fenpropathrin metabolites in apple fruits and apple plants (%TRR)

	Apple fruits		Apple plants	
	Benzyl-label	Cyclopropyl-label	Benzyl-label	Cyclopropyl-label
Fenpropathrin	91.8	93.6	60.6	65.6
Conjugated metabolites	2.5	2.5		
4’-OH-Fenpropathrin	0.8	0.8	1.9	1.8
Fenpropathrin-CH_2OH	0.6	0.6	1.6	1.3
Fenpropathrin-CH_2OH conjugates			1.1	0.9
2’- or 4’-OH-Fenpropathrin-CH_2OH conjugates			0.1	0.3
PBalc conjugates			4.8	
PBacid conjugates			1.3	
2’- or 4’-OH-PBalc conjugate			1.0	
2’- or 4’-OH-PBacid conjugates			1.2	
TMPA conjugates				4.3
TMPA-CH_2OH conjugates				3.4
TMPA-CH_2OH lactone conjugates				1.1
Other free metabolites	1.4*	0.2*	6.7**	3.5**
Other conjugated metabolites			12.0***	13.0***
Non-extractable	2.9	2.2	7.7	4.8

* contained at least 2 metabolites

** contained at least 5 metabolites

*** contained at least 10-20 metabolites

Tomatoes

In studies carried out in California, USA in 1983, greenhouse-grown tomato plants (cv. Bonny Best) were treated four times, 7–8 days apart, with [cyclopropyl-1-^{14}C]-fenpropathrin and [benzyl-^{14}C]-fenpropathrin (both radiochemical purity >99%) at rates equivalent to 0.224 kg ai/ha (Chen and Abell, 1985 and 1986; References FM-41-0031 and FM-51-0023). Fruit and leaves were extracted at harvest (PHI 19 days) as described for apples.

Table 17 Distribution of ^{14}C-fenpropathrin in tomato plant samples expressed as fenpropathrin equivalent

Sample	Benzyl-label	Cyclopropyl-label
	mg/kg	mg/kg
Leaves	5.8	4.0
Stem and petioles	0.49	0.53
Fruit	0.10	0.04

Table 18 Extractability of ^{14}C-fenpropathrin radioactivity (%TRR)

	Leaves		Stems and petioles	
	Benzyl-label	Cyclopropyl-label	Benzyl-label	Cyclopropyl-label
Acetone	78	78	78	80
Methanol/water	13	15	12	10
Unextractable	9	7	10	10

Table 19 Characterization of fenpropathrin metabolites in tomatoes (%TRR)

	Benzyl-label	Cyclopropyl-label
Fenpropathrin	66.1	29.6
Conjugated metabolites	28.2	58.6
Free metabolites	0.2	5.1
Un-extractable	5.5	6.7

Table 20 Identity of fenpropathrin metabolites in tomato plants (%TRR)

	Benzyl-label	Cyclopropyl-label
Fenpropathrin	36.5	39.1
Fenpropathrin-CH_2OH conjugates	2.2	2.1
4’-OH-Fenpropathrin-CH_2OH conjugates	1.0	0.9
Fenpropathrin-$(CH_2OH)_2$	3.1	2.7
PBald conjugates	0.4	
4’-OH-PBacid conjugates	7.0	
PBacid conjugates	2.0	
TMPA conjugates		4.6
TMPA-CH_2OH conjugates		7.5
Other free metabolites	5.7*	5.1*
Other conjugated metabolites	33.1**	30.6**

	Benzyl-label	Cyclopropyl-label
Non-extractable	9.3	7.4

* contained at least 10 metabolites

** contained at least 15 metabolites

In the fruit, the radioactivity was too low to allow full characterization, but some two thirds were present as unchanged fenpropathrin with a further 28% as conjugated metabolites. In the leaves, only 30% of the total residue was present as parent and just under 60% as conjugated metabolites. In the case of the benzyl label, the most prominent metabolites were conjugates of PBacid and its 4'-hydroxy derivative (4'-OH-PBacid), although these only constituted a minor proportion of the total radioactivity. The main metabolites reported in the case of the cyclopropyl label were conjugates of TMPA and hydroxymethyl-TMPA (TMPA-CH2OH).

In studies carried out in California, USA in 1994, greenhouse-grown tomato plants (cv. 5715) were sprayed four times with a 2.4% EC formulation containing [cyclopropyl-1-^{14}C]-fenpropathrin (radiochemical purity 98.7%) and [phenoxyphenyl-^{14}C]-fenpropathrin (radiochemical purity 98.4%) at rates equivalent to 0.224 kg ai/ha (Toia, *et. al.,* 1995; Reference FM-51-0070). Fruit and leaves were collected at harvest (PHI 3 days); fruit were washed with hexane/acetone. Total radioactivity was determined by combustion/LSC. The washes were evaporated, redissolved in hexane/acetone and analysed by radio-HPLC/TLC. The major radiolabelled zone was removed from the TLC plate, dissolved in acetone, filtered, evaporated and analysed by GC-MS. Tomato fruit were extracted using acetonitrile with the supernatant being analysed using LSC. Further extractions were made using methanol and dichloromethane. Extracts were analysed using radio-HPLC with UV detection and TLC. Radioactivity in dried, un-extracted material was determined by combustion/LSC. Soil samples were analysed by combustion/LSC to determine the total radioactive residue; only small amounts of radioactivity were detected.

Table 21 Radioactive residues in tomatoes following treatment with [^{14}C]-fenpropathrin

	Phenoxyphenyl-label		Cyclopropyl-label	
	mg/kg	%TRR	mg/kg	%TRR
Rinse	0.34	33.6	0.23	27.3
Rinsed fruit	0.68	66.4	0.62	72.8
Total	1.02	100	0.85	100

The majority of radioactivity was attributed to parent compound but small amounts of *trans*-TMPA-COOH and/or *trans*-TMPA-CH$_2$OH was tentatively identified.

Table 22 Radioactivity in extracts from tomato plants treated with ^{14}C-fenpropathrin; mg/kg fenpropathrin equivalents (dichloromethane extracts not included)

	Available	Fenpropathrin		Polar material		Miscellaneous	
		mg/kg	%	mg/kg	%	mg/kg	%
Phenoxyphenyl-label							
Rinses	0.34	0.34	97.9	0.00	0.0	<< 0.01	2.1

Extracts	0.65	0.62	95.3	0.01	2.1	<< 0.01	2.7
Total	0.99	0.96	96.2	0.01	1.3	0.03	2.5
Cyclopropyl-label							
Rinses	0.23	0.23	98.9	0.00	0.0	<< 0.01	1.1
Extracts	0.53	0.52	97.7	0.01	1.8	<< 0.01	0.05
Total	0.76	0.75	98.0	0.01	1.3	0.01	0.7

Beans

In 1984, five pinto bean plants (cv. Idaho 111) grown in a greenhouse in California, was treated three times with [cyclopropyl-1-^{14}C]-fenpropathrin and [benzyl-^{14}C]-fenpropathrin (both radiochemical purity >99%) at a rate equivalent to 0.224 kg ai/ha (Chen and Abell, 1985, 1986; References FM-41-0031 and FM-51-0024). Samples were collected 15 days after the final application. Fruit and leaves were extracted at harvest as described for apples.

Table 23 Distribution of ^{14}C-fenpropathrin in bean plant samples expressed as fenpropathrin equivalent

Sample	Benzyl-label mg/kg	Cyclopropyl-label mg/kg
Leaves	8.8	5.1
Stem and petioles	1.3	0.6
Bean pods	0.1	0.1
Beans	0.03	0.07

Table 24 Extractability of ^{14}C-fenpropathrin radioactivity (%TRR)

	Leaves		Stems and petioles	
	Benzyl-label	Cyclopropyl-label	Benzyl-label	Cyclopropyl-label
Acetone	63	73	74	81
Methanol/water	32	22	20	13
Unextractable	5	5	6	6

Table 25 Characterization of fenpropathrin metabolites in beans (%TRR)

	Benzyl-label	Cyclopropyl-label
Fenpropathrin	4.1	0.1
Conjugated metabolites	60.7	50.7
Free metabolites	17.0	4.1
Unextractable	18.2	45.1

Table 26 Identity of fenpropathrin metabolites in pinto beans leaves (%TRR)

	benzyl-label	Cyclopropyl-label
Fenpropathrin	46.7	46.4
PBald conjugates	19.5	
PBalc conjugates	4.0	
4'-OH PBacid conjugates	9.8	
2'-OH PBalc conjugates	0.4	
4'-OH PBalc conjugates	0.3	
PBacid conjugates	1.1	
TMPA conjugates		7.0
TMPA-CH_2OH conjugates		16.7

	benzyl-label	Cyclopropyl-label
TMPA-CH$_2$OH lactone conjugates		2.2
Other free metabolites	4.6**	3.3**
Other conjugated metabolites	8.5*	8.4**
Unknown conjugate		11.2***
Non-extractable	5.1	4.8

* contained at least 10 metabolites

** contained at least 6 metabolites

*** artefact of the acid hydrolysis product

Cotton

Cotton pants were treated four times with [cyclopropyl-1-^{14}C]-fenpropathrin and [phenoxyphenyl-^{14}C]-fenpropathrin (both radiochemical purity > 99%) applying a total of *ca*. 4.7–4.8 mg ^{14}C-fenpropathrin (Hitchings and Roberts, 1977; Reference FM-81-0005). The plant parts were extracted with acetonitrile/water and the main products in the extracts were found to be fenpropathrin itself with small amounts of TMPA and PBacid together with some polar material which is likely to have consisted primarily of conjugates of either PBacid or TMPA. In the leaves at harvest (the interval between treatment and harvest was 66 days for the benzyl label and 111 days for the cyclopropyl label) the total remaining activity included 70% parent in the case of the cyclopropyl label and 55% parent in the case of the phenoxyphenyl label. Most of the remaining activity was tentatively accounted for by PBacid and TMPA, mainly in conjugated forms. Examination of the plants grown on soils treated with 0.5 kg/ha of fenpropathrin and containing 0.86 mg/kg fenpropathrin equivalent showed only extremely low uptake of radioactivity (0.002 mg/kg in leaves and 0.01 mg/kg in bolls), demonstrating limited tendency for translocation.

In a study carried out outdoors in California in 1992, cotton plants (cv Acala Royale) were sprayed with [phenoxyphenyl-^{14}C]- or [cyclopropyl-^{14}C]-fenpropathrin (97.9% or > 99.9% radiochemical purity) on 4 occasions at a rate equivalent to 0.336 kg as/ha (total application rate 1.344 kg as/ha) (Kimmel. 1994, Reference FM-41-0067). Plants were harvested by hand 21 days after the final application. Seeds were delinted by hand and extracted using hexane and extracted radioactivity was analysed by LSC, TLC, HPLC-UV and GC-MS. The un-extracted material was further extracted with methanol/water, followed by diluted hydrochloric acid/methanol. Cotton lint and foliage were extracted using hexane/acetone and analysed by LSC, TLC and HPLC. Unextracted material was further extracted using methanol/water and analysed by LSC. The results are shown in Tables 27–29.

Table 27 Distribution of radioactivity of cotton plant parts expressed as fenpropathrin equivalents

	Phenoxyphenyl label		Cyclopropyl label	
	mg/kg	%TRR	mg/kg	%TRR
Seed rinses	0.71	62.7	1.07	67.5
Rinsed seed	0.42	37.3	0.52	32.6
Total seed	1.14	100.0	1.59	100.0
Lint rinses	48.6	63.9	31.8	76.2

	Phenoxyphenyl label		Cyclopropyl label	
	mg/kg	%TRR	mg/kg	%TRR
Rinsed lint	27.4	36.1	10.0	23.8
Total lint	76.0	100.0	41.8	100.0
Foliage	78.6	100.0	67.7	100.0

Table 28 The extractability of radioactivity from cotton plant parts (mg/kg fenpropathrin equivalents)

Label/sample	Hexane	Hexane/ acetone	Methanol/ water	Methanol/ HCl	Total extracted	Unextracted	Accountability (%TRR)
Phenoxyphenyl							
seeds	0.34	0.04	0.10	0.01	0.49	0.01	118.9
lint	-	17.2	6.2	-	23.4	4.0	100.0
foliage	-	55.8	22.2	-	78.0	-	-
Cyclopropyl							
seeds	0.36	0.05	0.10	0.01	0.51	0.02	101.6
lint	-	6.1	2.7	-	8.8	1.1	100.0
foliage	-	41.9	19.0	-	60.9	-	-

The majority of the radioactivity was associated with parent compound although a small number of low level metabolites were also detected.

Table 29 The Identity of fenpropathrin metabolites in cotton plant parts (mg/kg fenpropathrin equivalents)

	Phenoxyphenyl				Cyclopropyl			
	seed rinse	seed extract	lint	foliage	seed rinse	seed extract	lint	foliage
Fenpropathrin	0.71 (100%)	0.45 (93.8%)	69.2 (96.2%)	54.2 (69.5%)	1.1 (99.5%)	0.43 (85.6%)	40.7 (100%)	41.0 (67.4%)
PBacid		0.01		0.02				
TMPA						0.007		0.08
TMPA-lactone								0.22
Trans-TMPA-CH_2OH								1.2
Trans-TMPA-COOH								6.7
4’-OH-PBacid				1.8				
4’-OH-Fenpropathrin								0.06
Desphenyl-Fenpropathrin			0.15					
COOH-Fenpropathrin				0.15				0.26
$CONH_2$-Fenpropathrin				0.13				0.12
Polar material				11.2				9.1
other		12 peaks each < 0.005	Up to 4 peaks each 0.07-1.6	7-12 other peaks	2 peaks 0.002, 0.003	13 peaks each < 0.009-0.012	3 components	10-17 other peaks

Cabbage

The metabolism of ^{14}C-fenpropathrin was investigated in cabbages grown and treated in a greenhouse (Mikami, N. *et al.*, 1985; Reference FM-50-0044). ^{14}C-fenpropathrin labelled at either the cyano group (referred to as [cyano-^{14}C]-fenpropathrin), or the C$_{(1)}$ position of the cyclopropyl ring ([cyclopropyl-^{14}C]-fenpropathrin), or the phenoxyphenyl ring ([phenoxyphenyl-^{14}C]-fenpropathrin) were prepared (all radio-chemical purities > 99%). Each of the preparations were dissolved in methanol and evenly applied to the upper surface of two 3rd–4th leaves of cabbage seedlings at a rate of 22 µg per leaf (*ca*. 25cm^2, equivalent to 0.09 kg ai/ha). The cabbages were sampled immediately after application and at 3, 7, 14, 21, 28, 35 and 42 days after application.

The cabbage samples were separated into the treated leaves and non-treated shoot portion. The treated leaves were rinsed twice with methanol and the leaves and the untreated shoots were separately homogenized and extracted with a solution of methanol:chloroform:distilled water (4:2:1). Each of the rinse and extract was radioassayed and analysed by TLC. 97.3% of fenpropathrin was recovered from cabbages immediately after application of [cyano-^{14}C]-fenpropathrin. Direct TLC comparison of metabolites from three labelled preparations was used to distinguish products retaining the ester linkage from hydrolysis products. The extractable components in cabbages harvested 28 and 42 days after application are shown in Table 30.

Table 30 Extractable components in cabbage samples harvested 28 and 42 days after application.

	% of the applied ^{14}C					
	Cyclopropyl-^{14}C		^{14}CN		phenoxyphenyl-^{14}C	
	28	42	28	42	28	42
Surface Wash:						
Fenpropathrin	0.6	0.3	1.0	0.6	1.7	0.4
Others	0.3	0.1	0.4	0.3	0.3	0.1
Surface Wash Total	**0.9**	**0.4**	**1.4**	**0.9**	**2.0**	**0.5**
Extracts:						
Fenpropathrin	15.8	11.7	16.9	6.0	12.9	11.3
CONH$_2$-Fenpropathrin	0.7	< 0.1	0.3	< 0.1	0.9	< 0.1
COOH-Fenpropathrin	0.4	0.4	0.6	0.3	0.7	< 0.1
2'-OH-Fenpropathrin	0.1	< 0.1	0.4	< 0.1	0.4	< 0.1
Fenpropathrin-CH$_2$OH	0.4	0.3	0.5	0.5	0.6	0.2
TMPA-lactone	0.1	< 0.1	-	-	-	-
TMPA-CH$_2$OH-lactone	0.8	0.9	-	-	-	-
COOH-Fenpropathrin-conjugate	0.4	0.2	0.6	0.4	0.6	0.7
2'-OH-Fenpropathrin-conjugate	0.2	0.1	0.1	0.1	0.1	0.2
4'-OH-Fenpropathrin-conjugate	1.3	0.7	1.6	1.0	0.6	0.8
Fenpropathrin-CH$_2$OH-conjugate	3.5	4.0	3.4	4.3	4.2	4.0
2'-OH-fenpropathrin-CH$_2$OH-conjugate	4.8	4.5	4.6	4.5	5.9	6.2
4'-OH-fenpropathrin-CH$_2$OH-conjugate						
2'-OH-fenpropathrin-(CH$_2$OH)$_2$-conjugate	20.3	22.0	18.6	20.7	19.4	21.6
4'-OH-fenpropathrin-(CH$_2$OH)$_2$-conjugate						
TMPA-conjugate	0.9	0.8	-	-	-	-
TMPA-CH$_2$OH-conjugate	1.1	1.0	-	-	-	-
TMPA-COOH-conjugate	3.7	4.2	-	-	-	-
TMPA-CH$_2$OH-lactone-conjugate	11.3	11.1	-	-	-	-
PBalc-conjugate	-	-	-	-	0.1	0.1
PBacid-conjugate	-	-	-	-	0.8	1.1
2'-OH-PBacid-conjugate	-	-	-	-	6.9	7.4
4'-OH-PBacid-conjugate	-	-	-	-	4.5	4.6
Others	5.2	5.8	4.2	5.8	9.6	9.1
Extracts Total	**71.0**	**67.7**	**51.8**	**43.6**	**68.2**	**67.3**
Unextractable ^{14}C Total	2.6	5.1	6.7	11.3	4.0	7.5
Treated Leaves Total	74.5	73.2	59.9	55.8	74.2	75.3

	% of the applied ^{14}C					
	Cyclopropyl-^{14}C		^{14}CN		phenoxyphenyl-^{14}C	
	28	42	28	42	28	42
Untreated Shoots	0.9	1.2	0.6	0.7	0.4	0.4
Overall Total	**75.4**	**74.4**	**60.5**	**56.5**	**74.6**	**75.7**

The study demonstrated that after foliar application of ^{14}C-fenpropathrin to cabbages the radiocarbon remaining on the treated leaves (as shown by the surface wash) decreased with concomitant increase of ^{14}C in the plant tissues. Most of the recovered radiocarbon was in the treated leaves and less than 1.2% of the applied radiocarbon was found in the untreated shoots indicating that fenpropathrin and its metabolites hardly translocate from the application site to other parts of the plant.

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

Fate of hydrogen cyanide (HCN) and TMPA in abscised leaves

Mikami, N. *et al.*, also conducted work on the fate of HCN and 2,2,3,3-tetramethylcyclopropanecarboxylic acid (TMPA) in abscised leaves of apple, kidney bean, cabbage, mandarin orange, tomato and vine. 2,2,3,3-tetramethylcyclopropanecarboxylic acid labelled at the $C_{(1)}$ position of the cyclopropyl ring (^{14}C-TMPA) was prepared. Two abscised leaves from each plant were placed in 100 mL distilled water containing ^{14}C-TMPA at a concentration of 1.0 ppm. After cultivation for five days the leaves were extracted with methanol:chloroform:water (4:2:1).

In a separate experiment, abscised leaves of cabbage and bean plants were placed in a 100 ppm solution of ^{14}C-TMPA in order to obtain large quantities of metabolites for characterization. The extracts were partitioned between ethyl ether and distilled water. After acidification the aqueous layer was partitioned with ethyl acetate. The extractable components in abscised leaves of various plants over a 5 day period are shown in Table 31.

Table 31 Extractable components in abscised leaves of various plants over a 5 day period.

	% of the applied ^{14}C					
	Apple	Bean	Cabbage	Orange	Tomato	Vine
Extracts						
TMPA	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
TMPA-Gu	21.5	14.7	3.2	3.0	2.0	6.6
CH_2OH-TMPA-Gu	5.2	3.2	3.4	0.2	0.2	0.4
TMPA-CH_2OH-Gu	1.3	-	-	-	-	0.1
TMPA-Gu-Gu	5.4	2.8	-	-	12.8	0.7
TMPA-malonyl-Gu	0.7	56.2	70.5	3.1	4.3	0.1
CH_2OH-TMPA-malonyl-Gu	-	-	1.5	0.4	-	-
Others	1.5	3.0	0.2	0.5	1.1	0.2
Extracts Total	**35.6**	**79.9**	**78.8**	**7.2**	**20.4**	**8.1**
Unextractable ^{14}C Total	0.3	0.2	0.2	0.4	0.2	0.3

	% of the applied ^{14}C					
	Apple	Bean	Cabbage	Orange	Tomato	Vine
Abscised Leaves Total	**32.9**	**80.1**	**79.0**	**7.6**	**20.6**	**8.4**
Aqueous Solution						
TMPA	57.1	24.5	13.2	63.8	76.1	83.0
Others	1.2	0.8	0.9	0.7	1.6	0.5
Aqueous Solution Total	**58.3**	**25.3**	**14.1**	**64.5**	**77.7**	**83.5**
Overall Total	**94.2**	**105.4**	**93.1**	**72.1**	**98.3**	**91.9**

TMPA was readily converted in plants to more polar products. The metabolic pathways for TMPA varied dependent upon species of plant. The glucose ester was a main product in apple and vine plants. In orange, cabbages and bean plants, the malonylglucoside was mainly formed.

Further work was carried out using $K^{14}CN$. Two abscised cabbage leaves were treated for four hours with distilled water containing $K^{14}CN$ and then transferred to $K^{14}CN$-free distilled water. The treated leaves were extracted at specific intervals after dosing with $K^{14}CN$, and the extracts were subject to TLC. There was a gradual increase in the amount of volatile ^{14}C trapped in NaOH solution, most of the radiocarbon was considered to be $^{14}CO_2$. At least six ^{14}C metabolites were present in the extracts of the abscised leaves treated with $K^{14}CN$. The extractable components in abscised leaves of cabbage over a 2 day period are shown in Table 32.

Table 32 Extractable components in abscised leaves of cabbage over a 2 day period following treatment with $K^{14}CN$.

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

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

Summary of plant metabolism

Metabolism of fenpropathrin has been studied in apples, tomatoes (fruiting crops), beans (legume crops), cotton (pulses/oilseed crops) and cabbage (leafy crops). Trials were carried out at approximately recommended rate with fenpropathrin labelled in the phenoxyphenyl, benzyl, cyclopropyl or cyano positions. The majority of radioactivity was found in leaf samples. Low levels of radioactivity were found in fruit/beans. Parent fenpropathrin was the primary component of the residues in the fruits of the plants, but degradation products constituted the greater part of the residues present in the leaves. Breakdown products/metabolites detected in both fruits and leaves were similar to those detected in rats. It is therefore considered that crop residues are described adequately by defining them as the parent product alone.

The study on cabbage further demonstrated that after foliar application of ^{14}C-fenpropathrin the radiocarbon remaining on the treated leaves (as shown by the surface wash) decreased with concomitant increase of ^{14}C in the plant tissues. Most of the recovered radiocarbon was in the treated leaves and less than 1.2% of the applied radiocarbon was found in the untreated shoots indicating that fenpropathrin and its metabolites hardly translocate from the application site to other parts of the plant.

The general pattern of degradation in all the plant studies include breaking of the ester linkage to produce 3-phenoxybenzoic acid (PBacid) and the corresponding alcohol (PBalc) and aldehyde (PBald). From the acid side of the molecule, the main metabolite is 2,2,3,3-tetramethylcyclopropanecarboxylic acid (TMPA) which can give rise to 2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylic acid (TMPA-CH_2OH) and 5-hydroxymethyl-6,6-dimethyl-3-oxabicyclohexan-2-one (TMPA-CH_2OH lactone). PBacid can be hydroxylated at various positions on the phenoxy ring to produce, α-cyano-3-(2’-or 4’-hydroxyphenoxy)benzyl 2,2,3,3-tetramethylcyclopropanecarboxylate (2’-or 4’-OH-fenpropathrin). The proposed metabolic pathway of fenpropathrin in plants is shown in Figure 2.

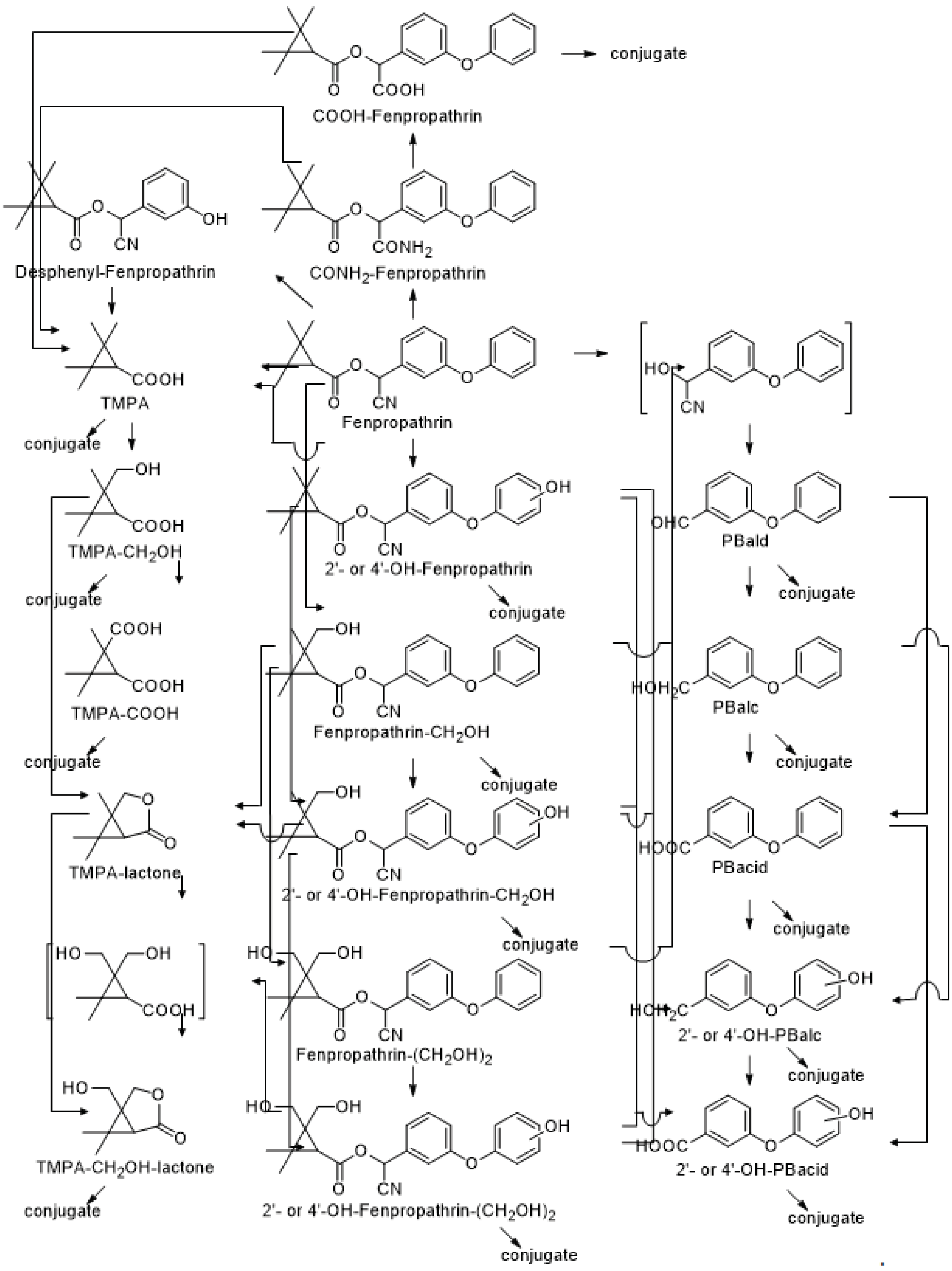


Figure 2 Metabolism and biotransformation pathway for fenpropathrin in plants

Environmental Fate in Soil

The fate and behaviour of fenpropathrin in soils was investigated using fenpropathrin labelled with ^{14}C either in the cyano group, the C-1 position of the cyclopropyl ring, the benzylic carbon or uniformly in the phenoxyphenyl ring. The reports had been reviewed by the 1993 JMPR, no new study was submitted.

Aerobic soil metabolism

A 365-day aerobic soil metabolism study was conducted in 1989 using [benzyl-^{14}C]-fenpropathrin (Cranor, 1990, Reference FM-91-0039). The test material was incubated in the dark at 25 °C with silt loam soil confirmed to be biologically active by plating and counting colonies. The nominal concentration of fenpropathrin was 10 µg/g soil; the measured concentration immediately after the addition was 10.2 µg/g. The soils were maintained at 70–75% field capacity throughout the study and sampled at intervals up to the end of the 365-day incubation. The samples were extracted with methanol (3–4 times) and then combusted to determine the un-extracted activity.

After 365 days, 18.4% of the dose remained as parent with accumulated volatiles accounting for 59.9% (99.8% of which was CO2) and un-extractable residues for 17.8%.

During the whole course of the study the maximum levels of the metabolites were 1.25% PBacid (Day 1), 0.21% $CONH_2$-fenpropathrin (Day 122), 0.55% desphenyl-fenpropathrin (Day 122), 0.19% 4'-OH-fenpropathrin (Day 122) and 0.39% COOH-fenpropathrin (Day 61), all in terms of per cent of the initial dose. In addition there were maxima of 0.07 (Day 14), 0.28 (Day 365), 0.34 (Day 61) and 0.16% (Day 30) of unidentified products. The ^{14}C mass balance for the whole period ranged from 98.7% to 107.1% with a mean of 102.3% and a value at the end of the study of 98.7%. The half-life was calculated, using a first order model, to be 152 days. These results were similar to those of previous degradation studies.

The production of labelled carbon dioxide was greatest from the cyano label over 208 days, and least from the phenoxyphenyl label (at day 148), indicating the relative readiness of the different parts of the molecule to mineralize. (Noble, 1976, Reference FR-61-0013).

In the soil extracts from the cyclopropyl label, the parent compound was the main component with small amounts of TMPA and unidentified polar compounds and, in one of the perfusions, the amide of fenpropathrin ($CONH_2$-fenpropathrin). No amide was found from the cyano label.

In the case of the phenoxyphenyl label, the liberation of carbon dioxide was much slower and the rates were erratic. Neither $CONH_2$-fenpropathrin nor PBacid was detected in the soils. In the perfusion mixtures, the main components isolated were the parent and unidentified hydrophilic compounds.

In the aerobic soil after 16 weeks, the most important component of the residue was the parent compound. The half-life of the fenpropathrin was about 4 weeks on moist soils (Leiston and moist Brenes) but more than 16 weeks on the drier ones (dry Brenes and Los Palacios). After 16 weeks the most prominent metabolite in drier soil was PBacid in the case of the phenoxyphenyl label and TMPA in the case of the cyclopropyl label (Roberts and Standen, 1976, Reference FR-61-0014). The other two metabolites found were $CONH_2$-fenpropathrin and COOH-fenpropathrin, but these were scarcely detectable in the soils with high moisture (Brenes at 16% moisture and Leiston). In the study using Brenes soils, to detect volatile activity, the percentage of the applied radioactivity evolving as CO_2 after 26 weeks was 16.0%, with 71.8% as the intact parent compound and 11.6% as unextracted activity. Other degradation products were only present at negligible levels.

Mikami *et al*., 1983 (Reference FM-30-0008) investigated two fresh Japanese soils, a sandy clay loam (Azuchi) and a light clay with 15% organic matter (Kodaira), under three different conditions: natural aerobic soil, anaerobic soil, and autoclaved aerobic soil. Under aerobic conditions the half-life of fenpropathrin was 11 and 17 days and after 24 weeks the level of fenpropathrin had declined from 1 mg/kg to 0.025 and 0.040 mg/kg respectively. After 8 weeks approximately 0.93 mg/kg remained under sterilized conditions. Sterilization retarded degradation, demonstrating the importance of biological processes. While there were only minor indications of degradation products in the sterile soils, at least 7 were detected in the methanol extracts of the non-sterile soil. With the cyclopropyl-labelled compound the main components of radioactivity were the parent fenpropathrin, desphenyl-fenpropathrin, 4’-OH-fenpropathrin, and small amounts of $CONH_2$-fenpropathrin and COOH-fenpropathrin. There were also indications of very small amounts of unidentified products. In the case of the phenoxyphenyl label, very low levels of PBacid were observed during the first 4 weeks. Un-extractable residues at the end of the study reached 44–45% of the added radioactivity in the Kodaira soil (the one containing a high proportion of organic matter), but only 24–32% in the lower-organic Azuchi soil. No TMPA was reported. A major part of the lost radioactivity was recovered as labelled CO_2 in fairly similar amounts from both labels.

Photodegradation in soil

Photodegradation studies carried out in Japan on light clay, sandy loam and sandy clay loam with fenpropathrin labelled with ^{14}C in the cyano group, the phenoxyphenyl ring or the C-1 position of the cyclopropyl ring and applied to thin-layer soil plates (Takahashi *et al*., 1983, 1985, Reference FM-30-0011). The fenpropathrin was applied at a rate of 1.1 $\mu g/cm^2$ and the plates exposed to natural sunlight for 14 days. Dark controls were run at the same time. Under irradiation, the half-lives of the CN-labelled fenpropathrin were between 1–5 days on the three soils. The mean fenpropathrin residues left in the soils at the end of the 14-day period amounted to 5.1, 29.4 and 32.9% of the amounts applied. The corresponding figures in the dark controls were 74, 84 and 96%; there was insufficient degradation for half-lives to be estimated. The main degradation product under irradiation with all three labels was $CONH_2$-fenpropathrin which reached a maximum in the three soils after 5, 7 and 7 days. Substantial amounts also occurred in the dark controls. For the most part, other metabolites were present in the irradiated soils in only very small amounts, except PBacid which reached a maximum of 11.4% of the total applied radioactivity after 7 days. Minor metabolites found were COOH-fenpropathrin, desphenyl-fenpropathrin, and 3-OH-Bacid. Loss of radioactivity from soils was greatly enhanced by irradiation. The nitrile carbon was the most susceptible followed closely by the C-1 of the cyclopropyl group. The phenoxyphenyl group was a more stable part of the molecule.

Summary: Aerobic soil metabolism

Studies on the metabolism of fenpropathrin in soil demonstrated that fenpropathrin is degraded in the soil by a combination of photochemical and microbiological processes. Metabolism proceeds via cleavage of the ester bonds, hydroxylation, and hydrolysis of the cyano group to $CONH_2$ and COOH groups. The major products detected after aerobic metabolism were the parent fenpropathrin, CO_2, and non-extractable residues. Metabolites included desphenyl-fenpropathrin, 4'-OH-fenpropathrin, phenoxybenzoic acid, and $CONH_2$-fenpropathrin, which was further degraded to COOH-fenpropathrin. The proposed metabolic pathway of fenpropathrin in soil is shown in Figure 3.

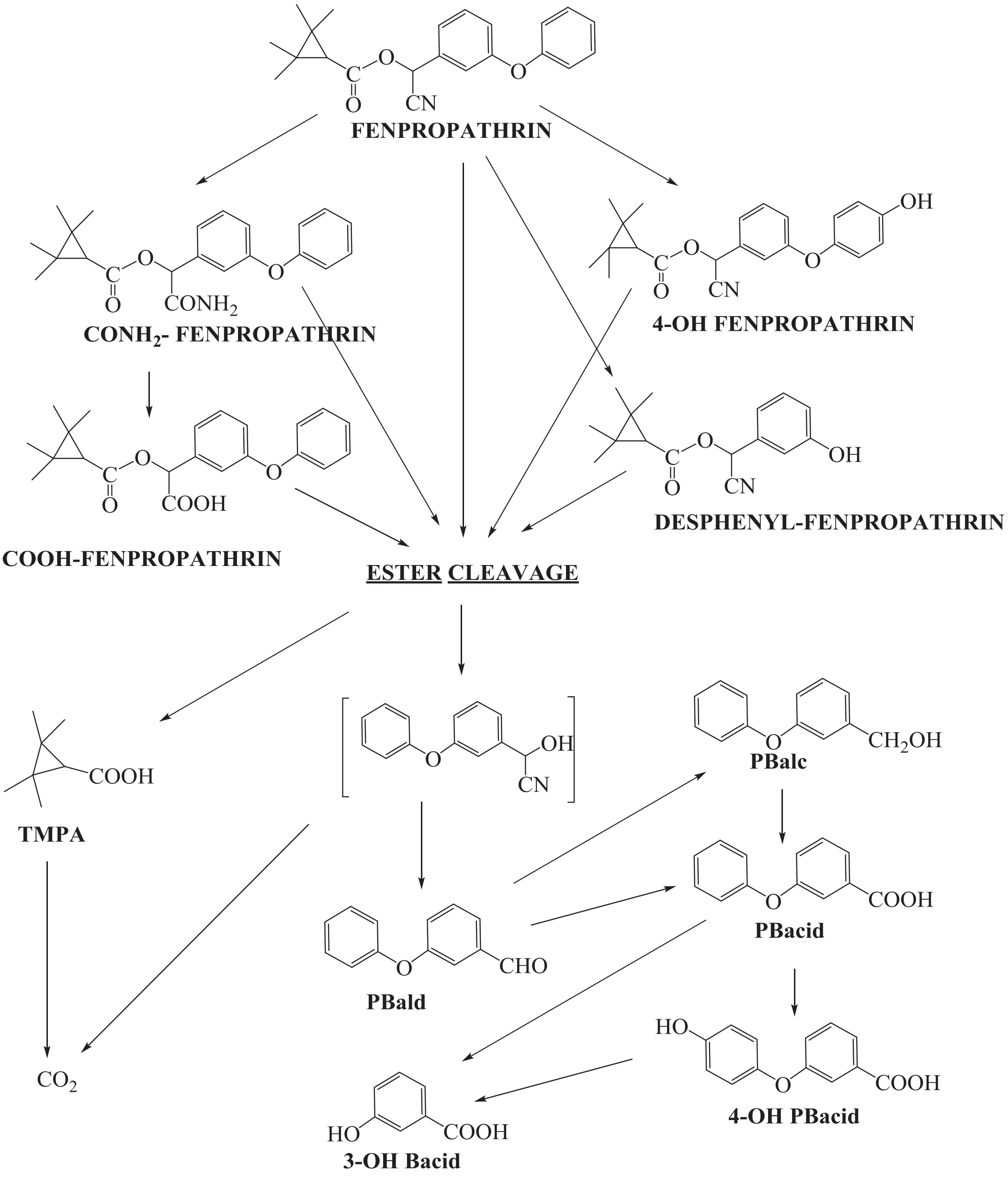


Figure 3 Metabolism and biotransformation pathway for fenpropathrin in soil

Residues in Succeeding or Rotational Crops

The crops included in this evaluation are permanent or semi-permanent crops, for which rotational crop studies are not required. No study was submitted for evaluation.

Residue analytical methods

Analytical methods have been developed for determination of residues of fenpropathrin in plant and animal. In general, the methods involve solvent extraction, clean-up by either silica gel or Florisil column, GLC using electron capture detection. The main variations depending on the substrates are in the extraction and clean-up procedures. Fruits and vegetables may be homogenized with water, shaken with acetone, and extracted with dichloromethane, using NaCl to minimize emulsification. After drying with anhydrous sodium sulfate and clean-up by silica gel column chromatography, the solvent is evaporated at <40 ºC and the residue dissolved in acetone before estimation by gas chromatography with electron capture detection. Other extraction procedures involve direct extraction of the homogenized material suspension in water or homogenization with methanol instead of water. The results of recovery studies carried out as part of method validation or concurrent recoveries obtained from supervised trial samples are summarised in Table 33.

Analytical methods for plant matrices

The base method, RM-22-4 consolidates RM-22-2 (for high moisture crops), RM-22-3 (for oily crops) and includes a procedure for processed oil and soapstock (Lai and Fujie, 1983; Reference FA-41-0040). Homogenized samples are extracted with hexane/acetone and cleaned up through silica gel and C18 solid phase extraction (SPE). Additional purification using gel permeation chromatography (GPC) are performed for oily matrices. Soapstock samples are dissolved in water, extracted with dichloromethane, before proceeding with the processed oil method which involves partition between hexane and acetonitrile and clean-up using alumina oxide. Analysis is by GC with electron capture detection (ECD) (column 3% OV 101 - high moisture and oily samples; DB-1 – processed oil and soapstock samples).

Method used for citrus fruit, pome fruit, grapes and their processed fractions

Citrus fruit samples were analysed using method RM-22-4, with modifications. Homogenized samples were extracted with hexane/acetone, partitioned into hexane and cleaned up with silica gel and C18 solid phase extraction (SPE). Residues of fenpropathrin were determined by gas chromatography with electron capture detection. The LOQ for the method was 0.01 mg/kg.

Method used for raspberry

Raspberry samples were analysed using the revised method RM-22-4 (revision May 1993), with modifications. Homogenized samples were extracted with hexane/acetone, partitioned into hexane and cleaned up with silica gel and C18 solid phase extraction (SPE). Residues of fenpropathrin were determined by gas chromatography with mass spectrometry (MS)-SIM. The LOQ for the method was 0.02 mg/kg.

Method used for tomato and processed fractions, cucumber, melon and squash

Method RM-22-4 was used for the analysis of fenpropathrin in fruiting vegetables, cucurbits and fruiting vegetables other than cucurbits. The LOQ was 0.01 mg/kg. A revised RM-22-4 method (revision May 1993) using a DB-17 column and nitrogen phosphorus detection (NPD) was used for certain raw and processed tomato samples.

Method used for tea

Method RM-22-2 was revised for analysis of fenpropathrin in tea. Tea samples were extracted with an acetonitrile-water mixture and the extracts filtered under suction (Lavakumar, S., *et al*., 2003; Report No. 11861 and Lavakumar, S., *et al*., 2004; Report No.14246). The filtrate was concentrated then diluted with 5 percent aqueous sodium chloride solution and subjected to extraction with hexane-ether mixture. The combined extracts were filtered through sodium sulfate and evaporated to near dryness before being dissolved in 5 mL hexane. Clean-up was performed and the eluate evaporated and dissolved in acetone. Fenpropathrin was quantified by gas chromatography using an electron capture detector. The LOQ is 0.05 mg/kg.

Methods used for soya beans

Method RM 22-4 was modified for soya bean determination. Soya bean seeds were extracted with acetonitrile-ethyl acetate mixture, centrifuged and cleaned up through silica gel column with hexane. The extract was eluted with ethyl acetate-hexane mixture, evaporated to dryness and re-suspended in hexane. Fenpropathrin residues were determined by gas chromatography using a micro electron capture detector (Tomaz, 2013; Reference FR-0570). The LOQ for the method is 0.01 mg/kg.

In the other soya bean study, residues were extracted with acetonitrile and salt mixture and fenpropathrin analysed by liquid chromatography with mass spectrophotometric detection (LC/MS/MS) Lopez, 2012; Reference FR-0571). The LOQ for the method was 0.01 mg/kg.

Methods used for cotton matrices

Method RM 22-4 was modified for cotton matrix determination. Fuzzy seed and processing fractions were extracted with hexane-acetone and cleaned up using gel permeation chromatography or partitioned into acetonitrile for oil removal with clean up through silica gel and C-18 Sep Pak columns. For cotton seed oil, samples were dissolved in hexane, portioned into acetonitrile and cleaned up by alumina oxide column chromatography. For soapstock, samples were dissolved in water, partitioned into dichloromethane, partitioned between hexane and acetonitrile and cleaned up by alumina oxide column chromatography. Fenpropathrin residues were determined by gas chromatography using an electron capture detector (Fujie, 1990; Reference FR-01-0306). The LOQ for the method is 0.01 mg/kg.

In the other soya bean study, residues were extracted with acetonitrile and salts mixture and fenpropathrin analysed by liquid chromatography with mass LC-MS/MS detection *utilising the transition of mz 350→125*. The LOQ for the method is 0.01 mg/kg (Lopez, 2012; Reference FR-0571).

Method used for coffee beans

Coffee samples were extracted with water and 0.1% acetic acid in acetonitrile and a mixture of salts (anhydrous magnesium sulfate, sodium acetate and sodium chloride are added and the mixture centrifuged. An aliquot of the supernatant was transferred to a tube containing anhydrous magnesium sulfate and centrifuged. The extract was diluted in methanol:water (20:80) and residues of fenpropathrin were analysed by LC-MS/MS. The LOQ was 0.01 mg/kg (Gravena, 2013; Reference FR-0572)

Analytical methods for animal matrices

Method RM-22A-1 was used to analyse for fenpropathrin residues in milk and animal tissue samples in the feeding study (Fujie, 1986; FR-61-0174 and FR-61-0175). In summary, the method involves extraction with hexane/acetone, acetonitrile/hexane partitioning, silica gel column chromatography and measurement by gas chromatography using the electron capture detector. The method was validated with an LOQ of 0.01 mg/kg fenpropathrin. Control samples fortified with fenpropathrin were analysed together with treated samples from the feeding studies and the concurrent recoveries were within the acceptable range of 70–120%.

Multi-residue Methods

The multi-residue method DFG S19 was investigated and validated for the determination of fenpropathrin in acidic plant matrices high water content (tomatoes and oranges) (Class, 2003; Reference FA-0093). Samples were extracted with acetone/water, following neutralization of the pH in the case of oranges. Extracts were partitioned with ethyl acetate/cyclohexane in the presence of sodium chloride and cleaned up using gel permeation chromatography (GPC) and silica gel. Analysis was by GC-MS (ions mz 181 and 265 for quantitation and 209 and 349 for confirmation). Recoveries (range and mean) and precision were within the acceptable limits. Linearity was demonstrated. There were no interfering peaks at the retention time for fenpropathrin in any of the crop matrices. The method is suitable for monitoring pesticide residues.

The multi-residue method DFG S19 was also validated for the determination of fenpropathrin in foodstuffs of animal origin (milk, bovine meat and fat, eggs) (Class, 2003; Reference FA-0094). Whole milk, egg and bovine meat samples were extracted with acetone/water and the extracts were partitioned with ethyl acetate/cyclohexane in the presence of sodium chloride. Fat samples were extracted with ethyl acetate/cyclohexane. Extracts were cleaned up using gel permeation chromatography (GPC) and silica gel. Analysis was by GC-MS (ions mz 181 and 265 for quantitation and 125, 152, 209 and 349 for confirmation). Recoveries (range and mean) and precision were within the acceptable limits. Linearity was demonstrated. There were no interfering peaks at the retention time for fenpropathrin in any of the animal matrices. The validated LOQ is 0.01 mg/kg for animal tissues and eggs. The method can be used for enforcement of MRLs and monitoring of residues.

A summary of recovery data from the methods used for plant and animal commodities supported in this submission are presented in the table 33.

Table 33 Summary of method validation and concurrent recovery data [a]

Commodity	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Plant commodities							
Almond hulls	0.02-7.5	6	90-115	103	8.9	RM-22-4	FR-0382
Almond nutmeat	0.02-0.1	6	84-119	96	14	RM-22-4	FR-0382
Apples	0.1 0.1 0.1 1.0	9 6 12 1	89-111 87-103 73-119 106	98 97 98 -	8 6 12 -	RM-22-4	FR-81-0305 FR-61-0190 FR-0370 FR-81-0305
Apple juice	0.1 0.1	1 12	106 73-96	- 87	- 12	RM-22-4	FR-81-0305 FR-0370
Apple wet pomace	0.2 1.0-4.0	1 4	109 75-98	- 89	- -	RM-22-4	FR-81-0305 FR-0370
Apple dry pomace	0.2 0.4-8.0	1 4	96 71-126	- 92	- 26	RM-22-4	FR-81-0305 FR-0370
Bell pepper	0.02-1.0	10	94-107	101	4	RM-22-4	FR-0558
Canned tomatoes	0.01-0.2	3	94-100	97	3	RM-22-4	FR-21-0344
Cherry	0.02-4.0	8	82- 110	99	9.6	RM-22-4	FR-0381
Coffee	0.01-0.5	13	79-116	98	15.3	LC-MS/MS	FR-0572
Cotton, fuzzy seed	0.1-0.2	38	70-101	83	-	RM-22-4	Fr-01-0306
Cucumber	0.01-11.4	15	66-115	89	13.8	RM-22-4	FR-0556
Grape	0.1 0.1 1.0 0.02 0.4	4 22 1 1 1	93-102 77-123 110 98 97	99 99 - - -	4 12 - - -	RM-22-4	FR-01-0308 FR-010335 FR-0374
Grape wet pomace	0.1 0.2 0.5 0.5	3 1 4 8	100-101 95 97-106 100	100 - 101 -	0.7 - 4 4	RM-22-4	FR-01-0308
Grape dry pomace	0.1 0.2 0.5 1.0 1.0	3 2 2 4 11	86-121 84-96 100-119 95-108 100	101 90 109 100 -	18 - - 6 12	RM-22-4	FR-01-0308
Grape juice	0.1 0.005 0.1	9 1 1	87-101 98 98	96 - -	5 - -	RM-22-4	FR-01-0308 FR-01-0335
Grapefruit	0.02 0.1 0.5	2 9 7 18	106-114 85-108 90-99	110 95 94 96	- 7 3 7	RM-22-4	FR-21-0343
Lemon	0.02 0.1 0.5	3 8 5 16	98-120 92-117 90-108	109 101 99 102	10 9 7 9	RM-22-4	FR-21-0342
Melon	0.02-1.0	16	82-98	90	4.8	RM-22-4	FR-0375
Non-bell pepper	0.02-10	12	88-115	97	8	RM-22-4	FR-0558

Commodity	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Olive fruit, no pit	0.02-15	15	70- 121[1/]	91	9.9	RM-22-4	FR-0561
Olive fruit, no pit	0.02-5.0	7	69-103[1/]	88	13.9	RM-22-4	FR-0561
Olive oil	0.02-20	12	76-115[1/]	96	13.0	RM-22-4	FR-0561
Olive oil	0.02-0.10	4	64-81[1/]	73	9.9	RM-22-4	FR-0561
Orange	0.01 – 0.1	10	79-109	98	9	DFG S19	(FA-0093)
Orange	0.01- 0.1	10	79-104	95	7	DFG S19	(FA-0093)
Orange	0.1	34	77-115	96	7	RM-22-4	FR-01-0307
Orange dried peel/fines	0.1	2	90-92	91	-	RM-22-4	FR-01-0307
	1.0	4	77-100	90	11		
Orange juice	0.1	3	97-98	97	0.6	RM-22-4	FR-01-0307
Orange molasses	0.1	3	90-94	92	2	RM-22-4	FR-01-0307
Orange oil	0.4	1	81	81	-	RM-22-4	FR-01-0307
	1.0	2	86-100	93	-		
	2.0	1	86	86	-		
		4		88	9		
Orange peel (wet)	0.1	1	91	91	-	RM-22-4	FR-01-0307
	1.0	4	96-102	100	3		
Orange pulp	0.1	2	82-98	90	-	RM-22-4	FR-01-0307
Peach	0.02-1.0	10	91-113	101	6.4	RM-22-4	FR-0384
Pear	0.1	5	91-102	97	4	RM-22-4	FR-0369
	0.1	7	74-109	95	12		
	0.1	14	94-117	99	6		
Pear pulp	0.1	2	98-101	100	-	RM-22-4	FR-0369
Pear skin	0.5	2	105-108	106	-	RM-22-4	FR-0369
Pear puree	0.1	1	99	-	-	RM-22-4	FR-0369
Pecans	0.02-0.1	6	71-90	82	9.3	RM-22-4	FR-0383
Plum	0.02-0.6	7	97-109	101	4.6	RM-22-4	FR-0385
Plum	0.05-2.0	4	96- 101	99	2.5	RM-22-4	FR-0559
Plum, dried	1.0, 10	2	101, 104	103	-	RM-22-4	FR-0385
Plum, dried	0.1, 2.0	2	94, 99	97	-	RM-22-4	FR-0559
Raisin/raisin waste	0.1	2	82-107	94	-	RM-22-4	FR-01-0308
	0.2	3	81-103	94	12		
	0.5	2	103-104	103	-		
	0.5	7	97	-	11		
Raspberry	0.02-10	10	74-110	88	-	RM-22-4	FR-0560
Soya beans	0.025, 0.25	2	74, 74	74	-	GC-ECD	FR-0570
Soya beans	0.01-0.1	10	77-100	90	8.7	LC-MS/MS	FR-0571
Squash	0.01-11.4	9	85-116	97	11.5	RM-22-4	FR-0557
Strawberry	0.1-0.5	27	81-115	98	8.2	RM-22-4	FR-11-0345
Tea	0.05- 2.0	16	88-93	90	2	RM-22-4	04-2974-2002; 04-4190-2004
Tomato	0.01-0.1	10	67-112	100	13	DFG S19	FA-0093
Tomato	0.01– 0.1	9	96-111	103	5	DFG S19	FA-0093
Tomato dry pomace	0.1	3	75-118)	96	23	RM-22-4	FR-41-0360
Tomato juice	0.1	3	100-101	101	0.7	RM-22-4	FR-41-0360
Tomato paste	0.1	3	100-105	102	3	RM-22-4	FR-41-0360
Tomato wet pomace	0.1	3	100-102	101	1	RM-22-4	FR-41-0360
Tomato, green	0.01-0.2	16	86-130	99	11	RM-22-4	FR-21-0344
Tomato, ripe	0.02-0.2	18	94-102	98	2	RM-22-4	FR-41-0360
Tomato, ripe	0.02- 0.2	10	85-113	95	10	RM-22-4	FR-21-0344
Foodstuffs of animal origin							
Bovine Fat	0.1	1	100	-	-	RM-22A-1	FR—61-0174
Bovine fat	0.01-0.1	10	72-117	94	14	DFG S19	FA-0094
Bovine fat	0.01-0.1	10	80-133	103	14	DFG S19	FA-0094
Bovine Kidney	0.1	3	81-93	88	6.8	RM-22A-1	FR—61-0174
Bovine Liver	0.1	1	114	-	-	RM-22A-1	FR—61-0174
Bovine muscle	0.01- 0.1	10	82-99	89	7	DFG S19	FA-0094
Bovine muscle	0.01-0.1	10	65-106	86	18	DFG S19	FA-0094
Bovine Muscle	0.1	1	-	94	-	RM-22A-1	FR—61-0174
Milk fat	0.4	1	104	-	-	RM-22A-1	FR—61-0174
Poultry fat	0.1	1	94	-	-	RM-22A-1	FR-61-0175
Poultry gizzard	0.1	1	94	-	-	RM-22A-1	FR-61-0175

Commodity	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Poultry liver	0.1	1	94	-	-	RM-22A-1	FR-61-0175
Poultry muscle	0.1	1	93.4	-	-	RM-22A-1	FR-61-0175
Whole Egg	0.1	6	94-102	96	3.6	RM-22A-1	FR-61-0175
Whole egg	0.01-0.1	10	93-102	99	3	DFG S19	FA-0094
Whole egg	0.01-0.1	10	71-105	99	10	DFG S19	FA-0094
Whole milk	0.1	12	83-119	96	9.4	RM-22A-1	FR—61-0174
Whole milk	0.01-0.1	10	85-93	89	3	DFG S19	FA-0094
Whole milk	0.01-0.1	10	77-105	96	8	DFG S19	FA-0094

[a] Data presented are only for registered uses and from studies submitted in this document.

Storage Stability under Frozen Conditions

The stability of fenpropathrin residues in commodities under frozen conditions has been investigated in apples, orange, cotton, pears, grapes, tomato and its processed products as well as in products of animal origin.

In addition to these individual studies, storage stability determinations, together with procedural recovery analyses were also carried out concurrently with the field trial samples for strawberry, olives (fruit and oil), cucumber, squash, melon, peppers, milk, eggs and kidney. All these storage stability studies are summarized below. For samples analysed within 30 days of harvest (soya beans, coffee beans, eggs and poultry tissues), storage stability requirements have been waived. The results are summarised in Table 34.

In general, results of the storage stability tests confirmed that residues of fenpropathrin in stored frozen samples were stable within the period of storage between sampling and analyses.

Apple, orange, cotton, pears and grapes

The stability of field incurred fenpropathrin was investigated in five matrices (Fujie, 1986; Reference FR-61-0190). Samples taken from residues trials conducted with fenpropathrin 2.4 EC were stored at -20 °C and four replicate samples taken at 3, 6, 9 and 12 months during storage. Fortified control samples (0.1 mg/kg) were analysed concurrently with each set of samples to validate analytical method recovery. Homogenized samples were extracted with hexane/acetone and cleaned up through silica gel and C18 solid phase extraction (SPE) (method RM-22-4). Additional purification using gel permeation chromatography (GPC) was performed for oily matrices (method RM-22-4). Analysis was by GC with electron capture detection (ECD).

Orange oil and dried peel

The stability of field incurred fenpropathrin was investigated in orange oil and dried peel samples from a processing study (Fujie, 1990; Reference FR-01-0307). Duplicate samples taken from processing study were stored at -20 °C and samples taken after 11 months storage. Analysis was by methods RM-22-4 and RM-22-2.

Strawberry

The storage stability data for strawberries were obtained by reanalysis of retained samples from a previous field trial (Mitten, 1989; Reference FR-11-0345). Samples harvested on 07.04.89 were received frozen in the laboratory on 18.04.89, macerated on 25.04.89 and were extracted for the initial analysis on 28.04.89. After sub-sampling, the retained samples were stored at -20 °C with periodic subsampling for reanalysis using method RM-22-4. The results demonstrate that fenpropathrin is stable in strawberries up to six months in frozen storage.

Raspberry

The storage stability data for strawberries were obtained by reanalysis of retained samples from a previous field trial (Samoil, 2007; Reference FR-0560). Samples were fortified with 2 mg/kg fenpropathrin then stored frozen for 216 days. Analysis using modified method RM-22-4.

Grape products

The storage stability of fenpropathrin in samples of grape juice, dry and wet pomace, hydrated raisins and raisin waste was determined from incurred residues from the field trial sites (Fujie, 1990; Reference FR-01-0335). Samples were held in frozen storage under similar conditions to the field generated samples.

Olive fruit and olive oil

The storage stability of fenpropathrin in samples of olive fruit without pit and in olive oil was determined by fortifying samples with 2 mg/kg fenpropathrin after receipt from the field trial sites (Samoil, 2007; Reference FR-0561). Samples were held in frozen storage under similar conditions to the field generated samples. After 208 days (olive fruit without pits) and 202 days (olive oil), the storage stability samples were analysed by method RM-22-4.

Cucumber

The stability of fenpropathrin residues in cucumber samples stored frozen under the same conditions as the field trial samples, was determined as part of the field phase of the study (Samoil, 1999; Reference FR-0556). Untreated samples from two field sites were fortified at 11.4 mg/kg fenpropathrin and returned to storage at -20 °C, together with the field trials samples. After 245 and 286 days in storage, the storage stability and field samples were analysed.

Melons

Storage stability of fenpropathrin on melons was determined as part of the field trial study (Green, 1995; Reference FR-0375). Untreated samples fortified at 0.1 mg/kg fenpropathrin were stored at -20°C under the same conditions as the field trial samples. Duplicate analyses were conducted on the day of fortification and again after 187 days of storage.

Squash

Storage stability of fenpropathrin in squash samples was verified by taking untreated control samples, fortifying at 1 and 11 mg/kg with fenpropathrin, and storing in freezers under the same conditions as the field samples. Storage stability samples were analysed after 230 days in storage (Samoil, 1999; Reference FR-0557).

Tomatoes and tomato canning waste

The stability of fenpropathrin was investigated in tomatoes (fortified samples) and tomato canning waste samples (field incurred residues) from a processing study (Lai, 1990; Reference FR-21-0344). Duplicate samples taken from a processing study were stored at -20 °C and samples taken after various time periods up to 6 months storage. Analysis was by method RM-22-4.

Tomato paste, tomato juice, wet and dry tomato pomace

The stability of fenpropathrin was investigated in tomato paste, tomato juice and wet and dry tomato pomace from a processing study (Green, 1994; Reference FR-41-0360). Duplicate control samples taken from a processing study were fortified at 0.1 mg/kg, stored at -20 °C and samples taken after 5 months storage. Analysis was by methods RM-22-4.

Non-bell peppers

Samples from field trials were fortified with fenpropathrin at 10 mg/kg and stored frozen at -25 °C for 323 days (Samoil, 2001; Reference FR-0558). 323

Milk and bovine kidney

The stability of fenpropathrin in milk when stored at -20 °C was determined by fortifying control milk samples bought from a store, with 0.1 mg/kg fenpropathrin and stored at -20 °C along with the milk samples from the residue feeding study (Fujie, 1986; Reference FR-61-0174). The fortified milk samples were analysed at a time interval greater than the storage interval for milk from the feeding study.

To determine the stability of fenpropathrin in tissues, kidney samples from the feeding study were reanalysed following an appropriate interval which reflected the time interval between sacrifice and the initial analyses. The analytical method RM-22A-1 was used to analyse all samples. Reanalysis of kidney samples following an additional 71 days in storage at -20 °C resulted in a mean recovery of 104% of initial analyses results.

Eggs

The stability of fenpropathrin in eggs following storage at -20 °C was determined by fortifying control egg samples bought from the store, with 0.1 mg/kg fenpropathrin and stored at -20 °C along with the egg samples from the residue feeding study (Fujie, 1986; Reference FR-61-0175). The fortified egg samples were analysed at a time interval greater than the storage interval for eggs from the feeding study.

Table 34 Summary of results of storage stability studies

Commodity	Spike level (mg/kg)	Storage interval (months)	Procedural recoveries (%)	Residues remaining (%)
Apple	2.53[a]	3	96±8.2	82
		6		78
		9		91
		12		82
Cotton	0.03[a]	3	96±8.2	133
		6		116
		9		n/a
		12		100
Cucumber	11.4	~9	87±12.6	76-96
Grapes	0.37[a]	3	96±8.2	n/a
		6		89
		9		87
		12		87
Grape juice		14	94-100	100
Dry pomace		14	98-99	93-98
Hydrated raisins		1-11	92-113	77-106
Raisin waste		1-11	80-112	77-94
Wet pomace		5-12	95-104	89-90
Pear	1.2[a]	3	96±8.2	99
		6		77
		9		78
		12		89
Melons	0.1	6	89-96	74
Non-bell peppers	10	0	88-104	88.4-104
		11		91-95
Olive fruit	2	7	78-89	78, 88, 89
Olive oil	2	7	83-90	85, 9ö, 93
Orange	0.25[a]	3	96±8.2	93
		6		120
		9		n/a
		12		88
Orange oil	0.965	11	81-100	130
Orange dry peel	0.12	11	9ö-100	91.5
Raspberry	2	7	74-110	86-88
Strawberry[1]	0.83-0.84[a]	0	98.3±8.2	
		1		91-101
		3		93-98
		6		87-96
Squash	1	7	92.9-94.6	74.2
	11	7	82.4-92.7	91.8
Tomato		6	83-130	100
Tomato paste	0.1	5	100-102	92
Tomato juice	0.1	5	75-118	92
Tomato wet pomace	0.1	5	100-105	102
Tomato dry pomace	0.1	5	100-105	97
Milk	0.1	2.5	96±9.4	100
Kidney	0.155[a]	2.2	88±6.8	104
Eggs	0.1	5	96±3.6	92

[a]: Level of initial residue in retained test portion; n/a = not analysed

USE PATTERN

Fenpropathrin is registered for use on a large number of crops in many countries, but only the registered use patterns for the crops for which supervised trials were provided are summarised in Table 35.

Table 35 Registered uses of fenpropathrin

Crop	Country	Formulation		Application				PHI (days)
		g ai/L or g ai/kg	type	Method	Rate kg ai/ha	Water L/ha	No or/ Season max kg ai/ ha	
Cane fruit	USA	300 g/L	EC	Foliar (by ground or aerial application)	0.22- 0.34	28-94 (aerial); 187 minimum (ground)	0.67 kg ai/ha/ season 14-day intervals	3
Citrus	USA	300 g/L	EC	Foliar (by ground or aerial application)	0.22- 0.45	187 (aerial); 468- 4700 (ground)	0.9 kg ai/ha/ season; 10-day intervals	1
Coffee	Brazil	300 g/L	EC	Foliar (by ground equipment)	0.06-0.12		2	14
Cotton*	USA	300 g/L	EC	Foliar (by ground or aerial application)	0.22- 0.45	28-94 (aerial); 94- 468 (ground)	0.9 kg ai/ha/ season	21
Grapes	USA	300 g/L	EC	Foliar (by ground or aerial application)	0.22- 0.45	230-1870 (air or ground)	0.9 kg ai/ha/ season; 7-day intervals	21
Fruiting vegetables, Cucurbits, including cucumber, muskmelon, honeydew melon, pumpkin, squash, watermelon	USA	300 g/L	EC	Foliar (by ground or aerial application)	0.22- 0.34	28-94 (aerial); 187- 935 (ground)	0.9 kg ai/ha/ season; 7-day intervals	7
Fruiting vegetables other than cucurbits, including tomato, eggplant, peppers	USA	300 g/L	EC	Foliar (by ground or aerial application)	0.22	47- 94 (aerial); 234- 1122 (ground)	0.9 kg ai/ha/ season; 7-days interval	3
Olive	USA	300 g/L	EC	Foliar (by ground equipment)	0.22- 0.34	935	0.9 kg ai/ha/ season; 14-day intervals	7
Pome fruit	USA	300 g/L	EC	Foliar	0.22- 0.45	700- 3740	0.9 kg ai/ha/ season; 10-days interval	14

Crop	Country	Formulation		Application				PHI (days)
		g ai/L or g ai/kg	type	Method	Rate kg ai/ha	Water L/ha	No or/ Season max kg ai/ ha	
Soya beans	Brazil	300 g/L	EC	Foliar (by ground equipment)	0.045		1	30
Stone fruit	USA	300 g/L	EC	Foliar (by airblast equipment)	0.22- 0.45	935- 3740	0.9 kg ai/ha/ season; 10-days interval	3
Strawberry	USA	300 g/L	EC	Foliar (by ground equipment)	0.22- 0.45	935- 2800	0.9 kg ai/ha/ season ≥30-day intervals	2
Tea	India	300 g/L	EC	Foliar (by knapsack sprayer)	0.05- 0.06 kg ai/ha (0.01- 0.015 kg ai/hL)	400- 500		7
Tree nuts	USA	300 g/L	EC	Foliar (by airblast equipment)	0.22- 0.45	468- 3740	0.9 kg ai/ha/ season; 10-days interval	3

Note: GAPs in the USA specify total seasonal rates in addition to individual application rates, therefore the seasonal maximum dose rate is considered primarily in evaluation of supervised trials.

* Do not feed gin trash or treated forage to livestock or allow animals to graze treated fields

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised trials have been conducted to support MRLs for the following crops citrus fruits, peach, plum, strawberries, olives, cucumber, melon, squash, tomato, peppers, soya beans, tree nuts, coffee beans and tea. The results of these supervised trials are summarized in the following tables:

Crop Group	Commodity	Table No.
Citrus fruit	Orange, lemon, grapefruit	36
Pome fruit	Apple, pear	37
Stone fruit	Peach, plum, cherry	38
Berries and other small fruits	Strawberry	39
	Raspberry	40
	Grape	41
Tropical/Subtropical fruits – edible peel	Olives	42
Fruiting vegetables, cucurbits	Cucumber	43
	Melon	44
	Squash	45
Fruiting vegetables other than cucurbits	Tomato	46
	Pepper	47
Pulses	Soya beans	48
Oilseeds	Cottonseed	49

	Cottonseed hulls	53
Tree nuts	Almonds, pecans	50
	Almond hulls	54
Seeds for beverages and sweets	Coffee beans	51
Teas	Tea, green, black	52

In addition to the description and details of the field trials and analytical methods, each report includes a summary of the procedural recoveries, and in many cases, concurrent recoveries in stored frozen samples.

In the trials, where multiple samples were taken from a single plot, the average value is reported. Where results from separate plots with distinguishing characteristics such as different formulations, different crop varieties or different treatment schedules were reported, results are listed for each plot.

Results have not been corrected for concurrent method recoveries. Residues and application rates have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to the maximum GAP have been used for the estimation of maximum residue levels. Those results included in the tables are underlined.

Citrus fruits

A total of 31 supervised trials were conducted in the United States on citrus fruits (18 on oranges, 6 on lemons and 7 on grapefruit). The trials on oranges were carried out from 1984 to 1987 and consisted of one to four applications of an EC formulation containing 300 g/L fenpropathrin, applied at rates ranging from 0.11 to 0.45 kg ai/ha for a total seasonal rate ranging from 0.11 to 1.8 kg ai/ha (Fujie, 1990; Reference FR-01-0307). Duplicate samples of mature oranges, consisting of a minimum of 10 fruits, were collected at various PHIs (0 day to 35 days). In some trials, fenpropathrin was applied pre-planting and sampled at normal harvest, 180 days after application.

The seven supervised trials on grapefruit in 1991 were all according to the US GAP for citrus fruits. The composite samples consisted of 16 fruits.

Upon harvest, all samples were frozen and maintained frozen at -20 ºC until analysis. Samples from the field trials were analysed within this demonstrated period of stability (9 months for oranges, one month for lemons, and two months for grapefruit). All samples were analysed using analytical method RM-22-4 with a limit of detection of 0.01 mg/kg.

Table 36 Residues of fenpropathrin in citrus fruits resulting from supervised trials in the USA

CITRUS	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total kg ai/ha	days		mg/kg	
GAP, USA for citrus fruit	300 EC	0.22-0.45	468-4700	2/0.9	1			
ORANGE								
M335 T-6083 Fresno, CA, 1984 (Navel)	300 g/L	0.113	935	1	3	Whole fruit	0.04, 0.04 (0.04)	Fujie, 1990 (FR-01-0307)
		0.22	935	1	3	Whole fruit	0.07, 0.13 (0.10)	
		0.45	935	1	0	Whole fruit	0.24, 0.40 (0.32)	
					1		0.25, 0.17 (0.21)	
					3		0.23, 0.17 (0.20)	
					7		0.10, 0.20 (0.15)	
					14		0.04, 0.09 (0.07)	
					21		0.06, 0.09 (0.08)	
					28		0.05, 0.12 (0.09)	
					35		0.21, 0.05 (0.13)	
		0.67	935	1	3		0.20, 0.32 (0.26)	
		0.11	935	2/ 0.22	3		0.13, 0.08 (0.11)	
		0.22	935	2/ 0.44	3		0.02, 0.13 (0.08)	
		0.45	935	2/ 0.90	0		0.37, 0.33 (0.35)	
					1		0.28, 0.22 (0.25)	
					3		0.16, 0.27 (0.22)	
					7		0.11, 0.42 (0.27)	
					14		0.27, 0.15 (0.21)	
					21		0.15, 0.19 (0.17)	
					28		0.22, 0.24 (0.23)	
					35		0.08, 0.25 (0.17)	
		0.67	935	2/ 1.34	0		0.51, 0.21 (0.36)	
					1		0.41, 0.32 (0.37)	
					3		0.57, 0.62 (0.60)	
					7		0.40, 0.34	

CITRUS	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total kg ai/ha	days		mg/kg	
							(0.37)	
					14		0.29, 0.43 (0.36)	
					21		0.21, 0.09 (0.15)	
					28		0.15, 0.17 (0.16)	
					35		0.15, 0.42 (0.29)	
					180		< 0.01, < 0.01 (< 0.01)	
M335 T-6085 Delano, CA, 1984 (Navel)	300 g/L	0.22	5612	2/ 0.44	180	Whole fruit	< 0.01,< 0.01 (< 0.01)	Fujie, 1990 (FR-01-0307)
		0.45	5612	2/ 0.90	180	Whole fruit	< 0.01,< 0.01 (< 0.01)	
M335 T-6085 Porterville, CA, 1984 (Navel)	300 g/L	0.22	5612	2/ 0.44	180	Whole fruit	< 0.01,< 0.01 (< 0.01)	Fujie, 1990 (FR-01-0307)
		0.45	5612	2/ 0.90	180	Whole fruit	< 0.01,< 0.01 (< 0.01)	
M335 T-6087 Madera, CA, USA, 1984 (Navel)	300 g/L	0.22	5612	2/ 0.44	180	Whole fruit	< 0.01,< 0.01 (< 0.01)	Fujie, 1990 (FR-01-0307)
		0.45	5612	2/ 0.90	180	Whole fruit	< 0.01,< 0.01 (< 0.01)	
M335 T-6088 Madera, CA, 1984	300 g/L	0.45	5612	1	0	Whole fruit	0.25, 0.26 (0.26)	Fujie, 1990 (FR-01-0307)
					1		0.20, 0.23 (0.22)	
					3		0.08, 0.09 (0.09)	
					7		0.21, 0.20 (0.21)	
					28		0.26, 0.13 (0.20)	
					35		0.19, 0.13 (0.16)	
		0.45	5612	2/ 0.90	1	Whole fruit	0.35, 0.30 (0.33)	Fujie, 1990 (FR-01-0307)
					3		0.25, 0.26 (0.26)	
					7		0.18. 0.21 (0.20)	
					14		0.32, 0.24 (0.28)	
					21		0.23, 0.18 (0.21)	
M335 T-6157 Lindsay, CA, 1984 (Navel)	300 g/L	0.22	2338	2/ 0.22	180	Whole fruit	< 0.01,< 0.01 (< 0.01)	Fujie, 1990 (FR-01-0307)
		0.45	2338	2/ 0.90	180	Whole fruit	< 0.01,< 0.01 (< 0.01)	
R195 T-6389 Fresno, CA, 1985	300 g/L	0.45	3049	2/ 0.90	1	Whole fruit	0.30, 0.21 (0.26)	Fujie, 1990 (FR-01-0307)
					7		0.17, 0.17	

CITRUS	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total kg ai/ha	days		mg/kg	
(Valencia)							(0.17)	
					14		0.21, 0.15 (0.18)	
					21		0.05,0.03 (0.04)	
R195 T-6389 Fresno, CA, USA, 1985 (Navel)	300 g/L	0.45	3002	2/ 0.90	1	Whole fruit	0.14, 0.10 (0.12)	Fujie, 1990 (FR-01-0307)
		0.45	3002	3/ 1.4	1	Whole fruit	0.08, 0.17 (0.13)	
		0.45	3002	4/ 1.8	1	Whole fruit	0.43, 0.51 (0.47)	
R195 T-6391 Tempe, AZ, 1985 (Valencia)	300 g/L	0.11	2104	2/ 0.22	1	Whole fruit	0.05, 0.03 (0.04)	Fujie, 1990 (FR-01-0307)
		0.22	2104	2/ 0.44	1	Whole fruit	0.30, 0.17 (0.24)	
		0.45	2104	2/ 0.90	1	Whole fruit	1.1, 0.81 (0.96)	
		0.90	2104	2/ 1.8	1	Whole fruit	0.86, 1.3 (1.1)	
R195 T 6392 Donna, TX, 1985 (Valencia)	300 g/L	0.45	6547	2/ 0.90	1	Whole fruit	0.42, 0.63 (0.53)	Fujie, 1990 (FR-01-0307)
					7		0.45, 0.35 (0.40)	
					14		0.36, 0.43 (0.39)	
					21		0.34, 0.36 (0.35)	
R195 T-6393 Frostproof, FL, 1985 (Valencia)	300 g/L	0.45	5855	2/ 0.90	1	Whole fruit	0.16, 0.25 (0.21)	Fujie, 1990 (FR-01-0307)
					7		0.30, 0.19 (0.25)	
					14		0.27, 0.17 (0.22)	
					21		0.19, 0.17 (0.18)	
R195 T-6582 Weslaco, TX 1986 (Valencia)	300 g/L	0.45	1871	2/ 0.90	1	Whole fruit	1.2, 1.2 (1.2)	Fujie, 1990 (FR-01-0307)
					8		0.85, 1.4 (1.1)	
					15		0.57, 0.99 (0.78)	
					22		0.64, 0.81 (0.73)	
R195 T-6583 Frostproof, FL, 1985 (Valencia	300 g/L	0.45	2245	2/ 0.90	1	Whole fruit	0.26, 0.56, 0.32; (0.33)	Fujie, 1990 (FR-01-0307)
R195 T-6604 Fresno, CA, 1985 (Valencia)	300 g/L	0.45	3582	2/ 0.90	7	Whole fruit	0.22, 0.11 (0.17)	Fujie, 1990 (FR-01-0307)
R195 T-6723 Felda, FL, USA, 1986 (Valencia)	300 g/L	0.45	3255	2/ 0.90	1	Whole fruit	0.44, 0.48 (0.46)	Fujie, 1990 (FR-01-0307)

CITRUS	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total kg ai/ha	days		mg/kg	
R195 T-6730, Fresno, CA, 1986 (Navel)	300 g/L	0.45	935	2/ 0.90	1	Whole fruit	0.18, 0.18 (0.18)	Fujie, 1990 (FR-01-0307)
R195 T-6965 Fresno, CA, 1987 (Navel)	300 g/L	0.45	47 (aerial)	2/ 0.90	1	Whole fruit	0.07, 0.07 (0.07)	Fujie, 1990 (FR-01-0307)
R195 T-6966 Santa Paula, CA, 1987 (Valencia)	300 g/L	0.45	54 (aerial)	2/ 0.90	1	Whole fruit	0.04, 0.05 (0.05)	Fujie, 1990 (FR-01-0307)
LEMON								
T-7550 Yuma, AZ, 1990 (Unknown)	300 g/L	0.34	281	2/ 0.78	14	Whole fruit	0.14, 0.11 (0.13)	Lai, 1992 (FR-21-0342)
		0.45	281	2/ 0.9	14	Whole fruit	0.09, 0.10 (0.10)	
T-7551 Yuma, AZ, 1990 (Unknown)	300 g/L	0.34	271	2/ 0.78	14	Whole fruit	0.20, 0.22 (0.21)	Lai, 1992 (FR-21-0342)
		0.45	271	2/ 0.9	14	Whole fruit	0.34, 0.44 (0.39)	
T-7552 Yuma, AZ, 1990 (Rough)	300 g/L	0.34	281	2/ 0.78	14	Whole fruit	0.16, 0.12 (0.14)	Lai, 1992 (FR-21-0342)
		0.45	281	2/ 0.9	14	Whole fruit	0.26, 0.22 (0.24)	
V-1004A Porterville, CA, 1991 (Lisbon)	300 g/L	0.45	281	2/ 0.90	0	Whole fruit	0.21, 0.41 (0.31)	Lai, 1992 (FR-21-0342)
					1		0.62, 0.50 (0.56)	
					7		0.32, 0.38 (0.35)	
					14		0.27, 0.28 (0.28)	
V-1004B Fallbrook, CA, 1991 (Eureka)	300 g/L	0.45	935	2/ 0.90	0	Whole fruit	0.54, 0.51 (0.53)	Lai, 1992 (FR-21-0342)
					1		0.54, 0.46 (0.50)	
					7		0.50, 0.48 (0.49)	
					14		0.45, 0.57 (0.51)	
V-1004C Yuma, AZ, 1991 (Rough)	300 g/L	0.45	935	2/ 0.90	0	Whole fruit	0.73, 0.71 (0.72)	Lai, 1992 (FR-21-0342)
					1		0.49, 0.49 (0.49)	
					7		1.5, 0.85 (1.2)	
					14		0.72, 0.80 (0.76)	
GRAPEFRUIT								
V-1003A Mecca, CA, 1991 (Marsh Ruby)	300 g/L	0.45	935	2/ 0.90	0	Whole fruit	0.04, 0.06 (0.05)	Lai, 1992 (FR-21-0343)
					1		0.13, 0.11 (0.12)	

CITRUS	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total kg ai/ha	days		mg/kg	
					7		0.05, 0.06 (0.06)	
					14		0.09, 0.11 (0.10)	
V-1003B Bonsall, CA, 1991 (Ruby Red)	300 g/L	0.45	935	2/ 0.90	1	Whole fruit	0.23, 0.16 (0.20)	Lai, 1992 (FR-21-0343)
V-1003C Fillmore, CA, 1991 (Ruby Red)	300 g/L	0.45	935	2/ 0.90	1	Whole fruit	0.18, 0.18 (0.18)	Lai, 1992 (FR-21-0343)
V-1003D Myakka City, FL, 1991 (Thompson; Pink Marsh)	300 g/L	0.45	1758	2/ 0.90	0	Whole fruit	0.42, 0.51 (0.47)	Lai, 1992 (FR-21-0343)
					1		0.57, 0.36 (0.47)	
					7		0.34, 0.32 (0.33)	
					14		0.32, 0.23 (0.28)	
V-1003E Myakka City, FL, 1991 (Duncan)	300 g/L	0.45	1758	2/ 0.90	1	Whole fruit	0.36, 0.38 (0.37)	Lai, 1992 (FR-21-0343)
V-1003G Yuma, AZ, 1991 (Ruby Red)	300 g/L	0.45	935	2/ 0.90	1	Whole fruit	0.40, 0.27 (0.34)	Lai, 1992 (FR-21-0343)
V-1003I Vero Beach, FL,1991 (Red Ruby)	300 g/L	0.45	1871	2/ 0.90	1	Whole fruit	0.32, 0.36 (0.34)	Lai, 1992 (FR-21-0343)

* Report showed this value for "control samples". Trial samples residues were reported as < 0.01 mg/kg. The report indicated mislabelling.

Pome fruits

Apple, pear

Forty-seven supervised trials on pome fruit (27 in apples and 20 in pears) were conducted in the USA in 1984–1987, using a higher number of applications compared to the GAP in the USA (up to 0.9 kg ai/ha and a PHI of 14 days) (Fujie, 1990, Fujie and Leary, 1986; Reference: FR-81-0305, FR-01-0370, FR-81-0304, FR-01-0369). The EC formulation of fenpropathrin containing 300 g/L active ingredient, was applied to treated plots at the rates of 0.9–7.2 kg ai/ha from 7 to 42 days prior to harvest.

Samples were analysed within 1 year of harvest using the methods described in section of Methods of residue analysis, which were validated to an LOQ of 0.01 mg/kg. Concurrent recoveries from control samples fortified with fenpropathrin at levels of 0.1 and 0.2 mg/kg were within the acceptable range of 70-120%, with RSD below 20%. Results of the trials are summarized in Table 37.

Table 37 Residues of fenpropathrin in pome fruits resulting from supervised trials in the USA

Pome fruits	Application				DAT	Commodity	Residue mg/kg	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total (kg ai/ha/ season	days			
GAP, USA for pome fruit	300 EC (2.4 lb ai/gal)	0.22-0.45	700-3740	2/ 0.9	14			
APPLE								
R185 T-6719 Fletcher, NC, USA, 1986 (Red Delicious)	300 g/L	0.448	3741	8/3.584	14	Whole fruit	0.14, 0.14 (0.14)	FR-81-0305
R185 T-6720 Phelps, NY, USA, 1986 (Golden Delicious)	300 g/L	0.448	2806	8/3.584	14	Whole fruit	3.0; 4.1 (3.6)	FR-81-0305
R185 T-6721 Fenville, MI, USA, 1986 (Red Delicious)	300 g/L	0.448	1272	8/3.584	14	Whole fruit	1.4; 1.5 (1.5)	FR-81-0305
R185 T-6722 Wenatchee, WA, USA, 1986 (Red Bisbee)	300 g/L	0.448	3741	8/3.584	14	Whole fruit	3.8; 3.6 (3.7)	FR-81-0305
					42	Whole fruit	3.0; 2.5	
					42	Juice	0.37; 0.37	
					42	Wet pomace	5.6; 8.7	
					42	Dry pomace	20; 31	
R185 T-6729 Watsonville, CA, USA, 1986 (Red Delicious)	300 g/L	0.448	1871	8/3.584	14	Whole fruit	2.7; 2.1 (2.4) 2.5 * (* cooked 20 minutes)	FR-81-0305
R185 T-6880 Wenatchee, WA, USA, 1987 (Red Delicious)	300 g/L	0.448	3741	8/3.584	14	Whole fruit	2.4; 2.1 (2.3)	FR-81-0305
R185 T-6969 Wenatchee, WA, USA, 1987 (Aerial) (Red Delicious)	300 g/L	0.448	65	8/3.584	14	Whole fruit	0.39; 0.40 (0.40)	FR-81-0305
R185 T-6971 Waterlief, MI, USA,,1987 (Aerial) (Jonathan)	300 g/L	0.448	47	8/3.808	14	Whole fruit	0.30; 0.14 (0.22)	FR-81-0305
R185 T-6971 Sodus, NY, USA, 1987 (Aerial) (Cortland)	300 g/L	0.448	47	8/3.584	14	Whole fruit	0.78; 0.97 (0.88)	FR-81-0305
R185 T-6055 Hood River	300 g/L	0.336	3741	9/3.024	14	Whole fruit	3.1; 3.4 (3.3)	FR-0370

Pome fruits	Application				DAT	Commodity	Residue mg/kg	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total (kg ai/ha/ season	days			
County, OR, USA,1984 (Top Red Delicious)		0.448	3741	9/4.032	14	Whole fruit	4.1; 3.6 (3.8)	
					14	Wet pomace	13	
					14	Dry pomace	34	
					14	Juice	0.10	
R185 T-6056 Highland, NY, USA, 1984 (McIntosh)	300 g/L	0.336	3741	6/2.016	14	Whole fruit	1.1; 0.82 (0.96)	FR-0370
		0.448	3741	6/2.688	14	Whole fruit	1.2; 1.2 (1.2)	
R185 T-6058 Winchester, VA, USA, 1984 (Red Delicious)	300 g/L	0.336	3741	8/2.688	14	Whole fruit	1.3; 1.4 (1.4)	FR-0370
		0.448	3741	8/3.584	14	Whole fruit	1.1; 0.96 (1.0)	
R185 T-6059 Clemson, SC, USA, 1984 (Red Chief Red Delicious)	300 g/L	0.336	3741	8/2.688	14	Whole fruit	0.38	FR-0370
		0.448	3741	8/3.584	14	Whole fruit	0.57	
R185 T-6060 Fennville, MI, USA, 1984 (Jonathan)	300 g/L	0.336	1300	8/2.688	14	Whole fruit	1.4; 1.8 (1.6)	FR-0370
		0.448	1300	8/3.584	14	Whole fruit	2.6; 2.0 (2.3)	
R185 T-6061 Hulst Farms, CA, USA, 1984 (Pippen)	300 g/L	0.448	1871	2/0.896	14	Whole fruit	0.36; 1.4 (0.88)	FR-0370
		0.448	1871	4/1.792	14	Whole fruit	2.7; 2.4 (2.6)	
		0.448	1871	6/2.688	14	Whole fruit	2.7; 2.3 (2.5)	
		0.448	1871	8/3.584	14	Whole fruit	1.7; 1.6 (1.7)	
R185 T-6150 Biglerville, PA, USA, 1984 (York Imperial)	300 g/L	0.336	2806	8/2.688	10	Whole fruit	1.2; 3.0 (2.1)	FR-0370
		0.448	2806	8/3.584	10	Whole fruit	3.7; 3.6 (3.7)	
R185 T-6151 Phelps, NY, USA, 1984	300 g/L	0.448	2806	2/0.896	14	Whole fruit	0.95; 1.3 (1.1)	FR-0370
		0.448	2806	4/1.792	14	Whole fruit	2.1; 2.7 (2.4)	
		0.448	2806	5/2.688	14	Whole fruit	2.7; 3.2 (3.0)	
		0.448	2806	8/3.584	14	Whole fruit	2.6; 2.5 (2.6)	
R185 T-6152 Phelps, NY, USA, 1984 (Golden Delicious)	300 g/L	0.448	2806	8/3.584	7	Whole fruit	3.2; 3.2	FR-0370
		0.448	2806	8/3.584	14	Whole fruit	2.7; 2.3 (2.5)	
					14	Wet pomace	13; 11	
					14	Dry pomace	44; 45	
					14	Juice	< 0.01; < 0.01	
		0.448	2806	8/3.584	21	Whole fruit	2.1; 2.5 (2.3)	
		0.448	2806	8/3.584	28	Whole fruit	3.4; 2.9 (3.2)	
R185 T-6154 Wenatchee, WA, USA, 1984 (Golden Delicious)	300 g/L	0.336	3741	8/2.688	14	Whole fruit	1.5; 2.4 (2.0)	FR-0370
		0.448	3741	8/3.584	7	Whole fruit	2.3; 2.0 (2.2)	
		0.448	3741	8/3.584		Pulp	0.77; 0.20	
		0.448	3741	8/3.584		Peel	12; 11	
		0.448	3741	8/3.584	14	Whole fruit	2.2; 2.6 (2.4)	
		0.448	3741	8/3.584	21	Whole fruit	2.1; 2.1 (2.1)	
		0.448	3741	8/3.584	28	Whole fruit	2.2; 2.1 (2.2)	

Pome fruits	Application				DAT	Commodity	Residue mg/kg	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total (kg ai/ha/ season	days			
R185 T-6400) Wenatchee, WA, USA, 1985 (Golden Delicious)	300 g/L	0.112	3741	8/0.896	14	Whole fruit	1.3; 1.7 (1.5)	FR-0370
		0.224	3741	8/1.792	14	Whole fruit	2.4; 3.2 (2.8)	
		0.448	3741	8/3.584	14	Whole fruit	5.1; 7.3 (6.2)	
		0.896	3741	8/7.168	14	Whole fruit	7.6; 8.2 (7.9)	
R185 T-6401, Wenatchee WA, USA, 1985 (Golden Delicious)	300 g/L	0.448	3741	8/3.584	14	Whole fruit	5.2; 4.2 (4.7)	FR-0370
					14	Wet pomace	12; 12	
					14	Dry pomace	47; 38	
					14	Juice	0.11, 0.12	
							0.15, 0.16	
R185 T-6402 Hood River, OR, USA, 1985 (Red Delicious)	300 g/L	0.448	3741	2/0.896	14	Whole fruit	0.51; 0.65 (0.58)	FR-0370
		0.448	3741	4/1.792	14	Whole fruit	1.3; 1.5 (1.4)	
		0.448	3741	6/2.688	14	Whole fruit	1.6; 2.0 (1.8)	
		0.448	3741	8/3.584	14	Whole fruit	1.4; 1.1 (1.3)	
		0.448	3741	8/3.584	14	Whole fruit	1.4; 1.4	
					14	Pulp	0.07; 0.15	
					14	Peel	4.0; 5.5	
R185 T-6403 Haslet, MI, USA, 1985 (Red Delicious)	300 g/L	0.448	1010	2/0.896	14	Whole fruit	0.40; 0.56 (0.48)	FR-0370
		0.448	1010	4/1.792	14	Whole fruit	1.6; 2.1 (1.9)	
		0.448	1010	6/2.688	14	Whole fruit	2.8; 2.3 (2.6)	
		0.448	1010	8/3.584	14	Whole fruit	3.3; 4.0 (3.8)	
R185 T-6404 Fennville, MI, USA, 1985 (Red Delicious)	300 g/L	0.112	1132	8/0.896	14	Whole fruit	0.02 *	FR-0370 * Duplicate sample broken in shipment.
		0.224	1132	8/1.792	14	Whole fruit	2.6 *	
		0.448	1132	8/3.584	14	Whole fruit	3.8; 5.2 (4.5)	
		0.896	1132	8/7.168	14	Whole fruit	7.8; 8.8	
R185 T-6405 Wooster, OH, USA, 1985 (Cortland)	300 g/L	0.448	2806	8/3.584	14	Whole fruit	1.5; 1.5; 1.7; 5.7*; 1.8; 2.1 (1.7)	FR-0370 * Outlier, based on Dixon's Q test
R185 T-6406 Phelps, NY, USA, 1985 (Rhode Island Greening)	300 g/L	0.448	2806	8/3.584	14	Whole fruit	1.6; 1.6 (1.6)	FR-0370
					14	Whole fruit	1.4; 1.1 (1.3)	
					21	We pomace	3.8; 5.1	
					21	Dry pomace	12; 8.4	
					21	Juice	0.01; 0.01	
PEAR								
R186 T-6709 River Bottom, CA (Bartlett)	300 g/L	0.448	935	6/2.688	14	Whole fruit	1.3; 0.96 (1.1) 0.94*	FR-81-0304 * Washed under tap water with gentle hand rubbing
R186 T-6711 Medford, OR, USA, 1986 (Bartlett)	300 g/L	0.448	3741	6/2.688	14	Whole fruit	3.0; 2.8 (2.9)	FR-81-0304
R186 T-6712 Fenville, MI, USA, 1968	300 g/L	0.488	1300	6/2.688	14	Whole fruit	2.0; 1.6 (1.8)	FR-81-0304

Pome fruits	Application				DAT	Commodity	Residue mg/kg	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total (kg ai/ha/ season	days			
(Bartlett)								
R186 T-6713 Phelps, NY, USA, 1986 (Bartlett)	300 g/L	0.448	2806	6/2.688	14	Whole fruit	2.5; 2.3 (2.4)	FR-81-0304
R186 T-6886 Lagrange, CA, USA, 1987 (Bartlett)	300 g/L	0.448	935	6/2.688	14	Whole fruit	1.9; 1.6 (1.8)	FR-81-0304
R186 T-6972 Live Oak, CA (Aerial), USA, 1987 (Bartlett)	300 g/L	0.448	1878	6/2.688	14	Whole fruit	0.26; 0.32 (0.29)	FR-81-0304
R186 T-6973 Sodus, NY (Aerial), USA, 1987 (Bartlett)	300 g/L	0.448	47	6/2.688	14	Whole fruit	0.76; 0.96 (0.86)	FR-81-0304
R186 T-6062 Life Oak, CA, USA, 1984 (Bartlett)	300 g/L	0.336	3741	6/2.016	14	Whole fruit	0.84; 0.34 (0.59)	FR-0369
		0.448	3741	6/2.688	7	Whole fruit	0.52; 0.84	
					14		0.77; 0.46 (0.62)	
					21		0.59; 0.52	
					28		0.56; 0.42	
R186 T-6063 Live Oak, CA, USA, 1984 (Bartlett)	300 g/L	0.336	3741	6/2.016	14	Whole fruit	0.42, 0.36 (0.39)	FR-0369
		0.448	3741	2/0.892	14	Whole fruit	0.27; 0.27 (0.27)	
		0.448	3741	3/1.344	14	Whole fruit	0.55; 0.34 (0.45)	
		0.448	3741	5/2.24	14	Whole fruit	0.41; 0.42 (0.42)	
		0.448	3741	6/2.688	14	Whole fruit	0.65; 0.50 (0.53)	
R186 T-6064 Medford, OR, USA, 1984 (Bartlett)	300 g/L	0.448	3741	6/2.688	14	Whole fruit	0.96; 1.4 (1.2)	FR-0369
R186 T-6065 Wenatchee, WA, USA, 1984 (Bartlett)	300 g/L	0.336	3741	6/2.196	14	Whole fruit	0.87; 1.0 (0.94)	FR-0369
		0.448	3741	6/2.688	14	Whole fruit	1.5; 1.1 (1.3)	
R186 T-6066 Fenville, MI, USA, 1984 (Bartlett)	300 g/L	0.336	1422	6/2.016	14	Whole fruit	1.3	FR-0369
R186 T-6067 Phelps, NY, USA, 1984 (Bartlett)	300 g/L	0.448	2806	2/0.896	14	Whole fruit	1.2; 1.1 (1.2)	FR-0369
		0.448	2806	4/1.792	14	Whole fruit	1.5; 1.4 (1.5)	
		0.448	2806	5/2.24	14	Whole fruit	1.4; 1.4 (1.4)	
		0.448	2806	6/2.688	14	Whole fruit	1.7; 1.7 (1.7)	
					14	Pear halves in	< 0.01;	

Pome fruits	Application				DAT	Commodity	Residue mg/kg	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./Total (kg ai/ha/ season	days			
						syrup	< 0.01	
					14	Puree	< 0.01; < 0.01	
R186 T-6153 Phelps, NY, USA, 1984 (Bartlett)	300 g/L	0.448	2806	6/2.688	7	Whole fruit	2.7; 1.8 (2.3)	FR-0369
		0.448	2806	6/2.688	14	Whole fruit	2.2; 1.6 (1.9)	
		0.448	2806	6/2.688	21	Whole fruit	2.0; 1.9 (2.0)	
		0.448	2806	6/2.688	28	Whole fruit	1.0; 0.89 (0.95)	
R186 T-6394 Wenatchee, WA, USA, 1985 (Bartlett)	300 g/L	0.112	3741	6/0.672	14	Whole fruit	0.82; 0.57 (0.70)	FR-0369
		0.224	3741	6/1.344	14	Whole fruit	1.2; 0.72 (0.96)	
		0.448	3741	6/2.688	14	Whole fruit	1.7; 0.31 (1.0)	
		0.896	3741	6/5.376	14	Whole fruit	3.8; 2.6 (3.2)	
R186 T-6395 Hood River, OR, USA, 1985 (Bartlett)	300 g/L	0.448	3741	2/0.896	14	Whole fruit	0.31; 0.29 (0.30)	FR-0369 * Washed
		0.448	3741	4/1.792	14	Whole fruit	0.47; 0.78 (0.63)	
		0.448	3741	6/2.688	14	Whole fruit	0.62; 0.85 (0.74)	
		0.448	3741	8/3.584	14	Whole fruit	1.0; 0.89 (0.95)	
		0.448	3741	8/3.584	21	Whole fruit	0.91; 0.86	
					21		0.72*	
					21	Peel	5.9	
					21	Pulp	0.01	
R186 T-6396 Ukiah, CA, USA, 1985 (Bartlett)	300 g/L	0.448	3741	6/2.688	1 1 1	Whole fruit Pulp Peel	1.6; 1.4 0.01; < 0.01 12; 13	FR-0369
		0.448	3741	6/2.688	7	Whole fruit	2.0; 2.3 (2.2)	
		0.448	3741	6/2.688	14	Whole fruit	1.5; 1.6 (1.6)	
		0.448	3741	6/2.688	21	Whole fruit	1.4; 1.1 (1.3)	
		0.448	3741	6/2.688	28	Whole fruit	0.78; 0.91 (0.85)	
		0.448	3741	6/2.688	35	Whole fruit	0.79; 1.1 (0.95)	
R186 T-6397 Fennville, MI, USA.1985 (Bartlett)	300 g/L	0.448	1085	2/0.896	14	Whole fruit	1.6; 2.0 (1.8)	FR-0369
		0.448	1085	4/1.792	14	Whole fruit	2.4; 2.8 (2.6)	
		0.448	1085	6/2.688	14	Whole fruit	2.9; 4.5 (3.7)	
		0.448	1085	8/3.584	14	Whole fruit	4.8; 3.9 (4.4)	
R186 T-6398 Phelps, NY, USA, 1985 (Bartlett)	300 g/L	0.112	2806	6/0.672	14	Whole fruit	0.48; 0.54 (0.51)	FR-0369
		0.224	2806	6/1.344	14	Whole fruit	1.2; 1.0 (1.1)	
		0.448	2806	6/2.688	14	Whole fruit	2.2; 2.1 (2.2)	
		0.896	2806	6/5.376	14	Whole fruit	3.3; 3.2 (3.3)	

Stone fruit

Peach, plum, cherry

Supervised trials were conducted on stone fruits in the USA during 2003. Ten of the trials were on peaches (Green, 2004; Reference FR-0384), six on cherries (Green, 2004; Reference FR-0381), and seven on plums (Green, 2004; FR-0385). All trials were according to the US GAP on stone fruit, which includes applications of fenpropathrin 300 g/L EC formulation at the rate of 0.45 kg ai/ha for a maximum seasonal application of 0.9 kg ai/ha. Duplicate samples of mature fruits were collected 3 days after the last application. Sample size for peach ranged from 2.6 to 8.5 kg, ≥ 24 fruit. Upon collection, all samples were frozen at -20 ºC until extraction and analysis. All of the samples were analysed within the documented storage stability interval analysed using analytical method RM-22-4 (LOQ 0.01 mg/kg). Concurrent recoveries were within the acceptable range of 70 to 120%.

Table 38 Residues of fenpropathrin in stone fruit resulting from supervised trials in the United States

STONE FRUIT	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha/	days		mg/kg	
GAP, USA	300 g/L EC	0.22-0.45	935- 3740	0.9	3			
PEACHES								
V-25419-A Berks County, PA, USA, 2003 (John Boy)	300 EC	0.45 0.46	1122 1141	2/0.91	3	Whole fruit	0.66, 0.66 (0.66)	FR-0384
V-25419-B Saluda County, SC, USA, 2003 (Sureprince)	300 EC	0.45 0.49	1207 1178	2/0.94	3	Whole fruit	0.76, 0.63 (0.70)	FR-0384
		0.90 0.89	1216 1169	2/1.79	3	Whole fruit	1.3, 0.90 (1.1)	
V-25419-C Aieken, County, SC, USA, 2003 (Contender)	300 EC	0.44 0.44	1066 1057	2/0.88	3	Whole fruit	0.56, 0.73 (0.65)	FR-0384
V-25419-D Houston County, AL, USA, 2003 (Harvester)	300 EC	0.45 0.45	1094 1104	2/0.90	3	Whole fruit	0.91, 1.1 (1.0)	FR-0384
V-25419-E Clarke County, GA, USA, 2003 (Contender)	300 EC	0.44 0.45	1141 1225	2/0.89	3	Whole fruit	1.0, 1.0 (1.0)	FR-0384
V-25419-F Ottawa County, MI, USA, 2003 (Red Haven)	300 EC	0.44 0.44	1132 1132	2/0.88	3	Whole fruit	0.69, 0.72 (0.71)	FR-0384
V-25419-G Freestone County, TX, USA, 2003 (Fairtime)	300 EC	0.46 0.44	945 1085	2/0.90	3	Whole fruit	0.79, 0.67 (0.73)	FR-0384
		0.92 0.89	945 1085	2/1.8	3	Whole fruit	1.1, 0.66 (0.88)	
V-25419-H Sutter County, CA, USA, 2003 (Loadel)	300 EC	0.45 0.45	1066 1066	2/0.90	3	Whole fruit	0.61, 0.54 (0.58)	FR-0384

STONE FRUIT	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha/	days		mg/kg	
V-25419-I Stanislaus, County CA, USA, 2003 (Summerset)	300 EC	0.45 0.44	1291 1272	2/0.89	1	Whole fruit	0.50, 0.45 (0.48)	FR-0384
					4	Whole fruit	0.49, 0.39 (0.44)	
					7	Whole fruit	0.35, 0.24 (0.30)	
					10	Whole fruit	0.34, 0.34 (0.34)	
V-25419-J Fresno County, CA, USA, 2003 (Fayette)	300 EC	0.43 0.44	1178 1197	2/0.87	3	Whole fruit	1.0, 0.84 (0.92)	FR-0384
CHERRIES								
V-25380-A Wayne County, NY, USA, 2003 (Montmorency/ tart)	300 EC	0.46 0.47	973 982	2/0.93	3	Whole fruit	1.83, 1.97 (1.9)	FR-0381
		0.92 0.94	963 982	2/1.86	3	Whole fruit	5.16, 4.21 (4.7)	
V-25380-B Ottawa County, MI, USA, 2003 (Napoleon/ sweet)	300 EC	0.45 0.45	1104 1094	2/0.90	1	Whole fruit	1.76, 1.62 (1.7)	FR-0381
					3		2.02, 1.6 (1.8)	
					7		1.43, 1.4 (1.4)	
					10		1.38, 1.16 (1.3)	
V-25380-C Delta County, CO, USA, 2003 (Montmorency/ tart)	300 EC	0.45 0.45	954 945	2/0.90	3	Whole fruit	1.44, 1.43 (1.4)	FR-0381
V-25380-D Lehigh County, PA, USA, 2003 (Montmorency/ tart)	300 EC	0.45 0.45	991 1001	2/0.90	3	Whole fruit	3.18, 3.33 (3.3)	FR-0381
V-25380-E Santa Clara, CA, USA, 2003 (Bing/sweet)	300 EC	0.44 0.43	1001 982	2/0.87	3	Whole fruit	1.59, 1.43 (1.5)	FR-0381
		0.88 0.86	1010 982	2/1.74	3	Whole fruit	2.26, 1.90 (2.1)	
V-25380-F Grant County, WA, USA, 2003 (Van/ sweet)	300 EC	0.45 0.45	945 935	2/0.90	1	Whole fruit	2.74, 2.98 (2.9)	FR-0381
					3	Whole fruit	3.53, 3.23 (3.4)	
					7	Whole fruit	3.06, 2.76 (2.9)	
					10	Whole fruit	2.41, 2.3 (2.4)	
PLUMS								
V-25427-A Ottawa County, MI, USA, 2003	300 EC	0.44 0.45	1057 1094	2/0.89	1	Whole fruit	0.57, 0.62 (0.60)	FR-0385
					3	Whole fruit	0.51, 0.58	

STONE FRUIT	Application				DAT	Commodity	Residue	Reference
Trial year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha/	days		mg/kg	
(Stanley)							(0.55)	
					7	Whole fruit	0.71, 0.63 (0.67)	
					10	Whole fruit	0.44, 0.46 (0.45)	
V-25427-B Tulare County, CA, USA, 2003 (Angelona's)	300 EC	0.45 0.46	1029 1048	2/0.91	3	Whole fruit	0.23, 0.23 (0.23)	FR-0385
V-25427-C Sutter County, CA, USA, 2003 (French Prune)	300 EC	0.45 0.45	1038 1029	2/0.90	3	Whole fruit	0.29, 0.21 (0.25)	FR-0385
V-25427-D Tehama County, CA, 2003 (French Prune)	300 EC	0.45 0.45	1029 1029	2/0.90	3	Whole fruit	0.37, 0.27 (0.32)	FR-0385
V-25427-E Polk County, OR, 2003 (Moyer)	300 EC	0.44 0.45	1085 1104	2/0.89	4	Whole fruit	0.24, 0.20 (0.22)	FR-0385
V-25427-F Fresno County, CA, USA, 2003 (French Prune)	300 EC	0.44 0.44	1150 1169	2/0.88	4	Whole fruit	0.17, 0.18 (0.18)	FR-0385
		0.89 0.90	1160 1178	2/1.8	4	Whole fruit	0.61, 0.54 (0.58)	
V-27220-A Fresno County, CA, USA, 2004 (French Prune)	300 EC	0.44 0.45	1169 1188	2/0.89	3	Whole fruit	0.29, 0.34 0.27, 0.50 (0.35)	FR-0559

Berries and other small fruit

Strawberry

Ten supervised trials on strawberries were conducted in the US in 1987 and 1989 (Mitten, 1991; Reference FR-11-0345). Two additional trials were carried out in 1991 (Lai, 1992; Reference FR-21-0346). Eleven of the trials matched the current GAP in the US for strawberries (applications at 0.22 to 0.45 kg ai/ha for a total of 0.9 kg ai/ha per season and a PHI of 2 days). At several sites, replicate trials were carried out to study the effect of application rate and PHI on the residues in harvested samples. Mature samples were collected two days after the last application and immediately stored frozen at -20 ºC until analysis. Samples from the trials were analysed within this storage stability period with method RM-22-4. Concurrent recoveries from fortified samples analysed together with field samples ranged from 81-115%, mean = 98.3%, with RSD of 8.2% (n=27).

Table 39 Residues of fenpropathrin in strawberries resulting from supervised trials in the United States

Strawberry	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total/ kg ai/ha	days		mg/kg	
GAP, USA	300 EC	0.22-0.45	935-2800	2/0.9	2			
M391 T-7125 Watsonville, CA, USA, 1987 (Muir)	300 g/L	0.34	2245	0.34	1 3	Berries	1.2, 1.2 (1.2) 1.1, 1.2 (1.2)	FR-0345
		0.34	2245	2/0.68	1 3	Berries	1.3, 1.2 (1.3) 1.1, 1.1 (1.1)	
M391 T-7126 Watsonville, CA USA, 1987 (Selva)	300 g/L	0.34	2245	0.34	1 3	Berries	0.77, 0.87 (0.82) 0.69, 0.65 (0.67)	FR-0345
		0.34	2245	2/0.68	1 3	Berries	0.68, 0.58 (0.63) 0.56, 0.62 (0.59)	
1714-89-7279 Watsonville, CA, USA, 1989 (Selva)	300 g/L	0.11	1403	0.11	2	Berries	0.43, 0.40 (0.42)	FR-0345
		0.11	1403	2/0.22	2	Berries	0.10, 0.12 (0.11)	
		0.11	1403	3/0.34	2	Berries	0.08, 0.06 (0.07)	
		0.11	1403	4/0.44	2	Berries	0.11, 0.09 (0.10)	
		0.22	1403	0.22	2	Berries	0.78, 0.76 (0.77)	
		0.22	1403	2/0.44	2	Berries	0.35, 0.27 (0.31)	
		0.22	1403	3/0.66	2	Berries	0.22, 0.17 (0.20)	
		0.22	1403	4/0.89	2	Berries	0.12, 0.26 (0.19)	
		0.45	1403	0.45	2	Berries	1.2, 1.1 (1.2)	
		0.45	1403	2/0.90	2	Berries	0.72, 0.54 (0.63)	
		0.45	1403	3/1.35	2	Berries	0.35, 0.35 (0.35)	FR-0345
		0.45	1403	4/1.8	2	Berries	0.43, 0.53 (0.48)	
		0.90	1403	0.90	2	Berries	2.6, 2.8 (2.7)	
		0.90	1403	2/1.8	2	Berries	0.90, 1.3 (1.1)	
		0.90	1403	3/2.7	2	Berries	0.84, 0.87 (0.86)	
		0.90	1403	4/3.6	2	Berries	1.1, 0.74	

Strawberry	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total/ kg ai/ha	days		mg/kg	
							(0.92)	
1714-89-7281 Rosa, LA USA, 1989 (Chandler)	300 g/L	0.45	1440	0.45	2	Berries	0.83, 0.86 (0.85)	FR-0345
		0.45	1440	2/0.89	2	Berries	0.46, 0.50 (0.48)	
1714-89-7282 Bonsall, CA, USA, 1989 (Chandler)	300 g/L	0.45	2806	0.45	2	Berries	0.68, 1.0 (0.84)	FR-0345
		0.45	2806	2/0.90	2	Berries	0.59, 0.71 (0.65)	
		0.45	2806	3/1.3	2 29	Berries	0.43, 0.39 (0.41) 0.05, 0.05 (0.05)	
		0.45	2806	4/1.8	0	Berries	0.55, 0.56 (0.56)	
					1		0.55, 0.44 (0.50)	
					2		0.52, 0.55 (0.54)	
					3		0.45, 0.40 (0.43)	
					4		0.60, 0.57 (0.59)	
					7		0.41. 0.38 (0.40)	
					14		0.14, 0.13 (0.14)	
					21		0.09, 0.10 (0.10)	
							0.03, 0.04 (0.04)	
1741-89-7283 Fresno, CA, USA, 1989 (Chandler)	300 g/L	0.45	1403	2/0.90	2	Berries	0.63, 0.74 (0.69)	FR-0345
1741-89-7285 Santa Maria, CA, USA, 1989 (Muir)	300 g/L	0.45	2806	0.45	2	Berries	1.5, 1.4 (1.5)	FR-0345
		0.45	2806	2/0.90	2	Berries	1.2, 1.2 (1.2)	
		0.45	2806	3/1.3	2	Berries	0.72, 0.76 (0.74)	
		0.45	2806	4/1.8	2	Berries	0.56, 0.66 (0.61)	
1741-89-7287 Grand Rapids, MI, USA, 1989 (Not reported)	300 g/L	0.11	1225	1/0.11	2	Berries	0.07	FR-0345
		0.22	1225	2/0.22	2	Berries	0.16	
		0.45	1225	1/0.45	2	Berries	0.28	
		0.90	1225	2/0.90	2	Berries	0.63	

Strawberry	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total/ kg ai/ha	days		mg/kg	
		0.11	1225	2/0.22	2	Berries	0.09	
		0.22	1225	2/0.44	2	Berries	0.23	
		0.45	1225	2/0.90	2	Berries	0.48	
		0.90	1225	2/1.8	2	Berries	1.1	
1714-89-7427 Vancouver, WA, USA, 1989 (S. Huxson)	300 g/L	0.45	1403	0.45	2	Berries	0.21, 0.20 (0.21)	FR-0345
		0.45	1403	2/0.90	2	Berries	0.29, 0.49 (0.39)	
1714-89-7428 Gales Creek, OR, USA, 1989 (Benton)	300 g/L	0.45	1403	0.45	29	Berries	0.04, 0.03 (0.04)	FR-0345
		0.45	1403	2/0.90	0	Berries	0.38, 0.44 (0.41)	
					1		0.36, 0.47 (0.42)	
					2		0.36, 0.34 (0.35)	
					3		0.38, 0.37 (0.38)	
					4		0.28, 0.35 (0.32)	
					7		0.29, 0.32 (0.31)	
					14		0.19, 0.17 (0.18)	
					21		0.12, 0.11 (0.12)	
					28		0.23, 0.14 (0.19)	
V-1020A Wilson, NC, USA, 1991 (Chandler)	300 g/L	0.45	963	0.45	2	Berries	0.16, 0.18 (0.17)	FR-21-0346
		0.45	963	2/0.90	2	Berries	0.29, 0.22 (0.26)	
V-1020B Watsonville, CA, USA, 1991 (Selva)	300 g/L	0.45	935	0.45	2	Berries	0.29, 0.37 (0.33)	FR-21-0346
		0.45	935	2/0.90	2	Berries	0.59, 0.51 (0.55)	

Raspberry

Seven supervised trials on raspberries were conducted in the USA in 2005 (Samoil, 2007; Reference FR-0560) with higher rate and shorter PHI than the current GAP (applications at 0.22 to 0.34 kg ai/ha for a total of 0.67 kg ai/ha per season and a PHI of 3 days). Mature samples were collected 2–3 days after the last application and immediately stored frozen at -20 ºC until analysis. Samples from the trials were analysed within this storage stability period 216 days with method RM-22-4. Concurrent recoveries ranged from 74–110%, mean = 98.3%, with SD of 8.2% (n=10).

Table 40 Residues of fenpropathrin in raspberries resulting from supervised trials in the United States

Raspberry	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
GAP, USA	2.4 EC (300g ai/l; 2.4lb ai/gal)	0.22-0.34	935-2800	0.67	3			
CA*62 Corralitos, CA, USA, 2005 (Isabel)	300 g/L	0.45	505	2/0.9	2	Berries	1.0, 1.2 (1.1)	FR-0560
MI06 Holt, MI USA, 2005 (Heritage)	300 g/L	0.46 0.47	206	2/0.93	2	Berries	5.5, 6.1 (5.8)	FR-0560
NC12 Bridgetown, NJ USA, 2005 (Mandarin)	300 g/L	0.44 0.45	271	2/0.89	2	Berries	4.1, 7.1 (5.6)	FR-0560
NC14 Fletcher, NC USA, 2005 (Canby)	300 g/L	0.48	290	2/0.96	3	Berries	1.4, 1.9 (1.7)	FR-0560
OR07 Aurora, OR USA, 2005 (Marion)	300 g/L	0.49 0.46	411 383	2/0.95	2	Berries	4.7, 3.3 (4.0)	FR-0560
OR08 Aurora, OR USA, 2005 (Willamette)	300 g/L	0.47 0.49	393 402	2/0.96	2	Berries	1.8, 1.7 (1.8)	FR-0560
OR09 Aurora, OR USA, 2005 (Willamette)	300 g/L	0.47	393	2/0.94	2	Berries	2.3, 1.8 (2.1)	FR-0560

Grapes

Twenty five supervised trials were conducted on grapes in the USA during 1983-2001. (Fujie, 1990; Fujie, 1992, Green, 2002; FR-01-0308, FR-01-0335, FR-01-0374). All trials were within the US GAP on grapes. Duplicate samples of mature fruits were collected 21 days after the last application.

All samples were analysed using analytical method RM-22-4 with an LOQ of 0.01 mg/kg within the storage stability period. Concurrent recoveries were within the acceptable range of 70 to 120%.

Table 41 Residues of fenpropathrin in grapes resulting from supervised trials in the United States

GRAPES	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No./ Total kg ai/ha	days		mg/kg	
GAP, USA for grapes	300 EC (300g ai/l)	0.11-0.45	234-1870	2/ 0.9	21			
GRAPE								
M335 T-5952 Fresno, CA, USA, 1983 (Thompson Seedless)	300 g/L	0.112	1019	2/0.336	25	Bunches	0.15, 0.13	FR-01-0308
		0.224	1019					
		0.112	1019	2/0.224	25	Bunches	0.07, 0.12	
		0.112	1019					
R187 T-6077, Fresno, CA, USA, 1984 (Thompson Seedless)	300 g/L	0.224	1001	4/0.896	21	Bunches	0.31; 0.43 (0.37)	FR-01-0308 * dried 15 days in field ** repeat analysis
						Raisins*	0.45	
						Wet pomace	0.49	
						Dry pomace	2.45	
						Juice	0.02; 0.03	
						Raisin waste	0.03, 0.02**	
R187 T-6078, Madera, CA, USA, 1984 (Thompson Seedless)	300 g/L	0.224	1403	4/0.896	21	Bunches	0.60; 0.90 (0.75)	FR-01-0308 * dried 15 days in field ** repeat analysis
						Raisins*	1.52	
						Wet pomace	0.90	
						Dry pomace	4.90	
						Juice	0.09; 0.09	
						Raisin waste	0.13, 0.13**	
R187 T-6079, Delano, CA, USA, 1984 (Centennial)	300 g/L	0.224	2338	2/0.448	14	Bunches	0.49, 0.64	FR-01-0308
R187 T-6081, Santa Rosa, CA, USA, 1984 (Cabernet Sauvignon)	300 g/L	0.224	935	0.224	95	Fruit	0.27, 0.16	FR-01-0308
R187 T-6409, Fresno, CA, USA, 1985 (Thompson Seedless)	300 g/L	0.224	1019	4/0.896	1	Fruit	0.49, 0.35 0.43*	FR-01-0308 * washed under tap water
		0.224	1019	4/0.896	7	Fruit	0.72; 0.57	
		0.224	1019	4/0.896	14	Fruit	0.48; 0.40	
		0.224	1019	4/0.896	21	Fruit	0.42; 0.62 (0.52)	
		0.224	1019	4/0.896	28	Fruit	0.44, 0.58 (0.51)	
		0.224	1019	4/0.896	35	Fruit	1.1; 0.56 (0.83)	
R187 T-6410, CA, USA, 1985 (Thompson Seedless)	300 g/L	0.056	1871	4/0.224	21	Bunches	0.05; 0.16	FR-01-0308
		0.112	1871	4/0.448	21	Bunches	0.15; 0.10	
		0.224	1871	4/0.896	21	Bunches	0.45; 0.44 (0.45)	
		0.448	1871	4/1.792	21	Bunches	0.81; 1.5 (1.2)	
R187 T-6411, Santa Maria, CA, USA, 1985 (Chardonnay)	300 g/L	0.224	1871	0.224	21	Bunches	0.34, 0.21	FR-01-0308
		0.224	1871	2/0.448	21	Bunches	0.19; 0.96 (0.58)	
		0.224	1871	3/0.672	21	Bunches	1.0; 0.77 (0.89)	

GRAPES	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No./ Total kg ai/ha	days		mg/kg	
		0.224	1871	4/0.896	21	Bunches	1.6; 1.3 (1.5)	
R187 T-6412, Fresno, CA, USA, 1985 (Thompson Seedless)	300 g/L	0.224	1019	4/0.896	21	Bunches Raisins* Wet pomace Dry pomace Juice Raisins – off grade* Raisin waste Wine	1.0; 0.48 (0.74) 0.52 0.85 1.2 < 0.01 0.81 3.23 < 0.01	FR-01-0308 * dried 33 days in field
R187 T-6413, Ukiah, CA, USA, 1985 (Caignane)	300 g/L	0.224	1403	4/0.896	21	Bunches Juice Wine	3.3; 2.9 (3.1) < 0.01; 0.01 < 0.01; < 0.01	FR-01-0308
R187 T-6414, Prosser, WA, USA, 1985 (Chardonnay)	300 g/L	0.224	935	0.224	1	Bunches	1.8; 2.8	FR-01-0308
		0.224	935	0.224	7	Bunches	2.0; 1.9	
		0.224	935	0.224	14	Bunches	1.8; 1.6	
		0.224	935	0.224	21	Bunches	1.5; 1.3	
		0.224	935	0.224	28	Bunches	1.0; < 0.01	
		0.224	935	0.224	35	Bunches	0.94; 0.10	
R187 T-6415, Fredonia, NY, USA, 1985 (Concord)	300 g/L	0.224	935	4/0.896	1	Bunches	1.3; 0;82	FR-01-0308
					7		0.90; 0.92	
					14		0.47; 0.87	
					21		0.81; 1.4 (1.1)	
					28		0.80; 0.66	
					35		0.89; 0.91	
R187 T-6416, Wooster, OH, USA, 1985 (Concord)	300 g/L	0.056	935	4/0.224	21	Bunches	0.08; 0.22	FR-01-0308
		0.112	935	4/0.448	21	Bunches	0.16; 0.38	
		0.224	935	4/0.896	21	Bunches	1.4; 0.72 (1.1)	
		0.448	935	4/1.792	21	Bunches Wet pomace Dry pomace Juice	2.4; 2.8 (2.6) 3.8; 1.8 11; 10 0.01; < 0.01	
R187 T-6417, Fennville, MI, USA, 1985 (Concord)	300 g/L	0.224	926	0.224	21	Bunches	0.26; 0.16	FR-01-0308 * washed, frozen
		0.224	926	2/0.448	21	Bunches	1.5; 1.0 (1.3)	
		0.224	926	3/0.672	21	Bunches	1.2; 1.8 (1.5)	
		0.224	926	4/0.896	21	Bunches	1.5; 1.5 (1.5) 1.0*	
R187 T-6725, Phelps, NY, USA, 1986 (Catawba)	300 g/L	0.224	935	4/0.896	19	Bunches	1.4; 0.81 (1.1)	FR-01-0308
R187 T-6726, Fennville, MI, USA, 1986 (Concord)	300 g/L	0.224	926	4/0.896	14	Bunches	1.6, 1.2	FR-01-0308
R187 T-6728, Fresno, CA, USA, 1986	300 g/L	0.224	1001	4/0.896	21	Bunches	0.93; 1.1 (1.0) 0.87*	FR-01-0308 * washed under

GRAPES	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No./ Total kg ai/ha	days		mg/kg	
(Thompson Seedless)								tap water
R187 T-6731, Hughson, CA, USA, 1986 (Chenin Blanc)	300 g/L	0.224	935	4/0.896	21	Bunches Wine Juice Wet pomace Dry pomace Stems	1.3; 1.5 (1.4) < 0.01 < 0.01 0.99 2.6 1.2	FR-01-0308
R187 T-6829, Soledad, CA, USA, 1986 (Cabernet Sauvignon)	300 g/L	0.224	935	4/0.896	21	Bunches	5.7; 5.4 (5.6)	FR-01-0308
		0.224	935	4/0.896	21	Wet pomace Dry pomace Juice Wine	4.1 9.4 0.06 < 0.01	
R187 T-6835, Prosser, WA, USA, 1986 (Chardonnay)	300 g/L	0.414	1796	4/1.656	1	Bunches	1.0; 0.98	FR-01-0308 *duplicate analysis
		0.414	1796	4/1.656	7	Bunches	3.2; 1.9	
		0.414	1796	4/1.656	14	Bunches	1.6; 1.8	
		0.414	1796	4/1.656	21	Bunches Juice Wine Dry pomace	1.2; 1.3 (1.3) 0.04; 0.05; 0.04 < 0.01; < 0.01; < 0.01 4.2, 4.0*	
		0.414	1796	4/1.656	28	Bunches	0.72; 1.6	
		0.414	1796	4/1.656	35	Bunches	1.7; 2.3	
1714/90/GRA T-7544, Hughson, CA, USA, 1990 (Chenin Blanc)	300 g/L	0.224	224	4/0.896	21	Bunches	0.70: 0.92 (0.81)	FR-01-0335
1714/90/GRA T-7545, Fresno, CA, USA, 1990 (Thompson Seedless)	300 g/L	0.224	234	4/0.896	21	Bunches	0.53; 0.53 (0.53)	FR-01-0335 * sampled in field or processing plant ** duplicate analysis of one sample
		0.448	234	4/1.792	21	Bunches* Raisins*	0.75; 0.93 (0.84) 3.3; 3.1	
		0.448	234	4/1.792	21	Bunches * Raisins * Hydrated Raisin Raisin Waste Juice Wet pomace Dry pomace	0.53 3.3, 3.1** 2.4, 2.3** 6.0, 6.0 ** 0.13, 0.12** 1.1, 1.1** 2.0, 2.1**	
V-22939-A 22939/FR-0374, Madera, CA, USA, 2001 (Thompson Seedless)	300 g/L	0.465	935	2/0.964	21	Fruit	1.7; 1.6 (1.7)	FR-0374
		0.499	1010					
V-22939-B 22939/FR-0374, Fresno, CA, USA, 2001 (Thompson	300 g/L	0.460	926	2/0.927	21	Fruit	1.7; 1.5 (1.6)	FR-0374
		0.467	935					

GRAPES	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No./ Total kg ai/ha	days		mg/kg	
Seedless)								
V-22939-B 22939/FR-0374, Fresno, CA, USA, 2001 (Thompson Seedless)	300 g/L	0.924	926	2/1.863	21	Fruit	2.9; 2.7	FR-0374
		0.939	945	‘				

Assorted Tropical and Subtropical Fruits – Edible Peel

Olives

Three supervised trials were conducted on olives in the USA during 2005 (Samoil, 200; Reference FR-0561). Each trial consisted of one treated and one untreated plot, except CA64, which had two treated plots. At each trial, two applications of fenpropathrin 2.4 EC were made 7 days apart to treated plots at the rate of 0.45 to 0.48 kg ai/ha for a total of 0.9 kg ai/ha. In one plot for trial CA64, two applications at 5× rate (2.3 kg ai/ha for a total of 4.64 kg ai/ha) were made, to provide samples for processing into olive oil.

Samples of mature olive fruits were taken 7–8 and 14–15 days after the last treatment. Pits were removed from the olives. Samples were immediately frozen at -20 ºC until analysis in 200 days. The samples were analysed for residues of fenpropathrin using the method “Determination of fenpropathrin in olive, Revision 0”, a method based on method RM-22-4. The method was validated with an LOQ of 0.02 mg/kg for olives. Concurrent recoveries obtained during sample analysis ranged from 69 to 103% for olive fruit without pits. Note that apparent residues at levels of 0.01–0.03 mg/kg were seen in all control samples of olives without pits. The report explained that this should have no adverse effect on the data for treated olives since the residue levels obtained were at least 40× the magnitude of those from the controls.

Table 42 Residues of fenpropathrin in olives resulting from supervised trials in the United States

OLIVES	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./ Total kg ai/ha	days		mg/kg	
GAP, USA	300 EC (2.4 lb ai/gal)	0.22- 0.34	935	2/0.9	7			
CA63 Davis, CA, USA, 2005 (Manzanillo)	300 EC	0.45 0.45	776 786	2/0.9	7	Olive fruit, pitted	2.1, 2.3 (2.2)	FR-0561
					14	Olive fruit, pitted	1.8, 1.6 (1.7)	
CA64 Orange Cove, CA, USA, 2005	300 EC	0.46 0.46	963 945	2/0.92	7	Olive fruit, pitted	1.8, 2.0 (1.9)	FR-0561
					14	Olive fruit,	1.5, 2.3	

OLIVES	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./ Total kg ai/ha	days		mg/kg	
(Manzanillo)						pitted	(1.9)	
CA65 Healdsburg, CA, USA, 2005 (Leccino)	300 EC	0.48 0.47	505 496	2/0.95	8	Olive fruit, pitted	3.7, 3.4 (3.6)	FR-0561
					15	Olive fruit, pitted	1.2, 2.3 (1.8)	

Fruiting Vegetables, Cucurbits

Cucumber

Six supervised trials on cucumber were conducted in the USA in 1994 and 1996 (Samoil, 1999; Reference FR-0556). The EC formulation of fenpropathrin containing 300 g/L active ingredient, was applied 3 to 5 times to treated plots at the rate of 0.21 to 0.24 kg ai/ha for a total application of 0.7 to 1.1 kg ai/ha per plot. Applications were made at weekly intervals. At maturity, 6 to 8 days after the last application, samples were harvested and immediately frozen at -20 ºC. Samples were analysed within the storage stability period with RM-22-2 (LOQ of 0.01 mg/kg). Concurrent recoveries from fortified samples analysed together with the field samples ranged from 66–115% (mean = 89%, RSD= 13.8%, n=15).

Table 43 Residues of fenpropathrin in cucumber resulting from supervised trials in the United States

CUCUMBER	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total/ kg ai/ha	days		mg/kg	
GAP, USA	300 EC	0.22- 0.34	187- 935	0.9	7			
94-FL50 Gainesville, FL, USA, 1994 (Comet II)	300 EC	0.22 0.24 0.24	289 283 292	3/0.7	7	Whole fruit	0.05, 0.06, 0.04 (0.05)	FR-0556
94-OH*14 Fremont, OH USA, 1994 (Carolina)	300 EC	0.22 0.22 0.22	631 631 636	3/0.7	8	Whole fruit	< 0.01, < 0.01 (< 0.01)	FR-0556
96-TX*37 Weslaco, TX, USA, 1996 (Napoleon)	300 EC	0.22	281	4/0.9	7	Whole fruit	< 0.01, 0.01 (0.01)	FR-0556
96-GA*26 Tifton, GA, USA, 1996 (Straight Eight)	300 EC	0.22	281	4/0.9	7	Whole fruit	0.01, < 0.01 (0.01)	FR-0556
96-SC*16 Charleston, SC USA, 1996 (Regal)	300 EC	0.22	222	4/0.9	7	Whole fruit	< 0.01, < 0.01 (< 0.01)	FR-0556
96-OH*28	300 EC	0.22	742 (3x)	5/1.1	7	Whole fruit	< 0.01,	FR-0556

CUCUMBER	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total/ kg ai/ha	days		mg/kg	
Fremont, OH USA, 1996 (Score)			757 461				< 0.01 (< 0.01)	
96-MD02 Salisbury, MD USA, 1996 (Thunder)	300 EC	0.22 0.22 0.21 0.21	215 220 212 213	4/0.86	7	Whole fruit	0.03, 0.02 (0.03)	FR-0556
96-IN02 Lafayette, IN USA, 1996 (SMR 58)	300 EC	0.22 0.21 0.22 0.22	315 306 314 314	4/0.87	7	Whole fruit	< 0.01, < 0.01 (< 0.01)	FR-0556

Melon

Ten supervised trials on melons (cantaloupe) were conducted in the USA in 1994 (Green, 1995; Reference FR-0375). The EC formulation of fenpropathrin containing 300 g/L active ingredient, was applied 4 times to treated plots at the rate of 0.22 kg ai/ha for a total application of 0.9 kg ai/ha per plot. Applications were made at weekly intervals using tractor mounted boom sprayers or CO_2-powered backpack sprayers. At maturity, 7 days after the last application, samples were harvested and immediately frozen at -20 ºC. In two trials, samples were also collected 1, 3, and 14 days, and on days 3 and 7 after application. In two trials samples of treated melons were sectioned, separating the rind from the flesh. The samples were stored up to 107 days before analysis. Samples were analysed by method RM-22-4, which was validated to an LOQ of 0.01 mg/kg. Concurrent recoveries from fortified samples analysed together with the field samples ranged from 82–98% (mean = 90%, RSD= 4.3%, n=16).

Table 44 Residues of fenpropathrin in melon resulting from supervised trials in the United States

MELON	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		(mg/kg)	
GAP, USA	300 EC	0.22- 0.34	187- 935	0.9	7			
V-10868-B Ripon, CA USA, 1994 (Magnum)	300 EC	0.22 0.45	281- 290	2/0.88 4/1.8	7 7	Melon	0.2, 0.23 (0.22) 0.51, 0.64 (0.58)	FR-0375
V-10868-C Ripon, CA USA, 1994 (Magnum)	300 EC	0.22	281	4/0.88	1 3 7 14	Melon	0.24, 0.40 (0.32) 0.45, 0.35 (0.40) 0.31, 0.23 (0.27) 0.23, 0.25	FR-0375

MELON	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		(mg/kg)	
							(0.24)	
V-10868-D Uvalde, TX USA, 1994 (Explorer)	300 EC	0.22	281	4/0.88	7	Melon	0.10, 0.14 (0.12)	FR-0375
V-10868-E Uvalde, TX USA, 1994 (Explorer)	300 EC	0.22 0.45	271 281	4/0.88 4/1.8	7	Melon	0.14, 0.15 (0.15) 0.24, 0.21 (0.23)	FR-0375
V-10868-F Somerton, AZ USA, 1994 (Top Mark)	300 EC	0.22	281	4/0.88	7	Melon	0.12, 0.12 (0.12)	FR-0375
V-10868-G Fishers, Indiana USA, 1994 (Iroqouis)	300 EC	0.22 0.45	131 140	4/0.88 4/1.8	7 7	Melon	0.26, 0.28 (0.27) 0.46, 0.34 (0.40)	FR-0375
V-10868-H Cory, Colorado USA, 1994 (Mission Hybrid)	300 EC	0.22	281	4/0.88	7	Melon	0.08, 0.10 (0.09)	FR-0375
V-10868-I Hawkinsville, GA, USA, 1994 (Schoons)	300 EC	0.22	187	4/0.88	3 7	Melon	0.16, 0.07 (0.12) 0.06, 0.08 (0.07)	FR-0375
V-10868-J Germansville, PA USA, 1994 (Ball 1776)	300 EC	0.25 0.49	187 215	4/1.0 4/2.0	7 7	Whole melon Rind Centre Melon	0.17, 0.23 (0.20) 0.39, 0.36 (0.38) < 0.01, < 0.01 (< 0.01) 0.30, 0.21	FR-0375
V-10868-L Dos Palos, CA USA, 1994 (Iron Horse)	300 EC	0.22	187	4/0.88	7	Melon Rind Centre	0.20, 0.29 (0.25) 0.28, 0.35 (0.32) < 0.01, < 0.01 (< 0.01)	FR-0375

Squash

Seven supervised trials on summer squash were conducted in the US in 1994 and 1996 (Samoil, 1999; Reference FR-0557). The EC formulation of fenpropathrin containing 300 g/L active ingredient, was applied 4 times to treated plots at the rate of 0.2 to 0.25 kg ai/ha for a total application of 0.85 to 0.98 kg ai/ha per plot. Applications were made at weekly intervals. Samples were analysed with method RM-22-2 (LOQ of 0.01 mg/kg). Concurrent recoveries from fortified samples analysed together with the field samples ranged from 80–116% (mean = 97%, RSD= 11.8%, n=11).

Table 45 Residues of fenpropathrin in squash resulting from supervised trials in the United States

SQUASH	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No./ Total kg ai/ha	days		mg/kg	
GAP, USA	300 EC (2.4 lb ai/gal)	0.22- 0.34	187-935	2/0.9	7			
94-NJ33 Bridgeton, NJ, USA, 1994 (Lemon Drop)	300 EC	0.22	496 - 533	4/0.89	7	Summer squash	0.01, 0.02 (0.02)	FR-0557
94-FL75 Gainesville, FL, USA, 1994 (Dixier Hybrid)	300 EC	0.25 0.25 0.25 0.23	309	4/0.98	7	Summer squash	0.03, 0.04 (0.04)	FR-0557
94-WA*61 Moxee, WA, USA, 1994 (Early Straightneck)	300 EC	0.22	369	4/0.89	8	Summer squash	< 0.01, < 0.01 (< 0.01)	FR-0557
96-OH*10 Fremont, OH, USA, 1996 (Zucchini Select Hybrid)	300 EC	0.21 0.22 0.22 0.20	730 739 739 917	4/0.85	7	Summer squash	< 0.01, < 0.01 (< 0.01)	FR-0557
96-CA26 Fremont, CA, USA, 1996 (Chefini)	300 EC	0.21 0.20 0.23 0.25	243 243 290 243	4/0.89	6	Summer squash	0.01, 0.03 (0.02)	FR-0557
96-MD01 Salisbury, MD, USA, 1996 (Tigress)	300 EC	0.21 0.21 0.22 0.21	421 421 430 421	4/0.85	7	Summer squash	0.04, 0.02 (0.03)	FR-0557
96-IN01 Lafayette, IN, USA, 1996 (Black Beauty)	300 EC	0.22 0.21 0.22 0.21	318 309 318 309	4/0.86	7	Summer squash	< 0.01, < 0.01 (< 0.01)	FR-0557

Fruiting Vegetables other than Cucurbits

Tomato

Nine supervised trials conducted on tomatoes in the USA in 1993 (Green, 1994; Reference FR-41-0360). Treated plots received 4 applications of an EC formulation containing 300 g/L fenpropathrin, at the rate of 0.22 kg ai/ha, for a total application of 0.88 kg ai/ha. Mature tomato samples were collected 3 days after the last application and for decline trials, samples were also collected 1, 14, and 21 days after the last application. Upon collection, samples were stored at -20 ºC until analysis. All samples from this study were analysed within this validated storage interval. Residues of fenpropathrin were determined by method RM-22-4 with modifications. Concurrent recoveries from fortified samples analysed together with the field samples were within the acceptable range of 70 to 120%.

Table 46 Residues of fenpropathrin in tomato resulting from supervised trials in the United States

TOMATO	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
GAP, USA	300 EC (2.4 lb ai/gal)	0.17- 0.22	234 - 1122	0.9	3			
V-1032-A Stanislaus County, CA, USA, 1992 (UC-82)	300 EC	0.22 0.22 0.22 0.22 0.22 0.22	327 327 532 529 527 532	6/1.34	3	Whole fruit	0.34, 0.61 (0.48)	FR-41-0360
		1.0 1.0 1.0 1.0 1.0 1.0	327 327 532 529 527 532	6/6.0	3	Whole fruit	2.9, 2.5 (2.7)	
V-10633-A San Joaquin County, CA, USA, 1993 (Roma VF)	300 EC	0.22	701	4/0.88	3	Whole fruit	0.24, 0.18 (0.21)	FR-41-0360
V-10633-B San Joaquin County, CA, USA, 1993 (UC-82B)	300 EC	0.22	468	4/0.88	3	Whole fruit	0.22, 0.32 (0.27)	FR-41-0360
		0.45	468	4/1.8	3	Whole fruit	0.49, 0.49 (0.49)	FR-41-0360
V-10633-C Merced County, CA, USA, 1993 (Apex 1000)	300 EC	0.22	468	4/0.88	1	Whole fruit	0.38, 0.46 (0.42)	FR-41-0360
					3		0.30, 0.29 (0.30)	
					7		0.28, 0.27 (0.28)	
					14		0.19, 0.24 (0.22)	
V-10633-D Fayette County, OH, USA, 1993 (Heinz 9035))	300 EC	0.22	281	4/0.88	3	Whole fruit	0.64, 0.46 (0.55)	FR-41-0360
V-10633-E Ottawa County, MI, USA, 1993 (Rio Grande)	300 EC	0.22 0.22 0.22 0.22	302 286 320 296	4/0.88	3	.Whole fruit	0.04, 0.06 (0.05)	FR-41-0360
		0.45 0.45 0.45 0.45	295 293 314 290	4/1.8	3	Whole fruit	0.21, 0.13 (0.17)	FR-41-0360
V-10633-F	300 EC	0.22	217	4/0.88	3	Whole fruit	0.12, 0.10	FR-41-0360

TOMATO	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
Hamilton County, IN, USA, 1993 (Beefmaster)		0.22 0.22 0.22	211 238 238				(0.11)	
V-10633-G Hunterdon County, NJ, USA, 1993 (Better Boy)	300 EC	0.22 0.22 0.22 0.22	311 306 306 306	4/0.88	1	Whole fruit	0.21, 0.21 (0.21)	FR-41-0360
					3		0.18, 0.20 (0.19)	
					7		0.14, 0.13 (0.14)	
					14		0.09, 0.08 (0.09)	
V-10633-H Barnwell County, SC, USA, 1993 (Celebrity)	300 EC	0.22	281	4/0.88	3	Whole fruit	0.08, 0.07 (0.08)	FR-41-0360
V-10633-I Hidalgo County, TX, USA, 1993 (Florade)	300 EC	0.22	281	4/0.88	3	Whole fruit	0.15, 0.21 (0.18)	FR-41-0360

Peppers

A total of ten supervised trials on peppers (six on bell and four on non-bell) were conducted in the US in 1996 and 1998 (Samoil, 2001; Reference FR-0558). The application rates corresponded to US GAP (0.22 kg ai/ha with a total application of 0.9 kg ai/ha per season, but samples were taken at 2 and 4 days instead of the 3 day PHI. Samples of mature peppers were immediately frozen at -25 ºC to -15 ºC until analysis. Samples were analysed for residues of fenpropathrin using a modification of method RM-22-4. All recoveries were conducted concurrently with the analyses of treated samples and storage stability samples. Non-bell peppers fortified with fenpropathrin at 0.02, 0.10, 0.50, and 10 mg/kg showed respective recoveries of 100-130%, 95%, 92%, and 88-104%. Bell peppers were fortified with fenpropathrin at 0.02, 0.10, 0.40, 0.50 and 1.0 mg/kg and recoveries were 100–107%, 97–101%, 94–106% and 98%, respectively.

Table 47 Residues of fenpropathrin in peppers resulting from supervised trials in the United States

PEPPER	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
GAP, USA	300 EC (2.4 lb ai/gal)	0.17- 0.22	234 - 1122	0.9	3			
BELL PEPPER								
96-CA*09 Spence County, Salinas, CA, USA, 1996 (Cal Wonder)	300 EC	0.22 0.22 0.22 0.22	430 440 440 430	4/0.88	2	Whole fruit	0.37, 0.36 (0.37)	FR-0558

PEPPER	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
96-CA*10 East Alisal, Salinas, CA, USA, 1996 (Cal Wonder)	300 EC	0.22 0.22 0.22 0.22	440 430 440 439	4/0.88	2	Whole fruit	0.31, 0.36 (0.34)	FR-0558
96-FL12 NW71st St, Gainesville, FL, USA, 1996 (Capistrano)	300 EC	0.22 0.22 0.22 0.22	290 290 281 281	4/0.88	2	Whole fruit	0.64, 0.70 (0.67)	FR-0558
98-GA*10 Goat Rd., Tifton, GA, USA, 1998 (Camelot)	300 EC	0.22 0.22 0.22 0.22	290 281 281 281	4/0.88	2	Whole fruit	0.40, 0.33 (0.37)	FR-0558
98-MD02 Nanticoke Rd, Salisbury, MD, USA, 1998 (King Arthur)	300 EC	0.22 0.22 0.22 0.22	496 505 505 505	4/0.88	3	Whole fruit	0.48, 0.51 (0.50)	FR-0558
98-SC*12 Savannah Hwy, Charleston, SC, USA, 1998 (Keystone Resistance Giant)	300 EC	0.22 0.22 0.22 0.22	374 374 374 393	4/0.88	4	Whole fruit	0.08, 0.11 (0.10)	FR-0558
NON-BELL PEPPER								
96-CA*09 Spence County, Salinas, CA, USA, 1996 (Jalapeno)	300 EC	0.23 0.22 0.22 0.22	430 440 440 430	4/0.89	2	Whole fruit	0.24, 0.23 (0.24)	FR-0558
96-CA*10 East Alisal, Salinas, CA, USA, 1996 (Jalapeno))	300 EC	0.23 0.22 0.22 0.22	440 430 440 439	4/0.89	2	Whole fruit	0.36, 0.43 (0.40)	FR-0558
96-GA*05 Goat Rd., Tifton, GA, USA, 1996 (Mesilla)	300 EC	0.22 0.22 0.22 0.22	281 281 281 281	4/0.88	2	Whole fruit	0.32, 0.29 (0.31)	FR-0558
96-TX*04 Weslaco, TX, USA, 1996 (Sonora Anaheim)	300 EC	0.22 0.22 0.22 0.22	281 281 281 281	4/0.88	2	Whole fruit	0.33, 0.43 (0.38)	FR-0558

Pulses

Soya beans

Eight supervised trials on soya beans were conducted in Brazil in 2010 and 2013 (Tomaz, 2010; Reference: FR-0570 and Lopez, 2013; Reference: FR-0571). The EC formulation of fenpropathrin containing 300 g/L active ingredient, was applied to treated plots at the rate of 0.045 kg ai/ha from 15 to 45 days prior to harvest. At maturity, 15, 30 and 45 days after the last application, samples were harvested, threshed manually, cleaned, sieved, air-dried and stored frozen at -20 ºC. Samples were analysed within 30 days of harvest by GC-ECD (2010 trials) and LC-MS/MS (2013 trials) (LOQ of 0.01 mg/kg). Concurrent recoveries were within the acceptable range of 70–120%, with RSD below 20%.

Table 48 Residues of fenpropathrin in soya beans resulting from supervised trials in Brazil

SOYA BEAN	Application			DLA	Sample	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	No.	days		mg/kg	
GAP, Brazil	300 g/L EC	0.045	1	30			
Pereiras/SP Brazil, 2010 (BRS 232)	300 EC	0.045	1	15 30 45	Seeds	< 0.01 < 0.01 < 0.01	FR-0570
Iracemápolis/SP Brazil, 2010 (BRS 245)	300 EC	0.045	1	15 30 45	Seeds	< 0.01 < 0.01 < 0.01	FR-0570
Cambará/PR Brazil, 2010 (BRS 245)	300 EC	0.045	1	15 30 45	Seeds	< 0.01 < 0.01 < 0.01	FR-0570
Santa Mariana/PR Brazil, 2010 (BRS 245)	300 EC	0.045	1	15 30 45	Seeds	< 0.01 < 0.01 < 0.01	FR-0570
Trial C01 Ibiporã/PR Brazil, 2012-2013 (Potência)	300 EC	0.045	1	15 30 45	Seeds	< 0.01 < 0.01 < 0.01	FR-0571
Trial C53 Câdido Mota/SP Brazil, 2012-2013 (Potência)	300 EC	0.045	1	30	Seeds	< 0.01	FR-0571
Trial C56 Jataizinho/PR Brazil, 2012-2013 (Força)	300 EC	0.045	1	15 30 45	Seeds	< 0.01 < 0.01 < 0.01	FR-0571
Trial C109 Dourados/MS Brazil, 2012-2013 (Potência)	300 EC	0.045	1	30	Seeds	< 0.01	FR-0571

Oilseeds

Cotton

Thirty-two supervised trials on cotton were conducted in the USA in 1983-1989, using a higher number of applications compared to the GAP in the USA (up to 0.9 kg ai/ha and a PHI of 21 days) (Fujie, 1990Reference: FR-01-0306). The EC formulation of fenpropathrin containing 300 g/L active ingredient, was applied to treated plots at the rates of 0.112-0.448 kg ai/ha from 18 to 35 days prior to harvest. Samples were analysed within 12–18 months of harvest. Concurrent recoveries from control samples fortified with fenpropathrin at levels of 0.1 and 0.2 mg/kg were within the acceptable range of 70–120%, with RSD below 20%.

Table 49 Residues of fenpropathrin in cotton fuzzy seed resulting from supervised trials in the USA

COTTON	Application				DAT	Commodity	Residue mg/kg	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No/Total kg ai/ha	days			
GAP, USA	300 EC	0.22-0.45		0.9	21			
R194 M335-6023 Bard, California, USA, 1983 (DPL-61)	300 EC	0.224	281	7/1.79	35	Fuzzy seed	< 0.01	FR-01-0306
R194 M335-6023 Bard, California, USA, 1983 (DPL-61)	300 EC	0.448	281	8/3.58	35	Fuzzy seed	< 0.01	FR-01-0306
R194 M335-6024 Arizona, USA, 1983 (Delta Pine 70)	300 EC	0.112	117	8/0.896	33	Fuzzy seed	< 0.01, < 0.01 (< 0.01)	FR-01-0306
R194 M335-6069* Yuma, Arizona, USA, 1984 (DPL-90)	300 EC	0.224	47	10/2.24	18	Fuzzy seed	0.01, < 0.01 (< 0.01)	FR-01-0306
R194 M335-6070 Donna, Texas, USA, 1984 (McNair 220)	300 EC	0.224	94	10/2.02	21	Fuzzy seed	0.02, 0.02 (0.02)	FR-01-0306
R194 M335-6071 Kingsville, Texas, USA, 1984 (GP-3774)	300 EC	0.224	23-94	10/2.24	18	Fuzzy seed	0.03, 0.03 (0.03)	FR-01-0306
R194 M335-6072A Bard, California, USA, 1984 (DPL-90)	300 EC	0.224	94	10/2.24	21	Fuzzy seed	< 0.01, < 0.01 (< 0.01)	FR-01-0306
R194 M335-6073 Fresno, California, USA, 1984 (Acala-SJ-5)	300 EC	0.224	430-702	10/2.24	21	Fuzzy seed	0.01, < 0.01 (0.01)	FR-01-0306
R194 M335-6074 Holtville California, USA, 1984 (Delta Pine 61)	300 EC	0.224	234	10/2.46	34	Fuzzy seed	0.33, 0.18 (0.26)	FR-01-0306
R194 M335-6075 Tifton, Georgia, USA, 1984	300 EC	0.224	99	10/2.24	21	Fuzzy seed	< 0.01, < 0.01 (< 0.01)	FR-01-0306

COTTON	Application				DAT	Commodity	Residue mg/kg	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No/Total kg ai/ha	days			
(McNair 220)								
R194 M335-6076 Greenville, Mississippi, USA, 1984 (Stoneville 825)	300 EC	0.224	94	10/2.24	20	Fuzzy seed	0.33, 0.25 (0.29)	FR-01-0306
R194 R194-6418 Fresno, California, USA, 1985 (SJ-5)	300 EC	0.224	281	10/2.24	3	Fuzzy seed	0.12, 0.14 (0.13)	FR-01-0306
					10		0.08, 0.21 (0.15)	
					14		0.03, 0.02 (0.03)	
					21		0.11, 0.13 (0.12)	
					28		< 0.01, < 0.01 (< 0.01)	
					35		< 0.01, < 0.01 (< 0.01)	
R194 R194-6419 Blythe, California, USA, 1985 (SJ-5)	300 EC	0.112	281	10/1.12	21	Fuzzy seed	< 0.01, 0.03 (0.02)	FR-01-0306
R194 R194-6419 Blythe, California, USA, 1985 (DPL-90)	300 EC	0.224	281	10/2.24	21	Fuzzy seed	0.01, < 0.01 (0.01)	FR-01-0306
R194 R194-6419 Blythe, California, USA, 1985 (DPL-90)	300 EC	0.448	281	10/4.48	21	Fuzzy seed	< 0.01, 0.06 (0.04)	FR-01-0306
R194 R194-6420* Maricopa, Arizona, USA, 1985 (Pima S-2)	300 EC	0.224	94	10/2.24	21	Fuzzy seed	0.02, 0.02 (0.02)	FR-01-0306
R194 R194-6421* Yuma, Arizona, USA, 1985 (DPL-90)	300 EC	0.224	47	10/2.24	21	Fuzzy seed	< 0.01, < 0.01 (< 0.01)	FR-01-0306
R194 R194-6422 College station, Texas USA, 1985 (DPL-41)	300 EC	0.224	42	10/2.24	21	Fuzzy seed	0.09, 0.07 (0.08)	FR-01-0306
R194 R194-6423 Kingsville, Texas, USA, 1985 (SP-37)	300 EC	0.224	47	10/2.24	3	Fuzzy seed	3.2, 3.4 (3.3)	FR-01-0306
					7		0.95, 0.56 (0.76)	
					14		0.19, 0.21 (0.20)	

COTTON	Application				DAT	Commodity	Residue mg/kg	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No/Total kg ai/ha	days			
					21		0.02, 0.02 (0.02)	
					28		< 0.01, 0.02 (0.02)	
					35		< 0.01, < 0.01 (< 0.01)	
R194 R194-6424 Donna, Texas USA, 1985 (McNair 220)	300 EC	0.224	94	10/2.24	21	Fuzzy seed	< 0.01, < 0.01 (< 0.01)	FR-01-0306
R194 R194-6425 Tifton Georgia USA, 1985 (Coker 304)	300 EC	0.224	94	10/2.24	22	Fuzzy seed	< 0.01, < 0.01 (< 0.01)	FR-01-0306
R194 R194-6426 ArkansasUSA, 1985 (Stoneville 825)	300 EC	0.224	47	10/2.24	30	Fuzzy seed	0.02, < 0.01 (0.02)	FR-01-0306
R194 R194-6427 Greenville Mississippi USA, 1985 (DPL-41)	300 EC	0.112	94	10/1.12	21	Fuzzy seed	0.08, 0.09 (0.09)	FR-01-0306
R194 R194-6427 Greenville Mississippi USA, 1985 (DPL-41)	300 EC	0.224	94	10/2.24	3	Fuzzy seed	0.56, 0.47 (0.52)	FR-01-0306
					14		0.47, 0.46 (0.47)	
					21		0.32, 0.40 (0.36)	
					36		0.30, 0.31 (0.31)	
R194 R194-6427 Greenville Mississippi USA, 1985 (DPL-41)	300 EC	0.448	94	10/4.48	7	Fuzzy seed	1.2	FR-01-0306
R194 R194-6715 Greenville Mississippi USA, 1986 (Acala SJ-5)	300 EC	0.224	281	8/1.792	21	Fuzzy seed	1.3, 1.1, 0.95, 0.77 (1.03)	FR-01-0306
R194 R194-6716 Case Grande, Arizona USA, 1986 (DPL-41)	300 EC	0.224	281	8/1.792	20	Fuzzy seed	0.59, 0.62 , 0.47, 0.43 (0.53)	FR-01-0306
R194 R194-6717 Greenville, Mississippi	300 EC	0.224	94	8/1.792	21	Fuzzy seed	0.08, 0.05 (0.07)	FR-01-0306

COTTON	Application				DAT	Commodity	Residue mg/kg	Reference
Trial Country, year (Variety)	Formulation (g ai/l)	kg ai/ha	Water (l/ha)	No/Total kg ai/ha	days			
USA, 1986 (Delta Pine 90)								
R194 R194-6718 Kleberg County, Texas USA, 1986 (GP 3774)	300 EC	0.224	47	8/1.792	21	Fuzzy seed	0.08, 0.06 (0.07)	FR-01-0306
R194 R194-6967Fresno, California USA, 1987 (GC 510)	300 EC	0.224	19	8/1.792	21	Fuzzy seed	0.22, 0.32 (0.27)	FR-01-0306
R194 R194-6968 Glen Alan, Mississippi USA, 1987 (DPL-50)	300 EC	0.224	19	8/1.792	21	Fuzzy seed	< 0.01, < 0.01 (< 0.01)	FR-01-0306
R194 R194-7376 Greenville, Mississippi USA, 1989 (DPL-41)	300 EC	0.336	47	5/1.68	21	Fuzzy seed	0.02, 0.02 (0.02)	FR-01-0306
R194 R194-7377 Grangerburg, Alabama USA, 1989 (McNair 220)	300 EC	0.336	468	5/1.68	21	Fuzzy seed	0.01, 0.01 (0.01)	FR-01-0306
R194 R194-7378 Rosa, Louisiana USA, 1989 (DPL-41)	300 EC	0.336	468	5/1.68	20	Fuzzy seed	0.29, 0.27 (0.28)	FR-01-0306
R194 R194-7379 Proctor, Arkansas USA, 1989 (DPL-41)	300 EC	0.336	468	5/1.68	21	Fuzzy seed	0.04, 0.07 (0.06)	FR-01-0306
R194 R194-7380 Fresno, California USA, 1989 (GC 510)	300 EC	0.336	47	5/1.68	21	Fuzzy seed	0.07, 0.05 (0.06)	FR-01-0306
R194 R194-7381* Greenville, Mississippi USA, 1989 (DPL-41)	300 EC	0.336	9	5/1.68	21	Fuzzy seed	0.03, 0.02 (0.03)	FR-01-0306
R194 Eakly, Oklahoma USA, 1989 (Paymater 145)	300 EC	0.336	9	5/1.68	21	Fuzzy seed	0.03, 0.04 (0.04)	FR-01-0306

Tree Nuts

Almonds and pecans

A total of ten supervised trials on tree nuts, five on almonds and five on pecans have been conducted in the US in 2003, following the GAP for tree nuts (Green, 2004; References FR-0382 and FR-0383). Two applications of fenpropathrin 2.4 EC were made to each treated plot at the rate of 0.45 (1×) or 0.90 kg ai/ha (2×). Applications were made at 7-day intervals. Samples of almond hulls as well as almond and pecan nutmeats were collected 3 days after the last application. At one site, samples were also collected at 1, 7, and 14 days after the last application.

All samples were immediately frozen and stored at -20 ºC until analysis. Residues of fenpropathrin in nutmeats were analysed using method RM-22-4. The method was modified for analysis of a 10 gram (instead of a 20 gram) sample. The LOQ remained 0.01 mg/kg. Recoveries obtained for the fortified almond and pecan nutmeat samples were between 83.5 and 119%, and 70.9 and 89.6%, respectively.

Table 50 Residues of fenpropathrin in tree nuts resulting from supervised trials in the United States

TREE NUTS	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
GAP, USA	300 EC (2.4 lb ai/gal)	0.22-0.45	468 - 3740	0.9	3			
ALMONDS								
V-25398-A Glen County, CA, USA, 2003 (Non-Pareil)	300 EC	0.45 0.45	935 1038	2/0.9	3	Nutmeat	< 0.0.1, < 0.01 (< 0.01)	FR-0382
	300 EC	0.89 0.90	935 1029	2/1.8	3	Nutmeat	0.02, 0.02 (0.02)	
V-25398-B Stanislaus County, CA, USA, 2003 (Carmel)	300 EC	0.45 0.45	1160 1132	2/0.9	3	Nutmeat	< 0.01, < 0.01 (< 0.01)	FR-0382
V-25398-C Kern County, CA, USA, 2003 (Sornora)	300 EC	0.45 0.46	945 963	2/0.91	3	Nutmeat	0.02, 0.03 (0.03)	FR-0382
V-25398-D Fresno County, CA, USA, 2003 (Non-Pareil)	300 EC	0.46 0.45	945 935	2/0.91	1	Nutmeat	0.0.1, < 0.01 (< 0.01)	FR-0382
					3	Nutmeat	0.0.1, < 0.01 (< 0.01)	
					7	Nutmeat	< 0.01, < 0.01 (< 0.01)	
					14	Nutmeat	< 0.01, < 0.01 (< 0.01)	
V-25398-E Tulare County, CA, USA, 2003 (Carmel)	300 EC	0.45 0.45	945 945	2/0.9	3	Nutmeat	< 0.01, < 0.01 (< 0.01)	FR-0382
PECANS								
V-25401-A	300 EC	0.44	1160	2/0.87	3	Nutmeat	< 0.01,	FR-0383

TREE NUTS	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
Saluda County. SC, USA, 2003 (Cape Fear)		0.43	1132				< 0.01 (< 0.01)	
		0.90 0.90	1169 1160	2/1.8	3	Nutmeat	< 0.01, < 0.01 (< 0.01)	
V-25401-B Tift, GA, USA, 2003 (Summer)	300 EC	0.45 0.45	1188 1197	2/0.9	3	Nutmeat	0.01, < 0.01 (0.01)	FR-0383
V-25401-C Drew County, AR, USA, 2003 (Stewart)	300 EC	0.46 0.46	1169 1188	2/0.92	3	Nutmeat	0.02, 0.02 (0.02)	FR-0383
V-25401-D Burleson County, TX, USA, 2003 (Cheyenne)	300 EC	0.43 0.44	1394 1394	2/0.87	1	Nutmeat	0.02, 0.05 (0.04)	FR-0383
					3	Nutmeat	0.02, 0.02 (0.02)	
					7	Nutmeat	0.02, 0.10 (0.06)	
					14	Nutmeat	< 0.01, < 0.01 (< 0.01)	
V-25401-E Hockley County, TX, USA, 2003 (Western Schley)	300 EC	0.46 0.45	1197 1188	2/0.91	3	Nutmeat	0.04, 0.05 (0.05)	FR-0383

Coffee Beans

Six supervised trials on coffee were conducted in Brazil in 2013 (Gravena, 2013. Reference FR-0572). Two applications of fenpropathrin EC containing 300 g ai/L were made to each treated plot at the rate of 0.12 kg ai/ha (1×). Applications were made at 14-day intervals. Samples of coffee berries were collected 14 days after the last application. In two trials, samples were also collected at 7 and 21 days after the last application. All samples were collected manually and packed in polyethylene bags for further processing. The berries were immediately sent to the experimental station and maintained at room temperature until completion of drying. Samples were dried by opening the bags throughout the day and night, except during rainy days when the packages were sealed. After drying, the coffee beans were placed in a manual peeler and at least 1 kg of coffee beans were packed, labelled, and stored frozen at -20 ºC until analysis.

All samples were analysed LC-with LC-MS/MS (LOQ of 0.01 mg/kg) within 30 days following harvest. Average recovery from control samples fortified with fenpropathrin at levels of 0.01 and 0.5 mg/kg ranged from 79–116% (n=13; mean=98%; and RSD = 15.3%).

Table 51 Residues of fenpropathrin in coffee beans resulting from supervised trials in Brazil

COFFEE	Application	DAT	Sample	Residue	Reference

Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No.	days		mg/kg	
GAP, Brazil	300 g/L EC	0.12		2	14			
Trial 01 Iraí de Minas/MG Brazil, 2012 (IAPAR 59)	300 g/L EC	0.12	301 303	2	7 14 21	Coffee beans	< 0.01 0.01 < 0.01	FR-0572
Trial 02 Indianópolis, MG Brazil, 2012 (Catuai Amarelo)	300 g/L EC	0.12	308 294	2	14	Coffee beans	< 0.01	FR-0572
Trial 03 Rolândia/PR Brazil, 2012 (Novo Mundo)	300 g/L EC	0.12	285 297	2	7 14 21	Coffee beans	< 0.01 < 0.01 < 0.01	FR-0572
Trial 04 Bebedouro/SP Brazil, 2012 (Novo Mundo)	300 g/L EC	0.12	298 304	2	14	Coffee beans	< 0.01	FR-0572
Trial 05 Monte Azul Paulista/SP Brazil, 2012 (Novo Mundo)	300 g/L EC	0.12	293 308	2	14	Coffee beans	< 0.01	FR-0572
Trial 06 Araguari/MG Brazil, 2012 (Novo Mundo)	300 g/L EC	0.12	302 303	2	14	Coffee beans	0.02	FR-0572

Tea

All supervised trials on tea were conducted in India during 2002-2004 (Lavakumar, 2003 and 2006; References 1424604-2974-2002; 1424604-4190-2004). All trials were according to the GAP for tea in India, which consists of one application of fenpropathrin at the rate of 0.06 kg ai/ha and a PHI of 7 days.

In ten trials, the collected tea leaves were processed into black tea. In six trials this was achieved through withering, crush/tear/curl process, oxidation and drying while in other trials by machine drying.

In two additional trials, the collected tea leaves were air-dried to prepare green tea. Since growing conditions and application rate/method for black tea and green tea are equivalent with the only difference being in processing methods, results from both sets of trials can be combined to support an MRL and STMR for tea, green and black.

Residues of fenpropathrin were analysed by a method based on RM-22-4 with an LOQ of 0.05 mg/kg.

Table 52 Fenpropathrin residues in black and green tea from supervised trials in India

TEA	Application	DAT	Residues1	Reference

Country, month/year, season (variety)	Form	kg ai/ha	Water l/ha	No.	days	mg/kg	
GAP in India	300 g/L EC	0.05 – 0.06	200 – 400	1	7		
BLACK TEA							
Valparai, India January 2002 Season I (UPASI-9)	300 g/L EC	0.06	400	1	0	2.74	04-2974-2002
					1	1.75	
					3	1.17	
					5	0.61	
					7	0.17	
					10	< 0.05	
					14	< 0.05	
Valparai, India September 2002 Season II (UPASI-9)	300 g/L EC	0.06	400	1	0	2.69	04-2974-2002
					1	1.69	
					3	1.10	
					5	0.61	
					7	0.18	
					10	< 0.05	
					14	< 0.05	
Valparai, India May 2003 Season III (UPASI-9)	300 g/L EC	0.06	400	1	0	2.22	04-2974-2002
					1	1.45	
					3	0.91	
					5	0.49	
					7	0.14	
					10	< 0.05	
					14	< 0.05	
Valparai, India January 2002 Season I (UPASI-9)	300 g/L EC	0.12	400	1	0	5.47	04-2974-2002
					1	3.13	
					3	2.24	
					5	1.04	
					7	0.37*	
					10	< 0.05	
					14	< 0.05	
Valparai, India September 2002 Season II (UPASI-9)	300 g/L EC	0.12	400	1	0	5.24	04-2974-2002
					1	3.02	
					3	2.22	
					5	1.08	
					7	0.36*	
					10	< 0.05	
					14	< 0.05	
Valparai, India May 2003 Season III (UPASI-9)	300 g/L EC	0.12	400	1	0	4.40	04-2974-2002
					1	2.57	
					3	1.89	
					5	0.86	
					7	0.30*	
					10	< 0.05	
					14	< 0.05	
Valparai, India January 2004 Fourth Season (UPASI-9)	300 g/L EC	0.06	400	1	0	0.85	04-2974-2002
					1	0.50	
					3	0.17	
					5	< 0.05	
					7	< 0.05	
					10	< 0.05	
					14	< 0.05	
Valparai, India January 2004 Fourth Season (UPASI-9)	300 EC	0.12	400	1	0	1.62	04-2974-2002
					1	0.93	
					3	0.30	
					5	< 0.05< 0.05	
					7	< 0.05	
					10	< 0.05	
					14		
Gudalur, India June 2004 (Mixed clones)	300 EC	0.06	450	1	0	2.22	Submission by Government of India to JMPR 1993
					7	0.14	
					10	< 0.05	
					14	< 0.05	

TEA	Application				DAT	Residues1	Reference
Country, month/year, season (variety)	Form	kg ai/ha	Water l/ha	No.	days	mg/kg	
Tocklai, India November 2005 (Mixed clones)	300 EC	0.06	400	1	0 7 10	12.0 1.38 0.12	Submission by Government of India to JMPR 1993
GREEN TEA							
Valparai, India January 2004 Fourth Season (UPASI-9)	300 g/L EC	0.06	400	1	0 1 3 5 7 10 14	1.96 1.32 0.83 0.45 0.13 < 0.05 < 0.05	04-4190-2004
Valparai, India January 2004 Fourth Season (UPASI-9)	300 g/L EC	0.12	400	1	0 1 3 5 7 10 14	4.20 2.43 1.55 0.90 0.29* < 0.05 < 0.05	04-4190-2004

Animal feeds

The trial conditions are described under the corresponding food commodities.

Cottonseed hull

Samples were analysed within 12–18 months of harvest. Concurrent recoveries from control samples fortified with fenpropathrin at levels of 0.1 and 0.2 mg/kg were within the acceptable range of 70–120%, with RSD below 20%.

Table 53 Residues of fenpropathrin in cottonseed hulls resulting from supervised trials in the USA

COTTON	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (l/ha)	No/Total kg ai/ha	days		mg/kg	
GAP, USA	300 EC	0.22-0.45		0.9	21			
R194 M335-6070 Donna, Texas, USA, 1984 (McNair 220)	300 EC	0.224	94	9/2.02	21	Hulls	0.02, 0.02 (0.02)	FR-01-0306
R194 M335-6071 Kingsville, Texas, USA, 1984 (GP-3774)	300 EC	0.224	94	10/2.24	18	Hulls	0.03, 0.02 (0.03)	FR-01-0306
R194 R194-6427 Greenville Mississippi USA, 1985	300 EC	0.448	94	110/4.48	7	Hulls	1.0	FR-01-0306

COTTON	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (l/ha)	No/Total kg ai/ha	days		mg/kg	
(DPL-41)								

Almond hulls

All samples were immediately frozen and stored at -20 ºC until analysis. Residues of fenpropathrin in hulls were analysed using method RM-22-4. The method was modified for analysis of a 10 gram (instead of a 20 gram) sample. The LOQ remained 0.01 mg/kg. Recoveries obtained for the fortified almond hull samples were between 90.3 and 115%, with an average recovery of 103%.

Table 54 Residues of fenpropathrin in almond hulls resulting from supervised trials in the USA

TREE NUTS	Application				DAT	Commodity	Residue	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	kg ai/ha	Water (L/ha)	No/Total kg ai/ha	days		mg/kg	
GAP, USA	300 EC (2.4 lb ai/gal)	0.22- 0.45	468 - 3740	3/0.9	3			
V-25398-A Glen County, CA, USA, 2003 (Non-Pareil)	300 EC	0.45 0.45	935 1038	2/0.9	3	Hulls	2.4, 2.9 (2.7)	FR-0382
	300 EC	0.89 0.90	935 1029	2/1.8	3	Hulls	7.2, 7.3 (7.3)	
V-25398-B Stanislaus County, CA, USA, 2003 (Carmel)	300 EC	0.45 0.45	1160 1132	2/0.9	3	Hulls	2.7, 3.1 (2.9)	FR-0382
V-25398-C Kern County, CA, USA, 2003 (Sornora)	300 EC	0.45 0.46	945 963	2/0.91	3	Hulls	3.2, 2.9 (3.1)	FR-0382
V-25398-D Fresno County, CA, USA, 2003 (Non-Pareil)	300 EC	0.46 0.45	945 935	2/0.91	1	Hulls	4.7, 3.2 (3.9)	FR-0382
					3	Hulls	3.5, 3.4 (3.5)	
					7	Hulls	4.3, 2.7 (3.5)	
					14	Hulls	2.1, 4.5 (3.3)	
V-25398-E Tulare County, CA, USA, 2003 (Carmel)	300 EC	0.45 0.45	945 945	2/0.9	3	Hulls	4.3, 2.9 (3.6)	FR-0382

FATE OF RESIDUES IN STORAGE AND IN PROCESS

Processing studies on plums, tomato, olives, oranges, cottonseed and tea were conducted and are summarized in this section.

Processing orange into juice, oil, and other fractions

Orange samples from the following supervised trials treated at 1× or 2× the GAP application rate were sent for processing after harvest: Trials T-6389, T-6390, T-6640, T-6730, and T-6966 (Reference FR-01-0307). The study determined the magnitude of residues in raw unwashed and washed orange samples, peel, pulp, wet and dried peel, molasses, oil and juice.

Duplicate samples were harvested 1–7 days after the last application, and sent to the processor. Samples were stored frozen for up to 9 months before analysis by GC-ECD (method RM-22-4. Orange oil samples were extracted into hexane, partitioned with acetonitrile and extracts purified through alumina oxide (method RM-22-2).

Samples were variously washed by hand, separated into pulp and peel, and processed into wet and dried peel, molasses, oil and juice. Fruit was washed in an in-line foam washer with soap and rinsed with water. Juice was extracted using an automated FMC in-line extractor and the sample passed through a juice finisher with 200 mesh, the juice being collected in a holding tank.

The resulting oil, water, peel, frit emulsion from the FMC extractor was passed through an in-line FMC finisher with 200 mesh screen and collected in a holding tank. This emulsion was heated to 49°C and appropriate enzymes added. The emulsion was then centrifuged to separate out the oil. The peel, rag, frit and seeds (together called as peel) from the juice and oil finishers were deposited in a peel hopper. This was further processed by being shredded and combined with 0.3% dehydrated lime slurry.

The slurry was then pressed (4.82 bar) and molasses collected from the press. The remaining pulp from the press was dried to approximately 8% moisture content and passed through a cyclone separator and cooler and collected. Dry fines were collected from the bottom of the cyclone separator. Procedural recoveries were 77-115% (mean 95% ± 7% RSD, n=57) at fortification levels of 0.1–2.0 mg/kg.

Table 55 Residues of fenpropathrin in orange processed fractions

Processed Fractions	Residues (mg/kg)	Average (mg/kg)	Processing factor	Reference (Trial No.)
Orange RAC	0.43, 0.51	0.47		R195 T-6390
Juice		< 0.01	< 0.02	
Oil		37	79	
Molasses		< 0.01	< 0.02	
Wet peel		0.27	0.57	
Dried peel		0.74	1.6	
Dried fines of peel		0.66	1.4	
Orange (RAC)	0.04, 0.05	0.045		R195 T-6966
Washed fruit	0.02, 0.03	0.025	0.6	
Juice	< 0.01, < 0.01	< 0.01	< 0.2	
Oil	0.99, 0.94	0.97	22	
Molasses	0.01, 0.01	0.01	0.2	
Wet peel	0.04, 0.03	0.035	0.8	
Dried peel	0.13, 0.11	0.12	2.7	
Dried fines of peel	0.16, 0.16	0.16	3.6	
Orange (RAC)	0.22,0.11	0.17		R195 T-6604

Processed Fractions	Residues (mg/kg)	Average (mg/kg)	Processing factor	Reference (Trial No.)
Pulp		< 0.01	< 0.06	
Peel		0.47	2.8	
Orange (RAC	0.18, 0.18	0.18		R195 T-6730
Washed fruit		0.13	0.72	
Orange (RAC)	0.09, 0.19	0.14		R195 T-6389
Pulp		< 0.01	< 0.07	
Peel		0.40	2.9	

Processing plums to dried plums/prunes

Plums from trial V-25427-F [treated twice with 1.8 kg ai/ha (2x) per application] were processed to dried plums (Reference FR-0385) by drying in a dehydrator at 187ºC for approximately 18 hours. After drying, samples were frozen and analysed within 176 days of harvest of fresh plums.

In another study, fresh plum samples from Trial V-27220-A treated at the GAP rate of 0.9 kg ai/ha were dried in a drying tunnel at about 185 ºC (Reference FR-0559) for approximately 18 hours. The dried prunes were pitted by hand, bagged and frozen at -20 ºC until analysis within 195 days from harvest of fresh samples. The period of storage was within the demonstrated stability of plums under frozen conditions.

All samples were analysed for fenpropathrin using a modification of method RM-22-4 which is described under analytical methods. Concurrent recoveries of fortified samples ranged from 70–120%.

Table 56 Residues of fenpropathrin in fresh and dried plums

Matrix	Residues (mg/kg)	Average (mg/kg)	Processing factor	Reference (Trial No.)
Plum (RAC)	0.61, 0.54	0.58		FR-0385
Dried plum	0.59, 0.59	0.59	1.0	
Plum (RAC) – prior to processing	0.22, 0.23	0.23		FR-0559
Dried plum	0.58, 0.57	0.58	2.6	
Average			**1.8**	

Processing tomatoes to pomace, juice, paste and other fractions

The magnitude of residues in raw unwashed and washed tomatoes, canned and pre-canned tomatoes, wet and dry pomace and juice was determined following applications of a 2.4 EC at 1.5× GAP rate (Reference FR-21-0344). Duplicate samples of mature green fruit were sent to the processor. Certain samples were gas-ripened or vine ripened before being sampled for analysis. Samples were stored at -20 °C for up to 5 months before analysis by GC-ECD (method RM-22-4), as described above in section 3. Gas ripened fruit was held in an atmosphere of 0.008–0.015%v/v ethylene at 20 °C for 3–6 days before being sampled. Samples were variously washed in chlorine solution, waxed and/or processed into pre-canned and canned tomatoes, wet and dry pomace, juice and paste. The canning process involved washing in chlorine solution followed by a water rinse, peeling by placing in 20%

sodium hydroxide (lye) solution at 79 °C, rinsing in water, dipping in 1% citric acid solution and further rinsing. Once tomatoes were placed in the can hot (100°C) tomato juice was added and the can was sealed and heated for 55 minutes at 100 °C. Procedural recoveries were 83–112% (with the exception of one sample of green fruit at 130%) at fortification levels of 0.01–0.2 mg/kg.

In another study, the magnitude of residues in raw tomatoes, wet and dry pomace, juice and paste were determined following applications of a 2.4 EC at 7.5× GAP rates to commercial crops (Reference FR-41-0360). Duplicate samples of mature green fruit were harvested 3 days after the last application and sent to the processor. Samples were stored at -20 °C and were analysed within 5 months by GC-ECD (method RM-22-4). Samples were hand sorted and defective fruit were discarded as cannery waste. The tomatoes were then soaked in a 0.5% lye solution for 3 minutes at 54 °C and rinsed using a high pressure spray for 30 seconds. Samples were then batch steam peeled at 5.6–7 bar pressure and processed through a pulp finisher machine where the pulp/pomace was separated from juice. The wet pomace was dried to produce dry pomace and the juice concentrated in a batch vacuum concentrator to approximately 30–32% solids. 1% salt was added to the resulting paste and the temperature was raised to 90 °C before the paste was sealed in cans. The cans were subject to heating at 100 °C for 20 minutes in a water batch before being cooled under running tap water. Juice was reconstituted from the paste with water, salt and ascorbic acid, heated to 90 °C, canned, and the cans treated as for canned paste.

Procedural recoveries were 94–105% (with the exception of two samples of dry pomace at 78 and 118%) at fortification levels of 0.1 mg/kg.

Table 57 Residues of fenpropathrin in tomato processed fractions

Processed Fractions	Residues (mg/kg)	Average (mg/kg)	Processing factor	Reference (Trial No.)
Tomato gas ripe	0.07, 0.07	0.07		FR-21-0344
Washed fruit	0.03, 0.04	0.035	0.5	
Unwashed, pre-canned (RAC)		0.13		
Washed, pre-canned		0.07	0.54	
Pre-canning		< 0.01	< 0.08	
Canned		< 0.01	< 0.08	
Canning waste	0.95, 1.1	1.0	7.7	
Tomato gas ripe	0.12, 0.06	0.09		FR-21-0344
Washed fruit	0.07, 0.11	0.09	1	
Raw tomato (RAC)		0.14		
Washed, pre-canned		0.12	0.86	
Processed, pre-canned		0.01	0.07	
Canned		< 0.01	< 0.07	
Canning waste	0.83, 0.81	0.82	5.9	
Tomato gas ripe	0.05, 0.13	0.09		FR-21-0344
Washed gas ripe	0.04, 0.08	0.06	0.7	
Raw tomato (RAC)		0.13		
Washed fruit		0.14	1.1	
Processed pre-canned		0.02	0.15	
Canned		< 0.01	< 0.08	
Canning waste	1.2, 1.3	1.25	9.6	
Tomato gas ripe (RAC)	0.13, 0.03	0.08		FR-21-0344
Washed and waxed	0.05, 0.06	0.055	0.69	

Processed Fractions	Residues (mg/kg)	Average (mg/kg)	Processing factor	Reference (Trial No.)
Tomato gas ripe (RAC)	0.20, 0.10	0.15		FR-21-0344
Washed and waxed	0.08, 0.18	0.13	0.87	
Ripe fruit (RAC)	2.5, 2.9	2.7		FR-41-0360
Fruit from processor	1, 1.1	1.05	0.4	
Wet pomace	9.7, 11	10.4	3.9	
Dry pomace	51, 45	48	18	
Tomato paste	0.83, 0.81	0.82	0.3	
Tomato juice	0.12, 0.13	0.125	0.05	
OVERALL AVERAGE PROCESSING FACTORS				
Tomato gas ripe (RAC)				
Washed and waxed			**0.78**	
Washed gas ripe			**0.73**	
Washed, pre-canned			**0.83**	
Pre-canned			**0.1**	
Canned			**0.08**	
Canning waste			**12**	
Tomato (RAC)				
Wet pomace			**3.9**	
Dry pomace			**18**	
Tomato paste			**0.3**	
Tomato juice Tomato, canned			**0.05** **0.08**	

Processing olive fruit to oil

Samples for processing to olive oil were obtained from trial CA64 where olive plants were treated with fenpropathrin at approximately 5× the GAP application rate (Samoil, 2007; Reference FR-0561). After harvest, the fruits were pitted and delivered at ambient temperature to the processing facility, where the olives were processed into oil. The samples were cleaned of extraneous materials and then subjected to cold water wash and crushed. After crushing, the olives were pressed by hydraulic press to produce olive oil, which was then subjected to bleaching. Processed olive oil was stored at -20 ºC until analysis. The storage period was recorded as 167 days. Storage stability samples fortified with 0.2 mg/kg fenpropathrin were stored under the same conditions as the field samples and were analysed after 202 days. Recoveries ranged from 85–93% for olive oil.

Residues of fenpropathrin were determined by a method based on RM-22-4. The method was validated using store-bought oil. Recoveries ranged from 94–103%. The LOQ for olive oil was determined to be 0.02 mg/kg.

Table 58 Residues of fenpropathrin in olive oil

Processed Fractions	Residues (mg/kg)	Processing factor	Reference (Trial No.)
Olive fruit (RAC)	14.63		Trial CA64
Olive oil	15.60	1.07	(FR-0561)

Processing cottonseed to oil, meal and soapstock

Samples for processing to cottonseed oil into meal, oil and soapstock were obtained from trials M335-6070, M355-6071 and R194-6427 where cotton plants were treated with fenpropathrin using a range of application rates and timings (Fujie, 1990; Reference FR-01-0360). A Carver impact huller was used to obtain the fractions (kernels and hulls). The kernels were flaked in a Ferrell-Ross "flake-n-roll" to 0.02 mm thickness. The flakes were washed three times with hexane at a temperature of approximately 145 °C. This extraction process took 3 hours. The oil was recovered with a precision laboratory evaporator. During this process the oil reached a maximum temperature of 75 °C. Warm air was forced through the extractor to dissolve the cotton seed flakes. The oil was refined by the following steps:

1. NaOH was added to the oil while it was stirred at 250 rpm at a temperature of 20–24 °C for 15 minutes.
2. The oil was heated to 63–67 °C for 12 minutes and the stirring reduced to 70 RPM.
3. The oil was then allowed to settle for 60 minutes at a temperature of 60–65 °C.
4. The oil was refrigerated overnight or at least for 12 hours.
5. After refrigeration the oil was filtered to obtain the refined oil and soapstock fractions.

The processed samples were stored at -20 ºC until analysis. Recoveries ranged from 78–99% for processed products.

Fenpropathrin residues were determined by gas chromatography using an electron capture detector (Fujie, 1990; Reference FR-01-0306). Recoveries ranged from 94–103%. The LOQ for the method was 0.01 mg/kg. Table 59 summarizes the results of the cottonseed processing study.

Table 59 Residues of fenpropathrin in cottonseed products

Processed Fractions	Residues (mg/kg)					Processing factors					Mean processing factor	Reference (Trial No.)
	M335-6070		M335-6071		R194-6427							
Cottonseed (RAC)	0.02	0.02	0.03	0.03	1.2							(FR-01-0306)
Meal	0.01	0.01	0.01	0.03	0.09	0.5	0.5	0.33	1.0	0.08	0.5	
Crude oil	0.06	0.06	0.07	0.06	2.3	3.0	3.0	2.3	2.0	1.9	2.4	
Refined oil	0.06	0.06	0.10	0.07	2.6	3.0	3.0	3.3	2.3	2.2	2.8	
Soapstock	0.01	0.01	0.04	0.05	1.6	0.5	0.5	1.3	1.7	1.3	1.1	

The processing factors are summarised in table 60.

Table 60 Summary of results of processing

RAC/processed fraction	Residues					Fenpropathrin					
						Processing factors					PF, best estimate
RAC:Whole orange	0.47	0.045	0.17	0.18	0.14	-					
Juice	< 0.01	< 0.01				< 0.02	< 0.22				< 0.02
Oil	37	0.97				78.7	21.56				50.1

RAC/processed fraction	Residues					Fenpropathrin					
						Processing factors					PF, best estimate
Wet peal	0.27	0.035	0.47		0.4	0.6	0.78	2.76		2.86	2.82
Dried peel	0.74	0.12				1.6	2.67				2.1
Dried fines of peel	0.66	0.16				1.4	3.56				2.5
Pulp			< 0.01		< 0.01			0.06		0.07	0.065
RAC: Plum	0.58	0.23									
Dried plum	0.59	0.28				1.0	1.2				2.6
RAC: Tomato	0.07	0.09	0.09	1.05	0.4						
Washed fruit	0.035	0.09	0.06			0.50	1.0	0.67	0.00		0.67
Pre-canned RAC	0.13	0.14	0.13								
Washed pre-canned	0.07	0.12	0.14			0.54	0.86	1.08			0.86
Canned	< 0.01	< 0.01	< 0.01			0.077	0.071	0.077			< 0.075
Canning waste	1.0	0.82	1.25			8	6	10			8
Wet pomace				10.4	3.9				9.9	9.8	9.8
Dry pomace				48	18				46	45.0	45
Tomato paste				0.82	0.3				0.78	0.75	0.77
Tomato juice				0.125	0.05				0.12	0.1	0.12
RAC: Tomato	0.08	0.15									
Washed and waxed	0.055	0.13									0.78
RAC: **olives**	14.63										
Olive oil, bleached	15.6					1.1					1.1
RAC: **Cottonseed**	0.02	0.02	0.03	0.03	1.2						
Meal	0.01	0.01	0.01	0.03	0.09	0.5	0.5	0.33	1	0.075	0.5
Crude oil	0.06	0.06	0.07	0.06	2.3	3	3	2.3	2	1.9	2.3
Refined oil	0.06	0.06	0.10	0.07	2.6	3	3	3.3	2.3	2.2	3
Soapstock	0.01	0.01	0.04	0.05	1.6	0.5	0.5	1.3	1.7	1.3	1.3

RESIDUES IN ANIMAL COMMODITIES

Livestock Feeding Studies

Lactating dairy cattle

Lactating Holstein cows were orally administered technical grade fenpropathrin (purity 92.5%) via gelatin capsules for 28 consecutive days (Fujie, 1986; Reference FR-61-0174). Treatment levels were equivalent to 0, 25, 75 and 250 ppm fenpropathrin based upon the daily average food consumption of 18.54 kg/cow. Each dose was administered in two equal portions at the morning and evening milkings. Each treatment group consisted of four animals, except the control group which consisted of 2 animals. Controls animals were administered empty gelatin capsules only.

Milk samples were collected from individual animals in two portions corresponding to the morning and evening milkings on the days -1, 0, 3, 5, 8, 12, 16, 20, 24 and 28. Milk from days 26 and 27 (250 ppm and control groups) were pooled and processed to obtain pasteurized milk, milk fat solids (cream), milk sugars and protein concentrate. At day 28, three animals from each treatment group were sacrificed and samples of liver, kidney, fat and muscle were collected and frozen for analysis. Administration of fenpropathrin then ceased and the animals received untreated diet. At day 31, the remaining animal from each dose group and one of the control animals were sacrificed and tissues sampled accordingly.

Samples of control milk were fortified with fenpropathrin, stored and analysed to confirm the stability of fenpropathrin over the sample storage period (78 days). An average recovery of 105% indicated stability of fenpropathrin in milk samples. Milk samples in the study were stored from 37 to 73 days at -20 °C prior to analysis.

All samples were analysed for fenpropathrin using method RM-22A-1 within the test period of storage stability. Residues of fenpropathrin in the milk reached a plateau after three days. Average residues in the whole milk of the four cows of each group on Day 3 were 0.04, 0.17, and 0.33 mg/kg for the three dose levels.

Fenpropathrin residues in milk and tissues from animals sacrificed at days 28 and 31 are summarized in Tables 61–63. Residue levels were found to be dose related.

Table 61. Residues of fenpropathrin in milk (mg/L) following administration of fenpropathrin to dairy cows for periods of up to 28 days followed by a 3 day depuration phase

Dose Level (mg/kg)	Day/residue (mg/kg)													
	-1	0	1	3	5	8	12	16	20	24	26	27	28	31	Mean
25	< 0.01	< 0.01	0.02	0.05	0.05	0.04	0.06	0.04	0.04	0.05	na	na	0.05	-	
	< 0.01	< 0.01	0.02	0.03	0.03	0.04	0.03	0.04	0.03	0.04	na	na	0.03	< 0.01	
	< 0.01	< 0.01	0.02	0.04	0.03	0.04	0.03	0.03	0.04	0.03	na	na	0.05	-	
	< 0.01	< 0.01	0.02	0.04	0.04	0.05	0.06	0.04	0.04	0.05	na	na	0.04	-	
Mean	< 0.01	< 0.01	0.02	0.04	0.04	0.04	0.05	0.04	0.04	0.04	na	na	0.04		0.04[a]
75	< 0.01	< 0.01	0.03	0.09	0.09	0.08	0.07	0.08	0.08	0.09	na	na	0.12	-	
	< 0.01	< 0.01	0.07	0.16	0.11	0.13	0.18	0.17	0.22	0.18	na	na	0.16	0.02	
	< 0.01	< 0.01	0.06	0.14	0.10	0.12	0.10	0.07	0.10	0.09	na	na	0.06	-	
	< 0.01	< 0.01	0.13	0.28	0.19	0.18	0.35	0.18	0.17	0.21	na	na	0.16	-	
Mean	< 0.01	< 0.01	0.07	0.17	0.12	0.13	0.18	0.13	0.14	0.14	na	na	0.13	0.02	0.13[a]
250	< 0.01	< 0.01	0.16	0.33	0.30	0.29	0.34	0.22	0.42	0.26	0.30	0.32	0.31	-	
	< 0.01	< 0.01	0.15	0.30	0.28	0.36	0.35	0.32	0.23	0.21	0.28	0.28	0.24	0.04	
	< 0.01	< 0.01	0.17	0.34	0.38	0.31	0.37	0.33	0.36	0.34	0.41	0.39	0.37	-	
	< 0.01	< 0.01	0.14	0.36	0.37	0.40	0.44	0.45	0.34	0.46	0.45	0.34	0.35	-	
Mean	< 0.01	< 0.01	0.16	0.33	0.33	0.34	0.38	0.33	0.34	0.32	0.36	0.33	0.32	0.04	0.32[a]

na –not sampled for analysis; [a] Mean 1 – 28 days

Table 62 Maximum residues of fenpropathrin in tissue samples following administration of fenpropathrin to dairy cows for periods of up to 28 days

Dose level (mg/kg)	Tissue residue (mg/kg)			
	Muscle	Kidney	Liver	Fat
25	0.04	0.05	< 0.01	0.44
75	0.12	0.06	< 0.01	1.7
250	0.33	0.20	0.01	4.1

Table 63 Mean residues of fenpropathrin in tissue samples following administration of fenpropathrin to dairy cows for periods of up to 28 days followed by a 3 day depuration phase

Dose level (mg/kg)	Tissue residue (mg/kg)							
	Muscle		Kidney		Liver		Fat	
	After 28 days	3 day depuration	After 28 days	3 day depuration	After 28 days	3 day depuration	After 28 days	3 day depuration
25	0.02	0.01	0.03	0.01	< 0.01	< 0.01	0.33	0.31
75	0.06	0.10	0.04	0.06	< 0.01	< 0.01	1.0	0.83
250	0.20	0.12	0.16	0.14	0.01	< 0.01	3.8	2.6

Milk from sampling days 26 and 27 of the highest dose group were pooled and processed to obtain pasteurized milk, milk fat solids (cream), milk sugars and protein concentrate. The results show that pasteurization does not significantly reduce fenpropathrin residues in milk. Fenpropathrin concentrates in milk fat by a factor of about 10× (from mean of 0.34 mg/kg in whole milk to 3.7 mg/kg in milk fat). There is no concentration of fenpropathrin in milk sugars and protein concentrate (Table 64).

Table 64 Residues of fenpropathrin in milk fractions

Fraction	Residue (mg/kg)
Whole Milk	0.34[a]
Pasteurized milk	0.25
Milk fat	3.7
Non-fat milk portion	< 0.01
Milk sugar	< 0.01
Protein concentrate	0.02

[a] Mean residue in combined days 26 and 27 milk samples from highest dose.

Laying Hens

Groups of 20 White Leghorn laying hens were fed a diet containing technical grade fenpropathrin (purity 94.5%) at a nominal concentration of 0, 2.5, 7.5 and 25 ppm (measured concentrations: 2.45, 7.10 and 23.6 ppm) for a period of 28 days (Fujie, 1986; Reference FR-61-0175). The number and weight measurements of body weight and feed consumption were taken at regular intervals during the study.

At study termination, all birds were examined macroscopically. Composite samples of liver, gizzard, fat and muscle were prepared for analysis. Eggs from days 1, 2, 4, 7, 21 and 28 were analysed as whole eggs minus shell. Stability samples of treated diet were taken for analysis during the study. To determine the stability of fenpropathrin in stored eggs, control samples were fortified with fenpropathrin, stored and analysed at a time interval greater than the storage interval for eggs from the feeding study. An average recovery of 92% of fenpropathrin was obtained after 156 days of storage at -20 °C. All samples were analysed within 30 days of collection and storage.

Eggs and tissues were analysed for fenpropathrin. The method involved extraction with hexane/acetone, acetonitrile/hexane partitioning, silica gel chromatography and analysis by GC-ECD. Residues were found in the eggs only at the highest feeding level. A level of 0.02 mg/kg was reached on the seventh day and remained essentially constant until the end of the study. Average levels of fenpropathrin in the fat reached 0.02, 0.05 and 0.14 mg/kg for the three feeding levels. Residues in all tissues except fat were below the lower limit of determination, 0.01 mg/kg, at the end of the study. Metabolites could only be detected in liver after dosing with 25 ppm were TMPA (0.05 mg/kg) and PBA-glycin (0.03 mg/kg). The distribution of residues between white and yolk was not studied.

Fenpropathrin residues in eggs and poultry tissues from hens sacrificed at day 28 are summarized in Tables 65 and 66. Residue levels were found to be dose related.

Table 65 Residues of fenpropathrin in eggs (mg/kg) following administration of fenpropathrin to laying hens for a period of 28 days

Dose Level (mg/kg)	Day/Residue (mg/kg)						
	-1	1	4	7	14	21	28
2.5	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Mean	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
7.5	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
Mean	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
25	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	0.02 0.02	0.02 0.01	0.01 0.02	0.02 0.02
Mean	< 0.01	< 0.01	< 0.01	0.02	0.02	0.02	0.02

Table 66 Residues of fenpropathrin in tissue samples following administration of fenpropathrin to laying hens for 28 days

Dose	Tissue residue (mg/kg)			
level	Muscle	Gizzard	Liver	Fat
2.5	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	0.02 0.02 0.02
Mean	< 0.01	< 0.01	< 0.01	0.02
7.5	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	0.06 0.05 0.05
Mean	< 0.01	< 0.01	< 0.01	0.05
25	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	0.16 0.14 0.12
Mean	< 0.01	< 0.01	< 0.01	0.14

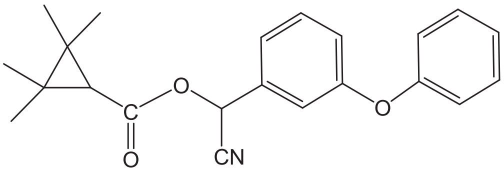
APPRAISAL

Fenpropathrin is a type II pyrethroid insecticide and acaricide used for the control of a variety of arthropods including aphids, worms, moths, beetles, mites, spiders, thrips, flies, fleas and other pests in agriculture.

Fenpropathrin was first evaluated by JMPR in 1993 when an ADI of 0–0.03 mg/kg bw was established and a number of MRLs recommended. In 2006 MRL for tea was recommended. The compound was re-evaluated for toxicology within the periodic review programme in 2012 when the Meeting reaffirmed the ADI of 0–0.03 mg/kg bw and established an ARfD of 0.03 mg/kg bw.

The Forty-fifth Session of the CCPR scheduled fenpropathrin for periodic re-evaluation of residues by the 2014 JMPR. Data to support proposed Codex MRLs on a number of commodities and on animal products were submitted for review.

The structural formulae and IUPAC name of fenpropathrin are:

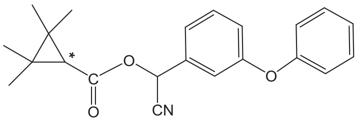


(*RS*)-α-cyano-3-phenoxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate.

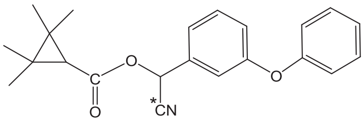
METABOLISM AND ENVIRONMENTAL FATE

The metabolism of fenpropathrin has been investigated in apple, tomato, beans, cotton, cabbage, lactating goat and laying hens. The crops selected represent those for which supervised trials have been provided.

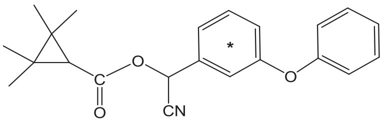
The fate and behaviour of fenpropathrin in plants, animals and soil were investigated using either [cyclopropyl-1-^{14}C]-fenpropathrin, [benzyl-^{14}C]-fenpropathrin, [phenoxyphenyl-^{14}C]-fenpropathrin or [cyano-^{14}C]-fenpropathrin (all with radiochemical purity >98%).



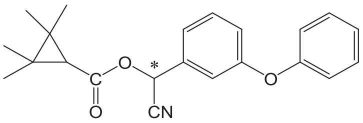
[cyclopropyl-1-^{14}C]-fenpropathrin



[cyano-^{14}C]-fenpropathrin

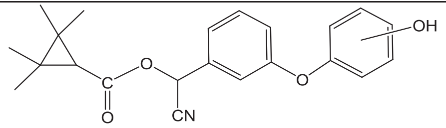
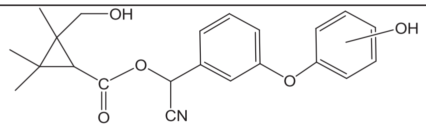
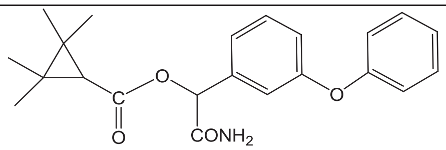
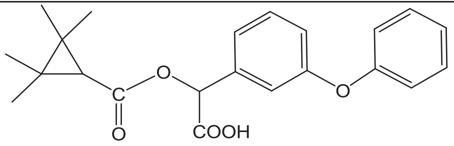
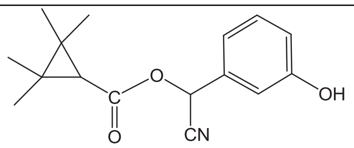
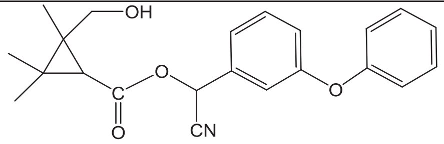
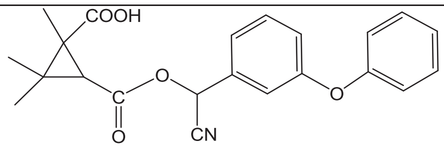
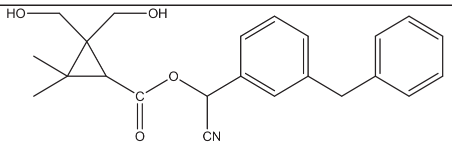
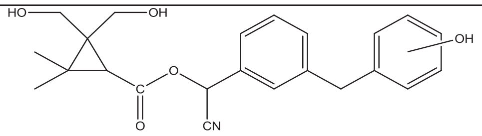
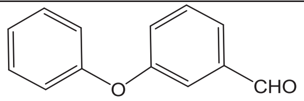
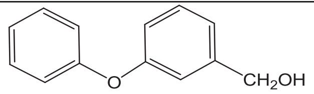
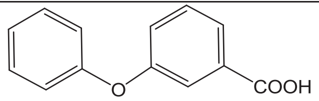


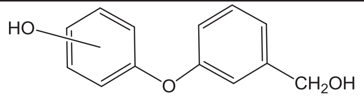
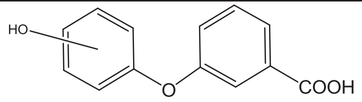
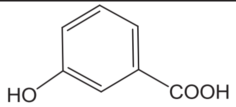
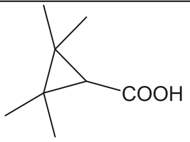
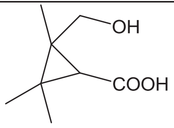
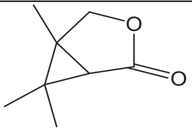
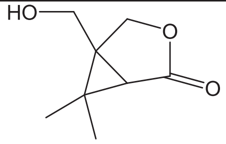
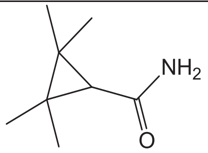
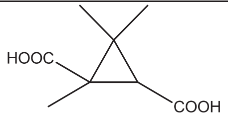
[phenoxyphenyl-^{14}C]-fenpropathrin



[benzyl-^{14}C]-fenpropathrin

The chemical and code names and structure of the major degradation compounds, referred to hereunder, are:

Compound Name	Structure	Found in:
2’-or 4’-OH-Fenpropathrin [α-cyano-3-(2’- or 4’-hydroxyphenoxy)benzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Plant, animal, soil
2’- or 4’-OH-Fenpropathrin-CH_2OH [α-cyano-3-(2’- or 4’-hydroxyphenoxy) benzyl-2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylate]		Plant, animal
$CONH_2$-Fenpropathrin [α-carbamoyl-3-phenoxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Soil, water plant
COOH-Fenpropathrin [α-carboxy-3-phenoxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Soil, plant
Desphenyl-Fenpropathrin [α-cyano-3-hydroxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate]		Animal, soil, plant
Fenpropathrin-CH_2OH [α-cyano-3-phenoxybenzyl 2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylate]		Plant, animal
Fenpropathrin-COOH [α-cyano-3-phenoxybenzyl 2-carboxy-2,3,3-trimethylcyclopropanecarboxylate]		Animal
Fenpropathrin-$(CH_2OH)_2$ [α-cyano-3-phenoxybenzyl 2,2-dihydroxymethyl-3,3-dimethylcyclopropanecarboxylate]		Plant
2’- or 4’-OH-Fenpropathrin-$(CH_2OH)_2$ [α-cyano-3-(2’- or 4’-hydroxyphenoxy)benzyl 2,2-dihydroxymethyl-3,3-dimethylcyclopropanecarboxylate]		Plant
PB aldehyde (PBald) [3-phenoxybenzaldehyde]		Plant, animal, soil
PB alcohol (PBalc) [3-phenoxybenzyl alcohol]		Plant, soil
PBacid [3-phenoxybenzoic acid]		Plant, animal, soil, water

Compound Name	Structure	Found in:
2’- or 4’-OH-PBalc [3-(2’- or 4’-hydroxyphenoxy)benzyl alcohol]		Plant
2’- or 4’-OH-PBacid [3-(2’- or 4’-hydroxyphenoxy)benzoic acid]		Plant, animal, soil
3-OH-Bacid [3-hydroxy-benzoic acid]		Animal, soil
TMPA [2,2,3,3-tetramethylcyclopropane-carboxylic acid]		Plant, animal, soil, water
TMPA-CH_2OH [2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylic acid]		Plant, animal
TMPA-lactone [5,6,6-trimethyl-3-oxabicyclohexan-2-one]		Plant
TMPA-CH_2OH lactone [5-hydroxymethyl-6,6-dimethyl-3-oxabicyclohexan-2-one]		Plant, animal
TMPA carboxamide [2,2,3,3-tetramethylcyclopropane-carboxamide]		Water
TMPA-COOH [2-carboxy-2,3,3-trimethylcyclopropanecarboxylic acid]		Plant, animal

Animal metabolism

Laboratory animals

The toxicological evaluation fenpropathrin was carried out by the 2012 JMPR. Absorption by rats was rapid and excretion was almost complete (97%) within 48 hours. About 56% of the administered dose was found in urine and 40% in faeces after 48 hours. The amount of radioactivity excreted via expired air was 0.005%. The low residues found in blood, liver, kidney, fat, muscle and brain 24 hours after dosing depleted rapidly over the following 7 days to barely detectable levels, and less than 1.5% of the administered dose remained in the body 8 days after treatment. The highest residue was found in the fat. About 29–53% of the parent compound was detected in the faeces and no parent compound was detected in the urine. The predominant urinary metabolites derived from the acid moiety were identified as TMPA–glucuronide and TMPA-CH_2OH (*trans*). Other metabolites identified were

TMPA-COOH (*trans*) and TMPA-CH_2OH-lactone in free form or as the glucuronide. The major urinary metabolites derived from the alcohol moiety were PBacid in free form and as the glycine conjugate, 4′-OH-PBacid–sulfate and 2′-OH-PBacid–sulfate. The urinary metabolites from the alcohol moiety were similar to those from other pyrethroids. The major faecal metabolite was identified as CH_2OH *trans*-fenpropathrin, followed by COOH *trans*-fenpropathrin, 4′-OH-fenpropathrin and 4′-OH,CH_2OH *trans*-fenpropathrin. Fenpropathrin and TMPA were the major components of ^{14}C in tissues. No sex-related differences in tissue distribution were observed.

Lactating goats

Two lactating goats per group were dosed for five consecutive days via capsules with either [phenoxyphenyl-^{14}C]-fenpropathrin or [cyclopropyl-1-^{14}C]-fenpropathrin at a rate equivalent to 50 ppm. Milk samples were collected at the morning and afternoon and urine and faeces once a day.

The mean average total radioactivity recovered following dosing with both labelled compounds was about 65.8% with 40% recovered in the urine and 25% in the faeces.

Excretion via milk was a minor route with radioactivity accounting for approximately 0.15% of the applied phenoxyphenyl labelled and approximately 0.087% for the cyclopropyl labelled compound. Total radioactive residues in the milk reached a steady state by the evening milking on the third day.

Following the treatments with 50 ppm [phenoxyphenyl-^{14}C]-fenpropathrin, the maximum total residue in milk was 0.25 mg/L. The major residue components were the parent fenpropathrin 78% TRR, (0.02 mg/kg). At around the plateau, the average concentration of the parent compound was 0.05 mg/kg (29%TRR) and PBacid-glycine 0.076 mg/kg (46%TRR). The other metabolites were < 10% TRR.

At sacrifice, the average residues were: in fat: fenpropathrin (0.50 mg/kg, 78%TRR), all metabolites were present at lower than 5%TRR; in muscle: fenpropathrin (0.011 mg/kg, 45%TRR), PBacid-glycine (22.4% TRR), PBacid (10.9% TRR) the other metabolites were below 3%; in liver: fenpropathrin (0.014 mg/kg, 3.2% TRR), PBacid-glycine (20%TRR), PBacid (14% TRR), 4'-OH-PBacid (11%TRR), the other metabolites were below 10% TRR; in kidney: fenpropathrin (0.01 mg/kg, 1.24%TRR), PBacid-glycine (39%TRR), PBacid (38%TRR) and the other metabolites were below 10%.

After the goats were administered with [cyclopropyl-1-^{14}C]-fenpropathrin, the average total residue in milk was 0.11 mg/L. The parent compound in milk amounted to maximum 70% TRR (0.086 mg/kg). All metabolites amounted to maximum 4% TRR.

At sacrifice, the average residues were: in fat: fenpropathrin (0.55 mg/kg, 81% TRR), all metabolites were present at lower than 3%TRR; in muscle: fenpropathrin (0.005 mg/kg, 11.3% TRR), TMPA-CH_2OH-lactone (19.4% TRR) andTMPA-CH_2OH (11%TRR); in liver: fenpropathrin

0.011 mg/kg (2.45% TRR), TMPA (18.2%TRR), TMPA-CH_2OH (15.8%TRR) and TMPA-CH_2OH lactone (12.5% TRR); in kidney: fenpropathrin 0.0076 mg/kg (1.48% TRR), TMPA-CH_2OH-lactone (40.8% TRR) and TMPA-CH_2OH (14.5% TRR) .

The other metabolites in muscle, liver and kidney were below 5%TRR.

Laying hens

Fenpropathrin, labelled in either the cyclopropyl or the phenoxyphenyl ring was administered in capsules to laying hens daily for 10 days at a nominal rate of either 0.5 or 5 mg/kg body weight.

The recovery of total radioactivity from excreta, eggs and tissues was between 75 and 84% of the total applied dose. Between 98.9 and 99.6% of the recovered activity was found in the faeces irrespective of the label.

Approximately 0.05% of the applied phenoxyphenyl- and 0.2% of the cyclopropyl-labelled compound was found in the eggs. At about the 6th or 7th day of the study residue levels in the eggs reached a plateau of about 0.05 and 0.2 mg/kg fenpropathrin equivalent for the two doses of the phenoxyphenyl-labelled and about 0.2 and 0.5 mg/kg for those of the cyclopropyl-labelled compounds.

In case of high dose group treated with phenoxyphenyl-labelled compound, the average concentration of parent fenpropathrin amounted to 31% of TRR (0.043 mg/kg) in eggs and all identified metabolites were present at less than 10%TRR. In breast and tight muscle, the average proportions of residues (%TRR) were: parent compound 19% (0.02 mg/kg), PBacid (22%), 3-OH-BAcid (13%). In liver and kidney the average percentage distributions of TRR were, respectively: parent (0.98%, 2.11%; 0.014 mg/kg, 0.096 mg/kg), 3-OH-BAcid (29%, 35%), 4-OH-PBacid (16%, 26%) and PBacid (14.7%, 8.7%). The other metabolites were <10% TRR in all tissues.

In case of cyclopropyl label the average concentration of parent fenpropathrin amounted to 9.8% TRR (0.038 mg/kg) in eggs, and one major metabolite, TMPA-CH_2OH, was present at 11.7%n TRR. All other metabolites amounted to <10% TRR and their concentrations were < 0.03 mg/kg. In muscle, the average proportions of residues (%TRR) were: parent compound 6% (0.033 mg/kg), TMPA-CH_2OH (16%), TMPA (15.7%), TMPA-CH_2-OH-lactone (12.3%. All other metabolites amounted to <10% TRR and their concentrations were < 0.03 mg/kg. In liver and kidney the average percentage distributions of TRR were, respectively: parent (1.2%, 0.04 mg/kg; 5.1%; 0.24 mg/kg), TMPA (26.4%, 47.5%), TMPA-CH_2OH (14.7%, 7.7%), TMPA-CH_2-OH-lactone (14.8%, 5.3%), TMPA-COOH (11.4%, 15.3%). The other metabolites were present at < 1.8%TRR.

The metabolic pattern in fat was similar in case of both labels. The fat contained maximum 0.90 mg/kg total residue of which the parent compound amounted to 64%TRR (0.58 mg/kg). The most prominent metabolite was TMPA (14%nTRR), the other metabolites occurred at < 6% TRR.

In summary, the major biotransformation reactions of fenpropathrin in animals consisted of oxidation at the methyl groups of the acid moiety and at the 2'- or 4'-positions of the alcohol moiety, cleavage of the ester and ether linkages and conjugation of the resultant carboxylic acids and alcohols.

The parent compound was detected in milk, eggs and tissues, and it was the main residue in fat about 80%TRR. The major metabolites >10% TRR following the treatment with phenoxyphenyl-labelled fenpropathrin were PBacid-glycine, PBacid and 3-OH-BAcid, and after dosing with cyclopropyl-labelled compound the major metabolites were TMPA-CH_2OH, TMPA, TMPA-CH_2-OH-lactone and TMPA-COOH.

Plant metabolism

Apples

One apple tree was treated 3 times with [cyclopropyl-1-^{14}C]-fenpropathrin and [benzyl-^{14}C]-fenpropathrin at a rate equivalent to 0.45 kg ai/ha. Samples were collected 14 days after the final application. Un-extractable residues ranged from 3% (both labels in fruit) to 8% (benzyl label in leaves).

Practically, the entire residue found in the fruit (92-94% TRR) was present as the parent compound. The parent compound was also the major component in the rest of the plant (61–66%TRR). All metabolites were < 5%TRR.

Tomatoes

Greenhouse-grown tomato plants were treated four times, 7–8 days apart, with [cyclopropyl-1-^{14}C]-fenpropathrin and [benzyl-^{14}C]-fenpropathrin at rates equivalent to 0.224 kg ai/ha. Fruit and leaves were extracted at harvest 19 DALT).

The total radioactive residues were 0.1 mg/kg and 0.4 mg/kg in fruits after treatments with benzyl and cyclopropyl labelled compound and consisted of the parent compound in about 66% of the benzyl label and 28% of conjugated metabolites. Their proportion was about the opposite for cyclopropyl label. The non-extractable residues were between 5.5% and 6.7%. Because of their low level, the radioactivity could not be fully characterised.

In tomato plants, the parent compound was present in 36–39% of TRR (0.1-0.04 mg/kg). Of the identified metabolites only fenpropathrin-$(CH_2OH)_2$ was present in free form (2.7–3.1% TRR). Numerous other metabolites were in conjugated forms. Non-extractable residues amounted to 7.4–9.3%.

In another study where tomato plants were treated in greenhouse four times with [cyclopropyl1-^{14}C]-fenpropathrin and[phenoxyphenyl-^{14}C]-fenpropathrin at a rate equivalent to 0.224 kg ai/ha. Fruits and plant materials, sampled 3 days after last application, contained the parent compound in 96–98% of TRR. Polar metabolites amounted to 1.3% of TRR. The surface rinses contained 98–99% of the parent compound determined in the fruits.

Beans

Pinto bean plants grown in greenhouse were treated three times with [cyclopropyl-1-^{14}C]-fenpropathrin and [benzyl-^{14}C]-fenpropathrin at a rate equivalent to 0.224 kg ai/ha. Samples were collected 15 days after the final application. Leaves and plant parts contained 98.8%, bean pods and seeds contained 1.1% and 0.1% of the residue. In beans treated with benzyl- and cyclopropyl-labelled compound, the residue in seed was composed of the parent compound (4.1% and 0.1% of TRR), conjugated metabolites (61–51% TRR) free metabolites (17–4%TRR) and un-extractable residues 18.2%–45% TRR. The bean leaves contained 46.7% parent compound and conjugates of PBald (19.5%TRR) after treatment with benzyl labelled compound. After treatment with cyclopropyl-labelled compound the residue composed of 46.4% parent fenpropathrin and conjugates of TMPA-CH_2OH (16.7%TRR). The other metabolites were present at < 10% TRR.

Cotton

Two studies were conducted treating cotton plants with [phenoxyphenyl-^{14}C]-fenpropathrin and [cyclopropyl-1-^{14}C]-fenpropathrin.

In the first study the plants were treated in greenhouse four times with syringe applying a total of ca. 4.7–4.8 mg ^{14}C-fenpropathrin. In the leaves at harvest 66 and 111 days after treatments with phenoxyphenyl- or cyclopropyl labelled compound the total remaining radio activity included 70% TRR and 55% TRR parent compound, respectively Most of the remaining radio activity was tentatively accounted for PBacid (2.% TRR) and trans-TMPA-COOH (11%TRR) mainly in conjugated forms. All other metabolites were below 2%TRR.

Plants grown on soils treated with 0.5 kg/ha of fenpropathrin contained ^{14}C residues in very low concentration (0.002 mg/kg in leaves and 0.01 mg/kg in bolls), demonstrating limited tendency for translocation.

In the second study outdoor cotton plants were treated four times at a rate equivalent to 0.336 kg ai/ha. Seeds collected 21 day after last treatment contained total radioactivity 1.14 mg/kg and 1.59 mg/kg, while the foliage contained 78.6 mg/kg and 67.7 mg/kg fenpropathrin equivalent after treatment with phenoxyphenyl- and cyclopropyl-labelled fenpropathrin, respectively. The seed, lint and foliage contained the parent fenpropathrin in 93.8%, 96.2% and 69.2% TRR, respectively, after treatment with phenoxyphenyl -labelled compound. The seed contained 12 metabolites each at < 0.005 mg/kg concentration. Following the application of cyclopropyl-labelled fenpropathrin the parent compound amounted to 85.6%, 100% and 67.4% TRR in seed, lint and foliage, respectively. A small number of metabolites were also detected, but not identified.

Cabbage

Cabbage plants were treated on the 3rd-4th leaves with [cyano-^{14}C]-fenpropathrin), [cyclopropyl-^{14}C]-fenpropathrin and [phenoxyphenyl-^{14}C]-fenpropathrin at a rate equivalent to about 0.09 kg ai/ha. The cabbages were sampled immediately after application and at 3, 7, 14, 21, 28, 35 and 42 days after application. The proportions of parent fenpropathrin in 28-day samples after treatment with cyano-, cyclopropyl- and phenoxphenyl-fenpropathrin were 16.9%, 15.8% and 12.9% of the applied dose, respectively, and it was present at somewhat lower proportion in 48-day samples. The major part of the residue (23-26% AD) composed of the conjugates of 2'-OH-fenpropathrin-CH_2OH, 4'-OH-fenpropathrin-CH_2OH, 2'-OH-fenpropathrin-$(CH_2OH)_2$ and 4'-OH-fenpropathrin-$(CH_2OH)_2$ and TMPA-CH_2OH-lactone-conjugate (11%). The other metabolites were present at $\leq$ 10% TRR after treatments with cyclopropyl- and phenoxyphenyl-labelled compounds.

Most of the recovered radiocarbon was in the treated leaves and less than 1.2% of the applied radiocarbon was found in the untreated shoots indicating that fenpropathrin and its metabolites hardly translocate from the application site to other parts of the plant.

Fate of hydrogen cyanide (HCN) and TMPA in abscised leaves

The fate of HCN and TMPA in abscised leaves of apple, kidney bean, cabbage, mandarin orange, tomato and vine was studied. Two abscised leaves from each plant were placed in 100 mL distilled water containing ^{14}C-TMPA at a concentration of 1.0 ppm. After cultivation for five days the leaves were extracted with methanol:chloroform:water (4:2:1).

TMPA was readily converted in plants to more polar products. The metabolic pathways for TMPA varied dependent upon species of plant. The glucose ester was a main product in apple and vine leaves. In orange, cabbages and bean leaves, the malonylglucoside was mainly formed.

Further on, two abscised cabbage leaves were treated for four hours with distilled water containing K^{14}CN and then transferred to K^{14}CN-free distilled water. The study demonstrated that if hydrogen cyanide were liberated during the hydrolysis of fenpropathrin, it would be rapidly converted to natural products.

Summary of plant metabolism

Metabolism of fenpropathrin has been studied in apples, tomatoes, beans, cotton and cabbage.

The general pattern of degradation in all the plant studies include break of the ester linkage to produce 3-phenoxybenzoic acid (PBacid) and the corresponding alcohol (PBalc) and aldehyde (PBald). From the acid side of the molecule, the main metabolite is TMPA which can give rise to TMPA-CH_2OH and TMPA-CH_2OH lactone. PBacid can be hydroxylated at various positions on the phenoxy ring to produce, 2'-or 4'-OH-PBacid.

The majority of radioactivity was found in leaf samples. Low levels of radioactivity were found in fruit/beans. The parent fenpropathrin amounted to the major part of the residue. Fenpropathrin and its metabolites hardly translocate from the application site to other parts of the plant.

Environmental fate

In soil

Studies on the metabolism of fenpropathrin in aerobic soil carried out with [phenoxyphenyl-^{14}C]-fenpropathrin demonstrated that fenpropathrin is degraded in the soil by a combination of photochemical and microbial processes. After 365 days, 18.4% of the dose remained as parent with accumulated volatiles accounting for 59.9% (99.8% of which was CO_2) and un-extractable residues for 17.8%. Metabolism proceeds via cleavage of the ester bonds, hydroxylation, and hydrolysis of the cyano group to $CONH_2$ and COOH groups. Metabolites included desphenyl-fenpropathrin, 4’-OH-fenpropathrin, phenoxybenzoic acid, and $CONH_2$-fenpropathrin, which was further degraded to COOH-fenpropathrin. The estimated half-life was about 4 weeks in moist soil (70–75% field capacity) and 16 weeks in a dryer soil with 16% water content.

Photodegradation studies were carried out with fenpropathrin labelled with ^{14}C in the cyano group, the phenoxyphenyl ring or C-1 position of the cyclopropyl ring. Irradiation greatly enhanced degradation of the fenpropathrin. The main degradation product under irradiation with all three labels was $CONH_2$-fenpropathrin which reached a maximum in the three soils after 5–7 days during the 14-day exposition.

Fenpropathrin is moderately stable in soil under aerobic condition. The photolysis increased the degradation of the surface residues.

Hydrolytic degradation

Fenpropathrin is stable to hydrolysis in water at pH 5 and pH 7 but it is hydrolysed at a moderate rate at pH 9.

Rotational crops

No study was submitted on rotational crops.

Methods of analysis

Analytical methods have been developed for determination of residues of fenpropathrin in plant and animal matrices. In general, the methods involve solvent extraction, clean-up by either silica gel or Florisil column, GLC using electron capture detection. Additional purification using gel permeation chromatography (GPC) was performed for oily matrices. The main variations depending on the substrates are on extraction and clean-up procedures. Fruits and vegetables may be homogenized with water, shaken with acetone, and extracted with dichloromethane, using NaCl to minimize emulsification. After drying with anhydrous sodium sulphate and clean-up by silica gel column chromatography, the solvent is evaporated at < 40 ºC and the residue dissolved in acetone before determination by gas chromatography with electron capture detection. Other extraction procedures involve direct extraction of the homogenized material suspension in water or homogenization with methanol instead of water. The methods were generally validated at 0.01 mg/kg LOQ level. The RSD of recoveries was < 20%

A multi residue method (DFG S19) was validated for the determination of fenpropathrin in plant materials of high water content and acidic plant matrices applying GC-MS detection (m/z 181 and 265 for quantification and 125, 152, 209 and 349 for confirmation).

In a supervised trial on soya bean, the residues were determined with LC-MS/MS utilising the transition of m/z 350→125. The LOQ was 0.01mg/kg.

Stability of residues in stored analytical samples

The stability of fenpropathrin residues in commodities under frozen conditions has been investigated in apples, orange, cotton, pears, grapes, tomato and its processed products as well as in products of animal origin.

Fenpropathrin was shown to be stable at least for the indicated periods (month) in: apple, orange, cotton, pears and grapes (12);cucumber (8); grape juice, dry pomace (14) wet pomace (12), hydrated raisins and raisin waste (11); melons (6); non-bell peppers (10); olives, olive oil (~7); orange oil and orange dried peel (11); raspberries (7); squash (7.5); strawberries (6); tomato (6), tomato paste (5), tomato juice (5) and wet and dry tomato pomace (5) tomato waste (5).

The residues were stable in eggs for 5 months, and milk and kidney at least for 2.5 months.

Definition of the residue

Livestock animal metabolism studies were conducted on lactating goats (50 ppm in feed) and laying hens (0.5 and 5 mg/kg body weight) applying [phenoxyphenyl-^{14}C]- and [cyclopropyl-1-^{14}C]-fenpropathrin.

In milk, at around the plateau at 3–5 days, the parent compound amounted to the major part of residues 28% TRR, (0.02 mg/kg) and 66% TRR, (0.086 mg/kg), respectively. The major metabolites were PBacid-glycine (46% TRR) from phenoxy label and all other metabolites were below 3%TRR from cyclohexyl label.

Following the treatments with 50 ppm [phenoxyphenyl-^{14}C]-fenpropathrin the average residues of the parent fenpropathrin amounted to 0.50 mg/kg (78%TRR) in fat, 0.011 mg/kg (45% TRR) in muscle, 0.14 mg/kg (3.2% TRR) in liver and 0.01 mg/kg (1.2% TRR) in kidney. The major metabolites in these tissues were PBacid-glycine (20–39% TRR), PBacid (11–38% TRR) and 4’-OH-PBacid (11% TRR). The other metabolites were present at lower than 10% TRR.

After the goats were administered with [cyclopropyl-1-^{14}C]-fenpropathrin, the average residues comprised of the parent compound 0.55 mg/kg (81% TRR) in fat, 0.005 mg/kg (11% TRR) in muscle, 0.11 mg/kg (2.5% TRR) in liver and 0.0076 mg/kg (1.5% TRR) in kidney. The major metabolites in these tissues were TMPA-CH_2OH-lactone (19–41% TRR), TMPA (18%TRR), TMPA-CH_2OH (11–16%TRR). The other metabolites were below 5%TRR.

In hens, the parent fenpropathrin was a major residue 0.043 mg/kg (31%TRR) and 0.038 mg/kg (9.7%TRR) in eggs following treatments with benzyl- and cyclopropyl-labelled compounds. The only metabolite exceeding 10% TRR was TMPA-CH_2-OH from cyclopropyl label. Following dosing with benzyl label, the average concentration of parent compound was 0.43 mg/kg (29%TRR) in fat, 0.029 mg/kg (1.6% TRR) in muscle, 0.096mg/kg (7%TRR) in kidneys and 0.014 mg/kg (1% TRR) in liver. After dosing with cyclopropyl label, the average concentration of parent compound was 0.033 mg/kg, (1.55%TRR) in muscle, 0.21 mg/kg (10% TRR) in kidneys, 0.036 mg/kg (2% TRR) in liver. The main metabolites from hens dosed with the benzyl-labelled compound were 3-OH-Bacid (4–29%TRR) or 4’-OH-PBacid (4–16% TRR and PBacid 3.2–24% TRR). While from the cyclopropyl-labelled group the TMPA (6–26% TRR) and its CH_2-OH, COOH, CH_2-OH-lactone derivatives (9–41%TRR) were the major metabolites. Several of these metabolites are also formed from other pyrethroid insecticides.

The parent fenpropathrin was the major residue in milk, meat and eggs and it was detected at low concentrations in liver and kidney. The polar metabolites listed above and the minor ones identified are of no toxicological significance.

The Meeting concluded that the parent fenpropathrin is a suitable marker for animal commodities for both enforcement and dietary risk assessment.

As the fenpropathrin residue concentrates in the fat, based on the distribution of residues in various tissues, supported by the logP_{ow} of 6.0 for fenpropathrin, the Meeting concluded that the fenpropathrin residue is fat soluble.

The fate of fenpropathrin residues was studied in apples, beans, cabbages, cotton and tomatoes. The parent fenpropathrin is the major residue in apple fruits (92–94% TRR), tomato fruits (30–66% TRR), in bean leaves (46% TRR), bean seeds (up to 4.1% TRR), in cabbage leaf extract (up to 16% TRR) and cotton seed (up to 94% TRR). The major metabolites were the conjugates of 2’-OH-fenpropathrin-$(CH_2OH)_2$ and of 4’-OH-fenpropathrin-$(CH_2OH)_2$ (max 19–22% TRR in cabbage), all other metabolites (1.3–9.8%TRR) were < 10% TRR in case of all labelled compounds.

The nature of metabolites is similar to that goats and hens. The polar metabolites listed above are of no toxicological significance.

The Meeting concluded that the parent fenpropathrin is suitable marker for plant commodities for both enforcement and dietary risk assessment.

Validated analytical methods, suitable for enforcement, are available for detecting fenpropathrin in various matrices.

The Meeting agreed in the following residue definition:

Definition of residue for compliance with MRL and for estimation of dietary intake for animal and plant commodities is the parent fenpropathrin.

The residue is fat soluble.

Results of supervised residue trials on crops

The Meeting received supervised trial data on citrus fruits, pome fruits, stone fruits, berries and small fruits, fruiting vegetable, cucurbits, olives, soya beans, cotton, tree nuts, coffee beans and tea.

The HR values were estimated based on the residues measured in single samples.

Citrus fruits

A total of 31 supervised trials were conducted in the United States on citrus fruits (18 on oranges, 6 on lemons and 7 on grapefruit). The current maximum GAP for citrus in the USA consists of two applications at 0.45 kg ai/ha with a total seasonal rate of 0.90 kg ai/ha and a PHI of 1 day.

The average residues in duplicate composite samples derived from treatments corresponding to US GAP were: in oranges: 0.05, 0.07, 0.12, 0.18, 0.25, 0.26, 0.27, 0.33, 0.33, 0.46, 0.53, 0.96, 1.2 mg/kg; in lemons: 0.51, 0.56, 1.2 mg/kg; in grapefruits: 0.12, 0.18, 0.2, 0.34, 0.34, 0.37, 0.47 mg/kg.

The Meeting noted that the GAP in USA is for citrus fruits, that the residue populations were not significantly different (Kruskal-Wallis H-test) and the median residues were within the 5 times range. Furthermore, the Meeting concluded that the residue date sets for lemons, oranges and grapefruits are suitable for estimation of residue levels for the citrus group. The meeting agreed to combine the datasets which in rank order were: 0.05, 0.07, 0.12, 0.12, 0.18, 0.18, 0.2, 0.25, 0.26, 0.27, 0.33, 0.33, 0.34, 0.34, 0.37, 0.46, 0.47, 0.51, 0.53, 0.56, 0.96, 1.2, 1.2 mg/kg.

The Meeting estimated a maximum residue value of 2mg/kg and, based on the processing factor of 0.065, HR of 0.098 mg/kg and STMR values 0.02 mg/kg for citrus fruit group.

Pome fruits

Forty-seven supervised trials on pome fruit (27 in apples and 20 in pears) were conducted in the USA in 1984-1987, using a higher number of applications and total seasonal rates compared to the GAP in the USA (up to 0.9 kg ai/ha and a PHI of 14 days).

Four trials in apple and pear complied with current US GAP. The residues were: in apple: 0,48, 0.58, 0.88 and 1.1 mg/kg; and in pear:0.27, 0.3, 1.2 and 1.8 mg/kg.

The Meeting noted that the GAP in USA is for pome fruit, that the residue populations were not significantly different and that the median residues were within the 5 times range.

The Meeting concluded that the residues in apples and pears could be combined: 0.27,0.3, 0,48, <u>0.58</u>, <u>0.88</u>, 1.1, 1.2 and 1.8 mg/kg.

The Meeting noted that in one of the pear samples the residue was 2 mg/kg, and estimated a maximum residue level of 3 mg/kg, HR of 2 mg/kg and STMR of 0.73 mg/kg

The Meeting withdraws its previous recommendation for maximum residue levels of 5 mg/kg

The meeting noted that the short-term intakes of apples and pears for children are 390% and 280% of ARfD, respectively.

There is no alternative GAP available to be considered.

Stone fruits

Supervised trials were carried out in USA on peaches (10), cherries (6) and plums (7) according to US GAP (2 × 0.45 kg ai/ha, 3 days PHI).

The average residues in duplicate composite samples derived from treatments corresponding to US GAP were: in peach: 0.44, 0.58, 0.65, 0.66, 0.70, 0.71, 0.73, 0.92, 1.0, 1.0 mg/kg; in plums; 0.18, 0.22, 0.23, 0.25, 0.32, 0.35, 0.67 mg/kg; and in cherries: 1.4, 1.5, 1.8, 1.9, 3.3, 3.4 mg/kg.

Since the residue populations are not similar, residue levels were estimated separately for each commodity.

The meeting estimated maximum residue, HR and STMR values for subgroups of: peaches 3 mg/kg, 1.1 mg/kg and 0.71 mg/kg; plums 1 mg/kg, 0.71 mg/kg and 0.25 mg/kg; and cherries 7 mg/kg, 3.53, and 1.85 mg/kg, respectively.

The meeting noted that the short-term intakes of peaches and cherries are 190% and 140% of ARfD for children, respectively.

There is no alternative GAP to be considered.

Berries and other small fruits

Strawberry

Eleven out of 12 trials conducted on strawberry in USA matched the US GAP (applications at up to 0.45 kg ai/ha for a total of 0.9 kg ai/ha per season and a PHI of 2 days).

The average residues in duplicate composite samples derived from treatments corresponding to US GAP were: 0.26, 0.38, 0.39, 0.48, <u>0.48, 0.55</u>, 0.63, 0.65, 0.69, and 1.2 mg/kg.

The Meeting estimated maximum residue level, HR and STMR values of 2 mg/kg, 1.2 mg/kg and 0.515 mg/kg, respectively.

Raspberry

Seven supervised trials on raspberries were conducted in the USA in 2005 with higher rate and shorter PHI (total 0.9 kg/ha with 2 days PHI) than the current GAP for caneberries (applications at up to 0.34 kg ai/ha for a total of 0.67 kg ai/ha per season and a PHI of 3 days).

As no trial matched the GAP, recommendation cannot be made.

Grape

Twenty five supervised trials were conducted on grapes in the USA during 1983–2001 (2 × maximum rate of 0.45 kg ai/ha, the maximum seasonal rate of 0.9 kg/ha with 21 days PHI).

The trial data did not match the critical GAP of the USA. As a result no recommendations could be made.

The Meeting withdraws its previous recommendation of 5 mg/kg.

Assorted tropical and subtropical fruits – Edible peel

Olives

Three supervised trials were conducted on olives in the USA during 2005 matching US GAP (3 × 0.34 kg ai/ha with total seasonal application rates of about 0.9 kg ai/ha and 7 days PHI).

The average residues in pitted olives from two composite samples derived from treatments corresponding to maximum application rates were in rank order: 1.9, <u>2.2,</u> and 3.6 mg/kg.

Three residue values were not considered sufficient for the estimation of maximum residue levels in olives.

Fruiting Vegetables, Cucurbits

Cucumber

Six supervised trials on cucumber were conducted in the USA in 1994 and 1996, following the GAP in the USA for cucurbit vegetables (applications at the rate of up to 0.34 kg ai/ha at 7 days intervals for a total of 0.9 kg ai/ha/season; PHI is 7 days).

The Meeting noted that the trials were not conducted at maximum GAP. For multiple treatments proportionality could be applied. As a result no recommendations could be made.

Melon

Ten supervised trials on cantaloupe were conducted in the USA in 1994 following the GAP in the USA for cucurbits (applications at the rate of up to 0.34 kg ai/ha for a total of 0.9 kg ai/ha/season; PHI is 7 days).

The Meeting noted that the trials were not conducted at maximum GAP. As the number of applications and or the applied dosage rate differed from maximum GAP, the proportionality could not be applied. As a result no recommendation could be made.

Summer squash

Seven supervised trials on summer squash were conducted in the US in 1994 and 1996, following the GAP in the US for cucurbits (applications at the rate of up to 0.34 kg ai/ha for a total of 0.9 kg ai/ha/season; PHI is 7 days)

The Meeting noted that the trials were not conducted at maximum GAP. As the number of applications and or the applied dosage rate differed from maximum GAP, the proportionality could not be applied. As a result, no recommendation could be made.

Fruiting vegetables other than Cucurbits

Tomato

Nine supervised trials conducted on tomatoes in the USA in 1993 matching the US GAP for fruiting vegetables other than cucurbits (applications at the rate of 0.22-kg ai/ha at 7 days intervals but not more often than 7 days, PHI is 3 days) were received.

The average residues in two composite samples derived from treatments corresponding to maximum application rates, in ranked order, were: 0.05, 0.08, 0.11, 0.18, <u>0.19</u>, 0.21, 0.27, 0.30, and 0.55 mg/kg.

Peppers

Ten supervised trials on peppers (6 on bell and 4 on non-bell) were conducted in the USA in 1996 and 1998. The application rates corresponded to US GAP (0.22 kg ai/ha up to 0.9 kg ai/ha, 3 days PHI), but samples were taken at 2 and 4 days instead of the 3 day PHI.

The average residues in two composite samples were: in Bell pepper: 0.10, 0.34, 0.37, 0.37, 0.50, 0.67 mg/kg; and Chili pepper: 0.24, 0.31, 0.38, 0.40 mg/kg

The Meeting noted that the residues obtained 2–4 days after last application were in a relatively narrow residue range (2×median) leading to lower maximum residue estimate than would be generally expected. Therefore the residue values obtained at day 2 (-33% of PHI) were considered acceptable. (Only one residue data (0.50 mg/kg) was obtained at day 3 and one at day 4 (0.10 mg/kg).

The Mann-Whitney U-test confirmed that the above data could be combined for the estimation of the maximum residue level and STMR. The ranked order of residues from supervised trials on peppers were: 0.10, 0.24, 0.31, 0.34, 0.37, 0.37, 0.38, 0.40, 0.50, and 0.67 mg/kg.

The Meeting noted that the GAP in USA is for fruiting vegetables, other than cucurbits and the residue populations were not significantly different (Kruskal-Wallis H-test) and the median residues were within 5 times range. However, the short-term intake for eggplants would exceed the ARfD by 110% for adults. Consequently, a recommendation for the fruiting vegetables crop group could not be made.

No alternative GAP was available for fruiting vegetables other than cucurbits.

The Meeting therefore agreed to estimate residue levels for individual commodities:

Tomato: maximum residue level of 1 mg/kg, HR of 0.64 mg/kg, STMR of 0.19 mg/kg.

Peppers including chili pepper: maximum residue level of 1 mg/kg, HR of 0.70 mg/kg, STMR of 0.37 mg/kg.

Chili peppers, dried (based on concentration factor of 7): maximum residue level of 7 mg/kg, HR of 4.9 mg/kg mg/kg, STMR of 2.59 mg/kg.

The meeting confirms its previous recommendation for maximum residue level of 1 mg/kg for tomatoes.

Pulses

Soya beans

Eight supervised trials on soya beans were conducted in Brazil in 2010 and 2013, following the GAP in Brazil (one applications at the rate of 0.045 kg ai/ha and a PHI of 30 days). Residues in all samples were below the limit of quantification (< 0.01 mg/kg).

The Meeting estimated a maximum residue and STMR values of 0.01 mg/kg.

Cotton

Thirty-two supervised trials on cotton were conducted in the USA in 1983–1989 with application rates up to 0.9 kg ai/ha and a PHI of 21 days. The current US GAP is up to 0.45 kg ai/ha with a seasonal maximum rate of 0.9 kg/ha and a PHI of 21 days.

As 5–10 applications were made with low dose rates, which do not represent the critical GAP, no recommendations could be made.

The Meeting withdraws its previous recommendation of 1 mg/kg.

Tree nuts

A total of ten supervised trials on tree nuts, 5 on almonds and 5 on pecans have been conducted in the US in 2003, with application rates of 0.45 and 0.9 kg ai/ha at 7 days intervals instead of the minimum 10 days specified on the label.

The following average residue levels in two composite samples were obtained from the trials on almonds and pecans:

Almond nutmeat: < 0.01(4), 0.03 mg/kg; and in pecans: < 0.01, 0.01, 0.02, 0.05, 0.06 mg/kg.

The Meeting noted that the GAP in USA is for tree nuts and the residue populations were not significantly different (Mann-Whitney U-test) and the median residues were within 5 times range. The Meeting agreed to combine the datasets for almonds and pecans which, in ranked order, were: < 0.01(4), < 0.01, 0.01, 0.02, 0.03, 0.05, and 0.06 mg/kg.

As the highest residue in an individual samples was 0.1 mg/kg, the Meeting estimated a maximum residue level of 0.15 mg/kg, HR of 0.1 mg/kg and STMR value of 0.01 mg/kg.

Coffee Beans

Six supervised trials on coffee were conducted in Brazil in 2013 following the current GAP there (two applications at a maximum rate of up to 0.12 kg ai/ha and a PHI of 14 days).

Residues in composite samples, in ranked order, were: < 0.01(4), 0.01, and 0.02 mg/kg.

The Meeting estimated a maximum residue level of 0.03 mg/kg and STMR value of 0.01 mg/kg.

Tea

All supervised trials on tea were conducted in India during 2002-2004. The compound was applied according to the GAP in India (0.05-0.06 kg ai./ha with a PHI of 7 days) and with double rate.

Six trials on black tea and in one trial green tea leaves (0.13 mg/kg) were analysed

The residues in composite samples following application at the GAP rate were: < 0.05, 0.13, 0.14(2), 0.17, 0.18, and 1.38 mg/kg.

The Meeting estimated a maximum residue level for green and black tea of 3 mg/kg and STMR value of 0.14 mg/kg.

The Meeting withdrew its previous recommendation of 2 mg/kg for maximum residue level for tea.

Animal feeds

Almond hulls

The US GAP specifies application rates of up to 0.45 kg ai/ha with a seasonal maximum of 0.9 kg ai/ha at 10 days intervals, and a PHI of 3 days.

In almond hulls, the ranked order of residue concentrations was 2.7, 2.9, 3.1, 3.5, and 3.6 mg/kg. The Meeting estimated a maximum residue level of 10 mg/kg, the highest residue is 3.6 mg/kg and the median residue is 3.1 mg/kg.

Cottonseed hull

The trial conditions did not match US GAP (2 × 0.45, max seasonal rate 0.9 mg/kg, PHI 21 days), no recommendations could be made.

Fate of residues during processing

Processing studies were carried out on plums, tomato, olives, oranges, cottonseed and tea.

The processing factors calculated and STMR-P values estimated are summarized below.

Summary of selected processing factors and STMR-P values for fenpropathrin

RAC/processed fraction	Processing factors					PF estimated	STMR-P (mg/kg)
RAC: Whole orange	-						
Juice	< 0.02	< 0.22				< 0.02	0.007
Oil	78.7	21.56				50.1	16.5
Wet peel	0.6	0.78	2.76		2.86	2.82	0.93
Dried peel	1.6	2.67				2.1	0.70
Pulp			0.06		0.07	0.065	0.021
RAC: Plum							
Dried plum	2.56					2.56	0.639
RAC: Tomato							
Canned	0.077	0.071	0.077			< 0.075	0.021
Wet pomace				9.9	9.8	9.8	1.867
Dry pomace				46	45.0	45	8.618
Tomato paste				0.78	0.75	0.77	0.145
Tomato juice				0.12	0.1	0.12	0.023

Note: The residues measured in RAC samples taken at the processing plants are considered as they better reflect the residues in unprocessed commodities than these measured in field samples

There is no concentration of residues in juice and molasses. Residues concentrate in oil (Pf=50.1), and dried peel (Pf=2.1).

The Meeting estimated a maximum residue level of 100 mg/kg and STMR-P of 16.5 mg/kg for citrus oil, Drying concentrates the residues of fenpropathrin in plums by a factor of 2.6×; The Meeting estimated a maximum residue level of 3 mg/kg, HR-P of 1.85 mg/kg and STMR-P of 0.65 mg/kg for dried plums (or prunes).

As no MRL could be estimated, the Meeting withdrew its previous recommendation of 3 mg/kg for cottonseed oil.

Residues in animal commodities

Estimation of dietary burden

The maximum and mean dietary burdens were calculated using the highest residues or median residues of fenpropathrin estimated at the current Meeting on a basis of the OECD Animal Feeding Table. Only almond hull, citrus pulp and tomato wet pomace can be used as animal feed based on recommended uses. The calculated maximum and mean animal burdens are summarised below

Summary of livestock dietary burdens (ppm of dry matter diet)

	US-Canada		EU		Australia		Japan	
	max	mean	max	Mean	max	mean	Max	mean
Beef cattle	0.09	0.09	0.045	0.045	1.46[a]	1.46[b]	0	0
Dairy cattle	0.43	0.04	0.18	0.18	1.46	1.46	0	0
Broilers	0	0	0	0	0	0	0	0
Layers	0	0	0	0	0	0	0	0

[a] Suitable for estimating maximum residue levels for milk, meat, fat and edible offal of cattle.

[b] Suitable for estimating STMRs for meat, fat and edible offal of cattle.

Farm animal feeding studies

Lactating Holstein cows were orally administered technical grade fenpropathrin (purity 92.5%) via gelatin capsules for 28 consecutive days in two equal portions at the morning and evening milkings. The treatment levels were 0, 25, 75 and 250 ppm fenpropathrin based upon the daily average food consumption.

Residues of fenpropathrin in the milk reached a plateau after three days. Average residues in the whole milk of the four cows of each group on Day 3 were 0.04, 0.17 and 0.33 mg/kg for the three dose levels. On Day 28, these levels were 0.04, 0.13 and 0.32 mg/kg. At the end of the three-day depuration period, residues had fallen to < 0.01, 0.02 and 0.04 mg/kg for the three levels. Pasteurization did not significantly reduce fenpropathrin residues in milk. The residues concentrated in milk fat by a factor of about 10 (from mean of 0.32 mg/kg in whole milk to 3.7 mg/kg in milk fat)

After 28 days of dosing, maximum and (average) residues, expressed in mg/kg, in muscle, kidney, liver and fat were 0.33 (0.2), 0.2 (0.16), 0.01 (0.01), and 4.1 (3.8) mg/kg, respectively, at the maximum 250 ppm dose level. The residues determined after feeding with 25 ppm fenpropathrin in feed, and the corresponding residues in tissues and milk resulted from the calculated mean and max dietary burden (1.46 ppm) are summarised below.

Dietary burden	Fat		Meat		Liver		Kidney		Milk
	Max	Mean	Max	Mean	Max	Mean	Max	Mean	Mean
25 ppm	0.44	0.33	0.04	0.02	< 0.01	< 0.01	0.05	0.03	0.04
1.46 ppm	0.026	0.018	0.002	0.001	< 0.0006	< 0.0006	0.003	0.002	0.002

Based on the data available the Meeting estimated maximum residue levels of 0.03 mg/kg, HR value of 0.026 mg/kg and STMR value of 0.018 mg/kg for mammalian fat except milk fat.

The Meeting estimated, at the LOQ of 0.01 mg/kg, maximum residue level of 0.01 mg/kg for mammalian meat and edible offal and 0.01 mg/kg for milk. The HR values for meat and edible offal are 0.002 mg/kg and 0.003 mg/kg, respectively

The Meeting estimated STMR values of 0.001 mg/kg for mammalian meat, 0.002 mg/kg for mammalian, edible offal of, and 0.002 mg/kg for milk.

The Meeting withdraws its previous recommendations for cattle meat, edible offal and milk.

Laying hens

Laying hens were dosed at nominal concentrations of 0, 2.5, 7.5 and 25 ppm levels for a period of 28 days. The fenpropathrin residues were below 0.01 mg/kg in case of dose groups 2.5 and 7.5 ppm over the study period. Eggs derived from 25 ppm dose contained 0.02 mg/kg fenpropathrin from day 7. Residues in muscle, gizzard and liver samples were below the LOQ of 0.01 mg/kg in all dose groups. The fenpropathrin residue in fat was 0.02, 0.05 and 0.14 mg/kg for dose groups of 2.5, 7.5 and 25 ppm. Metabolites could only be detected in liver after dosing with 25 ppm were TMPA (0.05 mg/kg) and PBA-glycin (0.03 mg/kg). The distribution of residues between white and yolk was not studied.

Taking into account that poultry feed is not treated with fenpropathrin according to the uses evaluated by the present Meeting, the Meeting estimated maximum residues levels in poultry meat , fat, edible offal and eggs of 0.01 mg/kg*.

The Meeting estimated STMR values of 0 for poultry products

The Meeting withdraws its previous recommendations for poultry products.

RECOMMENDATIONS

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

Definition of residue for compliance with MRL and for estimation of dietary intake for animal and plant commodities is fenpropathrin.

The residue in fat soluble.

CCN	Commodity Name	Recommended MRL, mg/kg		STMR/STMR-P mg/kg	HR/HR-P mg/kg
		New	Previous		
AM 0660	Almond hulls	10		3.1	3.6
MM 0812	Cattle meat	W	0.5 (fat)		
ML 0812	Cattle milk	W	0.1F		
MO 0812	Cattle, edible offal of	W	0.05		
FS 0013	Cherries	7[a]		1.85	3.53
HS 0444	Peppers Chili, dried	10		2.59	4.9
FC 0001	Citrus fruits	2		0.02	0.098
OR 0001	Citrus oil, edible	100		16.5	
SB 0716	Coffee beans	0.03		0.01	
SO 0691	Cotton seed	W	1		
OC 0691	Cotton seed oil, crude	W	3		
MO 0105	Edible offal (mammalian)	0.01		0.002	0.003
VO 0440	Egg plant	W	0.2		
PE 0112	Eggs	0.01*	0.01*	0	
VC 0425	Gherkin	W			
FB 0269	Grapes	W	5		
MF 0100	Mammalian fats (except milk fats)	0.03		0.018	0.026
MM 0095	Meat (from mammals other than marine mammals)	0.01		0.001 0.018 (fat)	0.002
ML 0106	Milks	0.01		0.002	
FS 2001	Peaches (including Nectarine and Apricots)	3[a]		0.71	1.1
VO 0051	Peppers	1		0.37	0.70
FS 0014	Plums (including prunes)	1		0.25	0.71
FP 0009	Pome fruits	3[a]		0.73	2
PF 0111	Poultry fats	0.01*		0	0
PM 0111	Poultry meat	0.01* (fat)		0	0
PO 0111	Poultry, Edible offal of	0.01*	0.01*	0	0
DF 0014	Prunes	3		0.65	1.85
VD 0541	Soya bean (dry)	0.01		0.01	
FB 0275	Strawberry	2		0.515	1.2
DT 1114	Tea, Green, Black (black, fermented and dried)	3	2	0.14	
VO 0448	Tomato	1		0.19	0.64
TN 0085	Tree nuts	0.15		0.01	0.1
JF 0001	Citrus juice			0.007	
JF 0048	Tomato juice			0.023	
	Tomato canned			0.021	
VW 0448	Tomato paste			0.145	

DIETARY RISK ASSESSMENT

Long-term intake

The evaluation of fenpropathrin resulted in recommendations for MRLs and STMR values for 24 raw and processed commodities. Where data on consumption were available for the listed food commodities, dietary intakes were calculated for the 17 GEMS/Food Consumption Cluster Diets. The results are shown in Annex 3 to the 2014 Report.

The IEDIs in the seventeen Cluster Diets, based on the estimated STMRs were 1-10% of the maximum ADI (0.03 mg/kg bw). The Meeting concluded that the long-term intake of residues of fenpropathrin from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short-term Intake (IESTI) for fenpropathrin was calculated for 24 raw and processed commodities for which maximum residue levels and STMR values were estimated. The results are shown in Annex 4 to the 2014 Report.

For cherries, peaches, and pome fruits the IESTI represented 140%, 180% and 390% of the ARfD of 0.03 mg/kg bw, respectively. No alternative GAP was available. On the basis of information provided to the JMPR it was not possible to conclude that the estimate of short-term intake of fenpropathrin, from the consumption of cherries, peaches and pome fruits, was less than the ARfD.

The other commodities considered by the JMPR were within 0–80% of ARfD. The Meeting concluded that the short-term intake of fenpropathrin when used in ways that have been considered by the MPR is unlikely to present public health concern.

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