FENPROPATHRIN (186)

IDENTITY

ISO common name: fenpropathrin

Chemical names:

IUPAC: (RS)-a-cyano-3-phenoxybenzyl ,2,3,3-tetramethylcyclopropanecarboxylate

CAS: cyano(3-phenoxyphenyl)methyl 2,2,3,3-tetramethylcyclopropanecarboxylate

Synonyms: S-3206, Danitol, Meothrin, Rody, OMS 1999, WL41706, SD41706, XE-938

CAS Registry No.: 64257-84-7(racemate); 39515-41-8 (unstated steriochemistry)

Structural formula:

Molecular formula: $C_{22}H_{23}NO_3$

Molecular weight: 349.43

Physical and chemical properties

Pure active ingredient

Information was given only for the technical material.

Technical material

Purity: 90%

Physical state: Liquid or solid
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°C
Ignition point: 325°C
Solubility in organic solvents (g/l at 23°C):
Acetone > 500
Acetonitrile > 500
Cyclohexanone > 500
Ethyl acetate > 500
Methanol 216
Xylene > 500

Solubility in water 36.3 ì g/l at 25.1°C
Octanol/water partition coefficient:
log P = 6.0 $\pm$ 0.20
Stability: Unstable in alkaline media.
No significant breakdown after 20 weeks storage at 60°C.

Formulation: EC

USE PATTERN

Fenpropathrin is used to control a range of insects, especially mites, in fruits and vegetables. Registered uses are summarized in Table 1. Most countries approve a range of application rates and pre-harvest intervals. The rate of application for tree fruits is normally expressed in terms of spray concentration but a complication arises with low-volume applications, although these are not currently established on a commercial scale. The number of applications permitted for a given crop is seldom specified in official registrations although in two countries, Denmark and Germany, only 2 or 3 applications are allowed for certain crops (see Table), not to limit residues but because the competent authorities operate a policy of rotating insecticides to minimize the development of pest resistance. In practice the number of applications is determined by infection pressure and the persistence of the active ingredient. This is comparatively long because in addition to its insecticidal properties fenpropathrin also exerts a considerable repellent action. The effects of a treatment normally last for 3-4 weeks.

Table 1. Registered uses of fenpropathrin.

Crop	Country	Application			PHI, days
		No.	Rate, kg ai/ha	Spray concn., kg ai/hl	
Apple	Austria			0.005-0.008	21
	Belgium			0.005	14

Crop	Country	Application			PHI, days
		No.	Rate, kg ai/ha	Spray concn., kg ai/hl	
	Cyprus			0.005	21
	Denmark	2	0.15		14
	France			0.01 -0.02	21
	Greece			0.005-0.02	21
	Hungary	2	0.06-0.1		14
	Italy			0.005-0.025	7
	Japan	2		0.007-0.01	14
	Nthlnds			0.005	14
	Portugal			0.02	7
	Spain	2	0.09-0.225	0.006-0.015	30
	Sweden	1	0.15	0.0075	30
	Swtzlnd	2		0.01	42
	UK	2		0.003-0.005	7
	USA	8	0.45		14
Beans	Cyprus			0.005	7
	Germany	3	0.04 -0.08*	0.033-0.067	3
	Portugal			0.01*	2
	Swtzlnd	1		0.01	14
		1		0.01*	7
Currant, black	Sweden	1	0.112	0.0075	60
Cabbage, Head	Swtzlnd	2		0.01	7
Citrus fruits	Cyprus			0.005	21
	Greece	2		0.02	21
	Italy	2		0.02	30
	Japan	4		0.005	7
Cotton seed	Greece		0.13-0.15		
	Spain	2	0.13-0.15		30
	USA	10	0.22		14

Crop	Country	Application			PHI, days
		No.	Rate, kg ai/ha	Spray concn., kg ai/hl	
Cucumber	Austria			0.005	3
	Belgium			0.005	3
	Cyprus			0.007-0.02	21
	Denmark	2		0.0075	3
	Germany	3	0.04-0.08*	0.033-0.067	3
	Greece			0.02	21
	Hungary		0.03-0.1		7
	Italy			0.01 -0.02	7
	Japan	5		0.005-0.01	1
	Nthlnds			0.005	3
	Norway			0.0075	4
	Swtzlnd			0.01*	7
Egg plant	Austria	1		0.005	3
	Belgium	3		0.005	3
	Cyprus			0.005	7
	Greece	1		0.01	21
	Japan	5		0.01	1
	Nthlnds	2		0.005	3
	Spain	2	0.09-0.225	0.006-0.015	7
Gherkin	Belgium	2		0.005	3
	Nthlnds			0.01*	7
Grapes	Austria			0.005	21
	Cyprus			0.007-0.02	21
	France		0.08-0.15		21
	Greece			0.025-0.02	21
	Hungary	2	0.06-0.1		14
	Italy			0.003-0.02	7
	USA	4	0.45		21
Hops	Austria	1		0.005	21

Crop	Country	Application			PHI, days
		No.	Rate, kg ai/ha	Spray concn., kg ai/hl	
	UK	2		0.006	7
Maize	Austria	1		0.005	3
Melons, except Water-melon	Austria	1		0.005	3
	Belgium	1		0.005	3
	Greece			0.02	21
	Japan	4		0.01	1
	Nthlnds	2		0.005	3
	Portugal			0.01*	2
	Spain	1		0.01	30
Mushrooms	Austria	1		0.005	3
Peach	France	2	0.1		21
	Greece			0.02	21
	Italy	2		0.02	7
	Japan	5		0.01	1
	Portugal			0.02	7
	Swtzlnd	2		0.01	42
Pear	Austria			0.005-0.008	21
	Belgium			0.005	14
	Cyprus			0.007-0.02	21
	Denmark	2	0.15		14
	France			0.01 -0.02	21
	Greece			0.005-0.02	21
	Hungary	2	0.06-0.1		14
	Italy			0.005-0.025	7
	Japan	2		0.007-0.01	14
	Nthlnds	1		0.005	14
	Portugal			0.02	7
	Spain	2	0.09-0.225	0.006-0.015	30

Crop	Country	Application			PHI, days
		No.	Rate, kg ai/ha	Spray concn., kg ai/hl	
	Swtzlnd	2		0.01	42
	USA	8	0.45		14
Peppers	Belgium	3			0.0053
	Greece	1			0.0221
	Japan	3			0.0051
	Spain	2	0.09-0.225	0.006-0.015	7
Peppers, Sweet	Austria	1		0.005	3
	Nthlnds	2		0.005	3
Plums	Sweden	1	0.168	0.0075	60
Potato	Cyprus			0.005	7
	Greece			0.01	21
	Italy	1		0.01	21
Pumpkins	Austria	1		0.005	3
Squash, Summer	Austria	1		0.005	3
	Belgium			0.005	3
	Italy	1		0.02	7
	Nthlnds	2		0.005	3
	Spain	2	0.09-0.225	0.006-0.015	7
Tomato	Austria			0.005	3
	Belgium	3		0.005	3
	Cyprus			0.007-0.02	7
	Denmark	2		0.0075*	3
	Germany	3	0.04-0.08*	0.033-0.067	3
	Greece	1		0.01 -0.02	21
	Hungary		0.03-0.1		7
	Italy	5		0.01 -0.02	7
	Japan	3		0.005-0.01	1
	Nthlnds	2		0.005	3
	Norway	1		0.008*	4

Crop	Country	Application			PHI, days
		No.	Rate, kg ai/ha	Spray concn., kg ai/hl	
	Portugal			0.01	2
	Spain	4	0.09-0.225	0.006-0.015	7
Strawberry	Sweden	1	0.08-0.1	0.0075	BF**

* Greenhouse
** Before flowering or after harvest

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residues in crops

A series of studies have been carried out in Europe, Japan and the USA to determine the level of residues likely to arise in crops when fenpropathrin is used according to the range of recommendations for use (Tables 2-9). Crops were mostly treated according to accepted or proposed use recommendations, although in the USA some crops were treated at higher rates. Crop commodities were generally sampled at maturity except in cases where the design of the study involved a range of pre-harvest intervals which were varied by changing the harvest date rather than the date of the last spray application.

The Tables are as follows.

Table
2 Residues of fenpropathrin in apples
3 Residues of fenpropathrin in pears
4 Residues of fenpropathrin in grapes
5 Residues of fenpropathrin in cotton seed
6 Residues of fenpropathrin in gherkins
7 Residues of fenpropathrin in egg plants
8 Residues of fenpropathrin in sweet peppers
9 Residues of fenpropathrin in tomatoes

Underlined residues in Tables 2-9 are from treatments according to GAP. Residues have not normally been corrected for recoveries.

Apples. The available data are summarized in Table 2. As would be expected residue levels were seen to increase with increased application rates, as measured by spray concentration, decreased PHI and, to some extent, increased numbers of applications.

Table 2. Residues of fenpropathrin in apples - whole fruit

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
France	10% EC	1	0.2	0.029	0	0.30	10

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
1981					7	0.36	10
					14	0.29	10
					21	0.31	10
France	10% EC	1	0.2	0.020	0	0.19	10
1981					7	0.20	10
					14	0.13	10
					21	0.13	10
France	10% EC	1	0.2	0.029	0	0.48	10
1981					7	0.49	10
					14	0.60	10
					22	0.16	10
France	10% EC	1	0.2	0.020	0	0.34	11
1983					7	0.25	11
					14	0.18	11
					21	0.06	11
France	10% EC	1	0.2	0.020	0	0.20	11
1983					7	0.16	11
					14	0.21	11
					21	0.10	11
France	10% EC	1	0.2	0.020	0	0.75	11
1983					7	0.50	11
					14	0.30	11
					21	0.50	11
Hungary	10% EC	4	0.1	0.007	0	0.08	40
1984					1	0.09	40
					5	0.08	40
					9	0.05	40
					14	0.04	40
Hungary	10% EC	1	0.1	0.010	0	0.20	5
1984					1	0.20	5
					3	0.14	5
					6	0.11	5
					10	0.07	5
					15	0.03	5

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
					30	0.01	5
USA	2.4 EC	9	0.34	0.009	14	3.3	71
1984		9	0.45	0.012	14	3.9	71
USA	2.4 EC	6	0.34	0.009	14	0.96	66
1984		6	0.45	0.012	14	1.2	66
USA	2.4 EC	8	0.34	0.009	14	1.4	67
1984		8	0.45	0.012	14	1.0	67
USA	2.4 EC	8	0.34	0.09	14	0.38	68
1984		8	0.45	0.12	14	0.57	68
USA	2.4 EC	2	0.45	0.024	14	0.88	77
1984		4	0.45	0.024	14	2.6	77
		6	0.45	0.024	14	2.5	77
		8	0.45	0.024	14	1.7	77
USA	2.4 EC	8	0.34	0.073	14	2.1	69
1984		8	0.45	0.096	14	3.7	69
USA	2.4 EC	2	0.45	0.016	14	1.1	70
1984		4	0.45	0.016	14	2.4	70
		5	0.45	0.016	14	3.0	70
		8	0.45	0.016	14	2.6	70
USA/84	2.4 EC	8	0.34	0.009	14	2.0	78
USA	2.4 EC	8	0.45	0.012	7	2.1	78
1984					14	2.4	78
					21	2.1	78
					28	2.2	78
USA	2.4 EC	2	0.45	0.045	14	0.48	80
1985		4	0.45	0.045	14	1.9	80
		6	0.45	0.045	14	2.6	80
		8	0.45	0.045	14	3.7	80
USA	2.4 EC	8	0.112	0.010	14	0.02	38
1985		8	0.224	0.020	14	2.6	38
		8	0.45	0.040	14	4.5	38
		8	0.9	0.080	14	8.3	38
USA/85	2.4 EC	8	0.45	0.16	14	1.4	39
USA/86	2.4 EC	8	0.45	0.012	14	0.14	T-6719*

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
USA/86	2.4 EC	8	0.45	0.016	14	3.6	T-6720
USA/86	2.4 EC	8	0.45	0.035	14	1.5	T-6721
USA	2.4 EC	8	0.45	0.012	14	3.7	T-6722
1986					42	2.8	
USA/86	2.4 EC	8	0.45	0.024	14	2.4	T-6729
USA/87	2.4 EC	8	0.45	0.012	14	2.3	T-6880
USA/87	2.4 EC	8	0.45	0.69**	12	0.40	T-6969
USA/87	2.4 EC	8	0.45	0.96**	14	0.22	T-6970
USA/87	2.4 EC	8	0.45	0.96**	14	0.88	T-6971

* All "T" references are in Fujie 1990b (No. 24).
** Aerial application, hence very low volume per ha.

Pears. The residues in pears were comparable to those in apples; the results are summarized in Table 3 and refer to essentially mature fruit. In two of the three US studies (Robinson, 1984f; 1985b) the samples were all taken on the same day, the variation of the pre-harvest interval being achieved by varying the date of the last application.

Table 3. Residues of fenpropathrin in pears: whole fruit.

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
France	10% EC	1	0.15	0.015	0	0.25	8
1980					7	0.21	8
					14	0.20	8
					21	0.12	8
France	10% EC	1	0.15	0.015	0	0.10	8
1980					8	0.05	8
					12	0.03	8
					22	<0.01	8
France	10% EC	1	0.2	0.02	0	0.65	9
1981					7	0.35	9
					14	0.24	9
					21	0.17	9
France	10% EC	1	0.2	0.02	0	0.42	9
1981					7	0.33	9
					14	0.1	9

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
					21	0.10	9
France	10% EC	1	0.2	0.02	0	0.19	9
1981					7	0.17	9
					14	0.19	9
					21	0.17	9
USA/84	2.4 EC	6	0.34	0.009	14	0.59	60
USA	2.4 EC	6	0.45	0.012	7	0.68	60
1984					14	0.62	60
					21	0.56	60
					28	0.49	60
USA/84	2.4 EC	6	0.34	0.009	14	0.39	61
USA	2.4 EC	2	0.45	0.012	14	0.27	61
1984		3	0.45	0.012	14	0.45	61
		5	0.45	0.012	14	0.42	61
		6	0.45	0.012	14	0.58	61
USA/85	2.4 EC	6	0.45	0.012	14	1.2	79
USA	2.4 EC	6	0.34	0.009	14	0.94	62
1984		6	0.45	0.012	14	1.3	62
USA/84	2.4 EC	6	0.34	0.024	14	1.3	63
USA	2.4 EC	2	0.45	0.016	14	1.2	64
1984		4	0.45	0.016	14	1.5	64
		5	0.45	0.016	14	1.4	64
		6	0.45	0.016	14	1.7	64
USA	2.4 EC	6	0.45	0.016	7	2.3	65
1984					14	1.9	65
					21	2.0	65
					28	0.95	65
USA	2.4 EC	6	0.11	0.003	14	0.70	37
1985		6	0.22	0.006	14	0.96	37
		6	0.45	0.012	14	1.0	37
		6	0.90	0.024	14	3.2	37
USA	2.4 EC	2	0.45	0.012	14	0.30	72
1985		4	0.45	0.012	14	0.63	72

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
		6	0.45	0.012	14	0.74	72
		8	0.45	0.012	14	0.95	72
USA/85	2.4 EC	8	0.45	0.012	21	0.09	72
USA	2.4 EC	6	0.45	0.012	1	1.5	73
1985					7	2.2	73
					14	1.6	73
					21	1.3	73
					28	0.85	73
					35	0.95	73
USA/86	2.4 EC	6	0.45	0.048	14	1.1	T-6709*
USA/86	2.4 EC	6	0.45	0.012	14	2.9	T-6711
USA/86	2.4 EC	6	0.45	0.035	14	1.8	T-6712
USA/86	2.4 EC	6	0.45	0.016	14	2.4	T-6713
USA/87	2.4 EC	6	0.45	0.048	14	1.8	T-6886
USA/87	2.4 EC	6	0.45	0.24**	14	0.3	T-6972
USA/87	2.4 EC	6	0.45	0.96**	14	0.9	T-6973

* All "T" references are in Fujie 1988 (No. 22).
** Aerial application, hence very low volume per ha.

Grapes. The data are shown in Table 4 and most of them refer to mature grapes, an exception being the results from Hungary at a 70-day pre-harvest interval. The US study T-6415 did not involve a variation of the date of the last application but the samples were all described as mature bunches so presumably the range of pre-harvest intervals was too narrow to have a significant effect on maturity. The other three relevant studies in the USA (T-6409, 6414 and 6835) were designed in such a way that the grapes were all harvested at a similar state of maturity.

Table 4. Residues of fenpropathrin in grapes: whole fruit.

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
France	10% EC	1	0.075	0.015	0	0.08	7
1980					7	0.04	7
					14	0.02	7
					21	0.01	7

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
France	10% EC	1	0.075	0.015	0	0.41	7
1980					8	0.23	7
					14	0.23	7

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
					21	0.12	7
France	10% EC	1	0.075	0.019	7	0.15	6
1978					14	0.10	6
					24	0.06	6
France	10% EC	1	0.05	0.009	63	0.01	6
1978		1	0.075	0.014	63	0.02	6
Hungary	10% EC	1	0.08		0	0.08	83
1983					1	0.09	83
					4	0.11	83
					6	0.10	83
					11	0.09	83
					14	0.09	83
					21	0.07	83
					29	0.07	83
					70	0.04	83
Hungary	10% EC	2	0.1	0.01	0	0.19	30
1984					1	0.16	30
					3	0.12	30
					8	0.06	30
					14	0.02	30
					21	0.01	30
					28	<0.005	30
					35	<0.005	30
USA/82	2.4 EC	1	0.45	0.024	21	0.11	4
USA/83	2.4 EC	2	0.22 0.11	0.021 0.011	25 25	0.14 0.10	T-5952*
USA/84	2.4 EC	4	0.22	0.022	21	0.37	T-6077*
USA/84	2.4 EC	4	0.22	0.015	21	0.75	T-6078*
USA/84	2.4 EC	2	0.22	0.009	14	0.57	T-6079*
USA/84	2.4 EC	1	0.22	0.024	95	0.22	T-6081*

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
USA/85	2.4 EC	4	0.22	0.022	1	0.42	T-6409*
					7	0.65	
					14	0.44	
					21	0.52	
					28	0.51	
					35	0.83	
USA/85	2.4 EC	4	0.056	0.003	21	0.11	T-6410*
		4	0.11	0.006	21	0.13	
		4	0.22	0.012	21	0.45	
		4	0.45	0.024	21	1.2	
USA/85	2.4 EC	1 2	0.22 0.22	0.012 0.012	21 21	0.28 0.58	T-6411*
		3	0.22	0.012	21	0.89	
		4	0.22	0.012	21	1.5	
USA/85	2.4 EC	4	0.22	0.022	21	0.74	T-6412*
USA/85	2.4 EC	4	0.22	0.016	21	3.1	T-6413*
USA/85	2.4 EC	1	0.22	0.024	1	2.3	T-6414*
					7	2.0	
					14	1.7	
					21	1.4	
					28	0.5	
					35	0.5	
USA/85	2.4 EC	4	0.22	0.024	1	1.1	T-6415*
					7	0.91	
					14	0.67	
					21	1.1	
					28	0.73	
					35	0.90	
USA/85	2.4 EC	4	0.056	0.006	21	0.15	T-6416*
		4	0.11	0.012	21	0.27	

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
		4	0.22	0.024	21	1.1	
		4	0.45	0.048	21	2.6	
USA/85	2.4 EC	1	0.22	0.024	21	0.21	T-6417*
		2	0.22	0.024	21	1.3	
		3	0.22	0.024	21	1.5	
		4	0.22	0.024	21	1.5	
USA/86	2.4 EC	4	0.22	0.024	19	1.1	T-6725*
USA/86	2.4 EC	4	0.22	0.024	14	1.4	T-6726*
USA/86	2.4 EC	4	0.22	0.022	21	1.0	T-6728*
USA/86	2.4 EC	4	0.22	0.024	21	1.4	T-6731*
USA/86	2.4 EC	4	0.22	0.024	21	5.6	T-6829*
USA/86	2.4 EC	4	0.41	0.023	1	0.99	T-6835*
					7	2.6	
					14	1.7	
					21	1.3	
					28	1.2	
					35	2.0	
USA/90	2.4 EC	4	0.22	0.098	21	0.81	T-7544**
USA/90	2.4 EC	4	0.22	0.094	21	0.53	T-7545**
		4	0.45	0.192	21	0.84	

* These "T"references are subdivisions of Fujie, 1990c (No. 25)
** These "T"references are subdivisions of Fujie, 1992 (No. 26)

Cotton seed. The available results are shown in Table 5.

Table 5. Residues of fenpropathrin in cotton seed in the USA. All trials with 2.4 EC.

Year	Application			PHI, days	Residue, mg/kg	Ref.
	No.	kg ai/ha	kg ai/hl			
1975	11	0.22	0.24	20	0.03	75
1975	11	0.22	0.24	20	0.02	76

Year	Application			PHI, days	Residue, mg/kg	Ref.
	No.	kg ai/ha	kg ai/hl			
	8	0.11	0.24	38	<0.01	57
1975	8	0.22	0.47	38	<0.01	57

Year	Application			PHI, days	Residue, mg/kg	Ref.
	No.	kg ai/ha	kg ai/hl			
	17	0.11	0.24	44	0.03	2
1975	17	0.22	0.47	44	0.07	2
	17	0.44	0.94	44	0.02	2
1975	8	0.22	0.47	22	<0.01	84
	3	0.28	0.6	62	<0.05	74
1974	3	0.56	1.6	62	<0.05	74
	3	0.28	0.6	62	<0.01	41
1974	3	0.56	1.2	62	<0.01	41
	8	0.22	0.079	35	<0.01	T-6023*
1983	8	0.45	0.16	35	<0.01	
1983	8	0.11	0.094	33	<0.01	T-6024
1984	10	0.22	0.47**	18	<0.01	T-6069
1984	9	0.22	0.24	21	0.02	T-6070
1984	10	0.22	0.24	18	0.03	T-6071
1984	10	0.22	0.24	21	<0.01	T-6072
1984	10	0.22	variable***	21	0.01	T-6073
1984	11	0.22	0.094	34	0.26	T-6074
1984	10	0.22	0.22	21	<0.01	T-6075
1984	10	0.22	0.24	20	0.29	T-6076
	10	0.22	0.079	3	0.13	T-6418
1985	10	0.15		10	0.15	
	14	0.03		14	0.03	
	21	0.12		21	0.12	
	28	<0.01		28	<0.01	
	35	<0.01		35	<0.01	
	10	0.11	0.039	21	0.02	T-6419
1985	10	0.22	0.079	21	0.01	
	10	0.45	0.161	21	0.03	
1985	10	0.22	0.24**	21	0.02	T-6420

Year	Application			PHI, days	Residue, mg/kg	Ref.
	No.	kg ai/ha	kg ai/hl			
1985	10	0.22	0.47**	21	<0.01	T-6421
1985	10	0.22	0.52	21	0.08	T-6422
1985	10	0.22	0.47	3	3.3	T-6423
				7	0.76	
				14	0.20	
				21	0.02	
				28	0.01	
				35	<0.01	
1985	10	0.22	0.24	21	<0.01	T-6424
1985	10	0.22	0.24	22	<0.01	T-6425
1985	10	0.22	0.47	30	0.01	T-6426
	10	0.11	0.12	21	0.09	T-6427
1985	10	0.22	0.24	3	0.52	
				7	0.47	
				14	0.36	
				21	0.32	
				36	0.31	
	10	0.45	0.48	7	1.20	
1986	8	0.22	0.08	21	1.0	T-6715
1986	8	0.22	0.08	20	0.53	T-6716
1986	8	0.22	0.24	21	0.07	T-6717
1986	8	0.22	0.47	21	0.07	T-6718
1987	8	0.22	1.18**	21	0.27	T-6967
1987	8	0.22	1.18**	21	<0.01	T-6968
1989	5	0.34	0.73	21	0.02	T-7376
1989	5	0.34	0.073	21	0.01	T-7377
1989	5	0.34	0.125	20	0.28	T-7378
1989	5	0.34	0.073	21	0.06	T-7379
		0.34	0.73	21	0.06	T-7380

Year	Application			PHI, days	Residue, mg/kg	Ref.
	No.	kg ai/ha	kg ai/hl			
1989	5	0.34	3.64**	21	0.03	T-7381
1989	5	0.34	3.64**	21	0.04	T-7382

* The "T" references are subdivisions of Fujie, 1990a
** Aerial application
*** The volumes applied varied from 430 to 700 l/ha

Gherkins. The data on gherkins shown in Table 6 were developed in greenhouse trials.

Table 6. Residues of fenpropathrin in greenhouse gherkins

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
Denmark	5% EC	1		0.0075	1	0.05	42
1984					2	0.05	
					4	0.01	
					7	<0.01	
Germany	10% EC	3	0.08	0.009	0	0.04	93
1983					1	0.03	
					3	<0.01	
					5	0.01	
					7	0.01	
Germany	10% EC	3	0.08	0.009	0	0.10	94
1983					1	<0.01	
					3	<0.01	
					5	<0.01	
					7	<0.01	
Germany	10% EC	3	0.08	0.009	0	0.06	95
1983					1	0.07	
					3	0.02	
					5	0.03	
					7	0.02	
Germany	10% EC	3	0.08	0.009	0	0.08	96
1983					1	0.15	
					3	0.10	
					5	0.07	
					7	0.03	

Egg plants. The results in Table 7 were submitted by Spain.

Table 7. Residues of fenpropathrin in egg plants, reference 97, all trials with 10% EC. No information on year.

Country,	Application	PHI,	Residue,

Year	No.	kg ai/hl	days	mg/kg
Japan	3	0.01	1	0.12
			3	0.04
			7	0.005
Japan	3	0.01	1	0.19
			3	0.16
			7	0.09
Japan	5	0.01	1	0.12
			3	0.04
			7	0.06
Japan	5	0.01	1	0.18
			3	0.16
			7	0.07
France	1	0.015		fruit
			0	0.07
			7	0.06
			14	<0.01
			21	<0.01
				pulp
			0	<0.01
			7	<0.01
			14	<0.01
			21	<0.01
France	2	0.015		fruit
			0	0.08
			7	0.01
			14	<0.01
			21	<0.01
				pulp
			0	<0.01
			7	<0.01
			14	<0.01
			21	<0.01

Sweet peppers. The results of 7 Spanish trials, 4 Japanese trials and 1 trial from Denmark are summarized in Table 8.

Table 8. Residues of fenpropathrin in sweet peppers, reference 97, all trials with 10% EC.

Country year	Application	PHI, days	Residue, mg/kg

	No.	kg ai/ha	kg ai/hl		
Japan*	3		0.01	1	0.91
outdoor				3	0.86
				7	0.32
Japan*	5		0.01	1	0.88
outdoor				3	0.58
				7	0.42
Japan*	3		0.01	1	0.92
outdoor				3	0.68
				7	0.24
Japan*	5		0.01	1	1.18
outdoor				3	0.76
				7	0.48
Denmark*	1		0.0075	0	0.13
outdoor				2	0.16
				5	0.21
				6	0.17
				13	0.12
Spain	3	0.18	0.01	0	0.30
1986				4	0.20
outdoor				7	0.08
				14	0.03
Spain	3	0.18	0.01	0	0.20
1986				4	0.170
outdoor				7	0.07
				14	0.04
Spain	3	0.18	0.01	0	0.28
1986				4	0.17
outdoor				7	0.07
Spain	1	0.15	0.01	2	0.52
1988				7	0.04

Country year	Application			PHI, days	Residue, mg/kg
	No.	kg ai/ha	kg ai/hl		
indoor				11	0.16
Spain	1	0.15	0.01	2	0.38
1988				7	0.25
indoor				11	0.45
				15	0.17
Spain	1	0.15	0.01	2	0.39
1988				7	0.15
indoor				11	0.15
				15	0.21
Spain	1	0.15	0.01	2	0.34
1988				7	0.38
indoor				11	0.11
				15	0.15

* No information on year. Trials were submitted by Spain.

Tomatoes. The results of trials from Denmark and Germany are summarized in Table 9.

Table 9. Residues of fenpropathrin in tomatoes.

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
Denmark	5% EC	1		0.0075	1	0.08	42
1984					3	0.08	
Indoor					5	0.17	
					7	0.09	
					14	0.05	
Germany	10% EC	3	0.08	0.009	0	<0.01 <0.01	85;1*
1983					1	<0.01 <0.01	
Indoor					3	<0.01 <0.01	
					5	0.01 0.01	

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
					7	0.03 0.03	
Germany	10% EC	3	0.08	0.009	0	0.26 0.21	86;1
1983					1	0.28 0.22	
Indoor					3	0.19 0.15	
					5	0.51 0.41	
					7	0.07 0.06	
Germany	10% EC	3	0.08	0.009	0	0.12 0.12	87;1
1983					1	0.18 0.19	
Indoor					3	0.16 0.17	
					5	0.13 0.14	
					7	0.22 0.23	
Germany	10% EC	3	0.08	0.009	0	0.11 0.11	88;1
1983					1	0.09 0.09	
Indoor					3	<0.01 <0.01	
					5	0.02 0.02	
					7	0.03 0.03	
Germany	10% EC	3	0.08	0.009	0	0.08 0.07	89;1
1983					1	0.21 0.18	
Indoor					3	0.17 0.15	
					5	0.08 0.07	
					7	0.08 0.07	
Germany	10% EC	3	0.08	0.009	0	0.73 0.73	90;1
1983					1	0.56 0.56	
Indoor					3	0.58 0.58	
					5	0.47 0.47	
					7	0.22 0.22	
Germany	10% EC	3	0.08	0.009	0	0.19 0.15	91;1
1983					1	0.29 0.23	
Indoor					3	0.46 0.37	
					5	0.49 0.39	
					7	0.30 0.24	

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
Germany	10% EC	3	0.08	0.009	0	0.19 0.15	92;1
1983					1	0.17 0.13	
Indoor					3	0.13 0.10	
					5	0.13 0.10	
					7	0.08 0.06	
Hungary	10% EC	1	0.05	0.005	0	0.01	18
1984					1	0.07	
Indoor					2	0.03	
					3	0.03	
					4	0.04	
					7	0.02	
Hungary	10% EC	1	0.05	0.005	0	0.04	19
1984					1	0.05	
Indoor					2	0.01	
					3	0.07	
					4	0.04	
					7	0.02	
Japan	10% EC	3	0.25	0.01	1	0.58	34
1986					3	0.60	
Indoor					7	0.58	
Japan	10% EC	5	0.25	0.01	1	1.1	34
1986					3	0.86	
Indoor					7	0.74	
Japan	10%EC	3	0.25	0.01	1	0.42	34
1986					3	0.37	
Indoor					7	0.25	
Japan	10% EC	5	0.25	0.01	1	0.67	34
1986					3	0.60	
Indoor					7	0.55	
Germany	10% EC	3		0.009	0	0.15	97
Outdoor					1	0.23	

Country, Year	Application				PHI, days	Residue, mg/kg	Ref.
	Form.	No.	kg ai/ha	kg ai/hl			
**					3	0.37	
					5	0.39	
					7	0.24	
Germany	10% EC	3		0.009	0	0.15	97
Outdoor					1	0.13	
**					3	0.1	
					5	0.1	
					7	0.06	
Germany	10% EC	3		0.009	0	0.12	97
Outdoor					1	0.19	
**					3	0.17	
					5	0.14	
					7	0.23	
Germany	10% EC	2		0.009	0	<0.01	97
Outdoor					1	<0.01	
**					3	<0.01	
					5	0.01	
					7	0.03	
Germany	10% EC	2		0.009	0	0.21	97
Outdoor					1	0.22	
**					3	0.15	
					5	0.41	
					7	0.06	
Denmark	10% EC	1		0.0075	1	0.08	97
Outdoor					3	0.08	
**					5	0.17	
					7	0.09	
					14	0.05	
Italy** Outdoor	10% EC	2		0.015	21	0.01	97

* From two independent laboratories

** No information on year. Trials used and submitted by Spain.

Animal transfer studies

Cattle. Lactating dairy cattle were administered fenpropathrin at rates equivalent to 25, 75 and 250 ppm in the feed based on the daily average food consumption of 18.54 kg/cow. The fenpropathrin was technical grade of 92.5% purity, unlabelled, and administered in gelatin capsules. There were four cows in each group and two as controls. Milk samples were taken periodically up to 28 days, when three animals of each group and one of the controls were slaughtered. Administration of fenpropathrin then ceased and the remaining animals were killed after a three-day period on untreated feed. Samples of liver, kidney, fat and muscle were collected for analysis from all of the animals. Residues of fenpropathrin itself in the milk reached a plateau after three days. Average residues in the whole milk of the four cows of each group were 0.04, 0.17, and 0.33 mg/l for the three dose levels. On the 28th day, these levels were 0.04, 0.13 and 0.32 mg/l. At the end of the three-day depuration period, residues had fallen to <0.01, 0.02 and 0.04 mg/l for the three levels. In a bulk pasteurized milk sample from the high-dose cows on the 26th and 27th days containing 0.25 mg/l in the whole milk residues were largely confined to the cream where the level reached 3.7 mg/l.

Levels of fenpropathrin in the tissues at terminal slaughter and after the depuration period (average of three cows) are shown in Table 10.

Table 10. Levels of fenpropathrin in cattle tissues following oral ingestion

Level in feed	Residues, mg/kg			
mg/kg	muscle	kidney	liver	fat
After 28 days on treated feed				
25	0.02	0.03	<0.01	0.33
75	0.06	0.04	<0.01	1.0
250	0.20	0.16	0.01	3.8
After three-day depuration period				
25	0.01	0.01	<0.01	0.31
75	0.10	0.06	<0.01	0.83
250	0.12	0.14	<0.01	2.6

Samples of milk and tissues taken from the cows at the highest feeding level killed after 28 days were analyzed for TMPA, PBacid and PBacid-glycine; measurable levels were not found in the lower-dose groups. None of these metabolites was detected in milk or muscle. Average levels in kidney were TMPA 0.1, PBacid 0.07 and PBacid glycine 0.04 mg/kg. Corresponding levels in the liver were 0.03, 0.09 and 0.02 mg/kg. In samples taken from the single cow killed after three days depuration the values were 0.07, 0.09 and 0.04 mg/kg in kidney and <0.02, <0.02 and 0.02 mg/kg in liver, demonstrating rapid losses from liver but less so from kidney. Fat analyses were not carried out as previous studies had shown that over 90% of the residues in fat were present as the parent compound (Fujie *et al*., 1986a).

The residues in whole milk at the plateau level were approximately 0.15% of the level in the feed and were reasonably consistent between the three dose levels. If cows were fed on a diet consisting entirely of dried apple pomace at the residue level found in the processing studies of 45 mg/kg, it could be argued that the maximum level in milk would be 0.07 mg/l. Assuming that these residues would all be present in the fat and that the fat content of the milk would be 4%, such a level would be equivalent to 1.75 mg/kg in the milk fat.

Residues in body fat at the end of the study were about 1.4% of the level in the feed. Using the apple pomace figure of 45 mg/kg, it is reasonable to conclude that residues in meat fat would not exceed 0.6 mg/kg.

Residues in meat (muscle) were about 0.08% of the feed level so that animals fed on apple pomace at 45 mg/kg would not be expected to have more than 0.05 mg/kg in muscle or kidney.

It should be recognized, of course, that it is unlikely that animals would be fed on diets consisting exclusively of apple pomace so that these estimated upper limits are very unlikely to be seen in practice, especially since the apple pomace figure itself is probably a considerable overestimate of what would actually occur.

Hens. Laying hens were fed diets containing unlabelled fenpropathrin of 94.5% purity at nominal levels of 2.5, 7.5 and 25 mg/kg of the technical product for a period of 28 days. Actual average contents were 2.45, 7.10, and 23.6 mg/kg. There were 20 hens in each treatment group including the control animals. Eggs were collected daily and those from days 1, 2, 4, 7, 21 and 28 were analyzed as whole eggs minus shell. All the hens were killed after 28 days and composite samples of liver, gizzard, fat and muscle were prepared for analysis.

In all analyses the lower limit of determination for fenpropathrin was 0.01 mg/kg. Residues in all tissues except fat were below this level at 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 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.

Both tissues and eggs from hens in the highest dose group were also analyzed for TMPA, PBacid, and PBacid-glycine. The limit of determination for each compound was 0.02 mg/kg and none of the metabolites was found in eggs or tissues except TMPA at 0.04 mg/kg and PBacid-glycine at 0.02 mg/kg (average of replicates), both in the liver. Fat samples were not analyzed for metabolites, since they were considered unlikely to have accumulated measurable levels of these hydrophilic compounds (Fujie *et al*., 1986b).

It is unlikely that poultry would receive feed items containing appreciable residues of fenpropathrin with the possible exception of cotton seed meal. From Table 11 it may be seen that with a maximum level of 1 mg/kg in raw cotton seed, it is unlikely that residues in meal would exceed 0.1 mg/kg. In the present study the level in fat reached only 0.02 mg/kg even at a total feed level of 2.5 mg/kg so that measurable residues would not be expected in either the meat or eggs of hens fed on cotton seed meal.

FATE OF RESIDUES

Nomenclature of metabolites (*see also* Figure 1 on following page)

3-phenoxybenzoic acid (PBacid)

3-phenoxybenzyl alcohol (PBalc.)

3-phenoxybenzaldehyde (PBald.)

2,2,3,3-tetramethylcyclopropanecarboxylic acid (TMPA)

2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylic acid (TMPA-CH_2OH)

5-hydroxymethyl-6,6-dimethyl-3-oxabicyclo[3.1.0]hexan-2-one (TMPA-CH_2OH-lactone)

In animals

Goats. Fenpropathrin labelled with ^{14}C in the benzyl ring or the C-1 position of the cyclopropyl ring was administered to lactating goats at a nominal rate of 50 mg/kg feed/day for five days. The animals were slaughtered and samples of kidney, liver, heart, loin and rear leg muscle and omental and perirenal fat taken within 4 hours of the last dose. The goats were milked every morning and evening and all milk was reserved for analysis.

The total radioactivity 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 the cyclopropyl label and 0.25 mg/l for the benzyl label, all expressed as fenpropathrin. Less that 3% of the activity in the milk was found in the butterfat. Of the total administered activity of 250 mg of fenpropathrin, 0.73 mg of fenpropathrin equivalent was recovered in the milk from the benzyl label and 0.43 mg from the cyclopropyl label (averages for the two goats in each treatment), so that milk represented only a minor route of excretion.

Figure 1. Metabolism and metabolic degradation of fenpropathrin in soils and plants.

L, photodegradation; S, soil; P, plant

*, ■, ⅄, 14C-labelled positions

The retention in body tissues was only moderate: a total of 2.8 mg of fenpropathrin equivalents for the benzyl label and 3.7 mg for the cyclopropyl label as compared with the total administered amount of 250 mg. 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 for both labels. Muscle levels were in the range of 0.02-0.04 mg/kg (Ku and Doran, 1990a).

Between 20% and 40% of the radiocarbon in the milk from animals receiving the benzyl 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. 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, 56 75% of the activity was associated with the parent material with moderate amounts of TMPA and its hydroxymethyl (<0.002-0.003 mg/kg), carboxy (<0.002-0.003 mg/kg) and lactone (<0.002-0.003 mg/kg) derivatives. 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. In the case of the benzyl label, most of the activity was in the form of PBacid (kidney 0.05-0.08 mg/kg; liver 0.03-0.06 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 (Ku and Doran, 1990a).

Cows. Two lactating cows were fed on a diet containing 0.11 mg/kg of fenpropathrin labelled in the benzyl ring with ^{14}C. The cows were milked twice daily and all milk, faeces and urine monitored for radioactivity. After 21 days the animals were slaughtered and radioactivity measured in muscle, fat and liver.

It was found that the equilibrium between intake and excretion in the urine and faeces was reached after about 5 days and that excretion thereafter averaged 96% of the amount ingested. No radioactivity was detected in any of the muscle, blood or fat samples; the limit of determination varied from 0.004 to 0.008 mg/kg of fenpropathrin equivalent. Residues in the milk samples were extremely small and very difficult to measure. The author estimated them to be between 0.0002 and 0.0003 mg/l of fenpropathrin equivalent (Crayford, 1975).

Hens. Fenpropathrin, labelled in either the cyclopropyl or the benzyl ring was administered to laying hens daily for 10 days. The product was given in the form of capsules at a nominal rate of either 0.5 or 5 mg/kg body weight. There were four treatment groups of 10 hens in each group and two control groups. Eggs were collected every morning and evening and excreta every morning. The hens were all killed within four hours of the last dose and kidneys, liver, heart, gizzard (and contents), ovaries, muscle and skin were retained for analysis.

The total doses for the four groups over the ten-day period were as follows:

Low dose, benzyl label 78 mg
High dose, benzyl label 820 mg
Low dose, cyclopropyl label 75 mg
High dose, cyclopropyl label 803 mg

The recovery of total radioactivity from excreta, eggs and tissues was between 75 and 82% 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. At about the 6th or 7th day of the study residue levels in the eggs reached a plateau of about 0.023 and 0.22 mg/kg fenpropathrin equivalent for the two doses of the benzyl label and about 0.05 and 0.4 mg/kg for those of the cyclopropyl label. In the body tissues the highest levels of radioactivity were found in the kidney and gizzard followed by the liver, showing about 3-4 mg/kg for the two high doses in the kidney and gizzard and 1.5-3 mg/kg in the liver without major differences between the two labels. Levels in the low-dose hens were about one-tenth of those in the higher dosed birds (Ku and Doran, 1990b).

The products associated with the activity in the solvent extracts from the benzyl label were mainly PBacid, 4-OH PBacid and its glycine conjugate, and 3-hydroxybenzoic acid (3-OH-Bacid), which was not encountered in the other animal studies. Only negligible amounts of the activity in the liver and kidney remained as the parent (1-2%) but fat residues contained nearly 50% of the unchanged compound. The occurrence of 3-OH-Bacid in the kidney, accounting for 35% of the kidney activity, demonstrates that cleavage of the ether linkage of PBacid must have occurred. It also appears that 4′-hydroxylation of PBacid occurred readily in the liver and kidney. Nevertheless, even in the case of the high-dose birds, residues of any single component seldom exceeded 1 mg/kg except 3-OH-Bacid and 4′-OH-PBacid in the kidneys. The pattern of distribution of activity among the metabolites was similar in the case of the cyclopropyl labelled group except that the main metabolites, as would be expected, were TMPA and its derivatives. There were mainly TMPA CH2 OH the carboxy compound (TMPA COOH), and TMPA CH_2OH lactone. Fenpropathrin was only a very minor component of the residues in the liver, kidney, heart and meat but reached 63% of the residues in fat. In the high-dose group with this label no component exceeded 1 mg/kg except TMPA itself in the kidney.

As would be expected from the comparatively high proportion of 3-OH-Bacid in the kidney, this metabolite accounted for a major proportion of the activity in the excreta from the benzyl-labelled group and this together with 4′-OH PBacid accounted for nearly 65% of the excreted activity on the eighth day. Fenpropathrin constituted only about 10% of the excreted activity. Most of the remainder was made up of PBacid and its glycine conjugate with small amounts of OH-fenp. In the case of the cyclopropyl label, TMPA and its derivatives (TMPA CH^2OH, TMPA COOH and TMPA CH_2OH lactone) accounted for half of the activity, the rest being made up of the parent and two of its hydroxylated derivatives, 4′-OH-fenp. and fenp.-CH_2OH (Ku and Doran, 1990b).

In plants

The degradation of fenpropathrin in plants has been studied in cotton, tomatoes, beans, and apples. The compounds involved are shown in Figure 1. The general pattern of degradation in all the plant studies has been rupture 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). The position is complicated, however, by subsequent hydroxylation either of these fragments or of the intact molecule. Thus, TMPA can give rise to 2 hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylic acid (TMPA-CH_2OH) and 5-hydroxymethyl-6,6-dimethyl-3-oxabicyclo[3.1.0]hexan-2-one (TMPA-CH_2OH lactone). PBacid can be hydroxylated at the 4′ position and the parent molecule at various positions on the phenoxy ring to produce, for example, Æ-cyano-3-(2′- or 4′-hydroxyphenoxy)benzyl-2,2,3,3-tetramethylcyclopropanecarboxylate (2′ or 4′-OH-Fenp.).

The results of the studies on individual plants are summarized under separate headings below.

Cotton. The degradation of fenpropathrin in cotton was shown to follow the familiar pattern of ester hydrolysis and conjugation of the resulting PBacid and TMPA. In these studies, cotton plants were grown either in the greenhouse in the UK or outdoors in boxes in Spain. Known amounts of fenpropathrin (740-2000 ìg), labelled with ^{14}C in either the benzyl or cyclocyclopropyl rings were applied at various times to the leaves or bolls of the plants (total amounts 2000 and 4690 ìg). In separate outdoor experiments in Spain, only soils were treated in order to examine uptake.

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, from the evidence presented, 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 cyclocyclopropyl label) the total remaining activity included 70% parent in the case of the cyclocyclopropyl label and 55% parent in the case of the benzyl label. Most of the remaining activity was probably 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 showed only extremely low uptake of radioactivity, demonstrating very limited tendency for translocation. See Table 11 (Hitchings and Roberts, 1977).

Table 11. Analyses of cotton and soil treated with [^{14}C]-fenpropathrin.

Sample	Radioactivity, mg/kg fenpropathrin equivalent			
	benzyl label		cyclopropyl label	
	extracted	unextracted	extracted	unextracted
soil at applicn.	0.86	0.03	0.78	0.034
soil at harvest	0.02	0.09	0.1	0.048
cotton leaves	0.002	0.003	0.014	0.004
cotton stems	0.004	0.01	0.017	0.01
cotton boll case	0.01	0.02	<0.01	0.01
cotton lint	-	0.01	-	0.02
cotton seed kernel	-	0.03	-	0.05
cotton seed hull	-	0.01	-	0.01

Tomatoes. In studies in California by the Chevron Chemical Company, tomato plants were treated four times with fenpropathrin (0.224 kg ai/ha) labelled with ^{14}C in either the cyclopropyl or the benzyl ring. Fruit and leaves were extracted at harvest (PHI 19 days) with a variety of solvents and the components of the residue characterized. In the fruit the residue was too low to allow full characterization, but some two thirds was 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 residue. The main metabolites reported in the case of the cyclocyclopropyl label were conjugates of TMPA and hydroxymethyl-TMPA

(TMPA-CH_2OH), part of which was also conjugated (Chen and Abell, 1985, 1986b).

Beans. Somewhat similar results were reported by the same authors for pinto beans. The residue after a PHI of 15 days in the beans themselves was too low (0.07 mg/kg) for full characterization; some 93% of the label remaining in the plants at harvest was found in the leaves. In the leaves, 46-47% of the remaining activity consisted of the parent compound. In the case of the benzyl label the main metabolites were conjugates of PBacid and 4′-OH-PBacid, together with conjugates of PBalc. and PBald. In the case of the cyclocyclopropyl label, the main metabolites were conjugates of TMPA and its two stereoisomeric mono-hydroxy derivatives (TMPA-CH_2OH) (Chen and Abell, 1985, 1986c).

Apples. In a study on apples samples from young trees were analyzed at harvest, 14 days after the last of three treatments at the comparatively high rate of approximately 0.448 kg/ha. Practically all of the residue found in the fruit (92-94%) was present as the parent compound. The parent compound was also the major component found in the rest of the plant (61-66% of the total activity). In apples the pattern of metabolites was more complex than in the preceding crops, although few individual metabolites accounted for more than 2% of the total residue. The most prominent metabolite from the benzyl label was conjugated PBalc. and from the cyclopropyl label TMPA, together with the usual complement of hydroxylated derivatives. Both labelled compounds also yielded small amounts of hydroxylated derivatives of intact fenpropathrin (2′- or 4′-OH-fenp.), existing in both free and conjugated forms (Chen and Abell, 1985, 1986a.)

Photodegradation on leaf surfaces. The four top leaves of small potted mandarin orange plants were treated with fenpropathrin labelled in the cyano group or the cyclopropyl or benzyl ring, to produce a deposit of 1.1 ì g/cm^2. After a 14-day exposure to sunlight the leaves were assayed for radioactivity. There were substantial evaporative losses but over 80% of the remaining activity (approximately 40% of that applied) was still in the form of the unchanged parent. There were little more than traces of degradation products, among which were identified CONH2 -fenp. and PBacid with a small amount of Æ-cyano-3-hydroxybenzyl 2,2,3,3-tetramethylcyclopropanecarboxylate. None of these reached more than 0.3% of the applied activity.

It is noteworthy that approximately 40% of the activity remaining on the leaves could be extracted with a surface wash; the rest had penetrated into the leaf (Takahashi *et al*., 1983, 1985).

Metabolites in samples from supervised field trials. Some of the fruit samples from supervised field trials were analyzed for PBacid and TMPA. The results are shown below in Table 12. As will be seen, PBacid did not reach the level of determination in any of the samples analyzed. TMPA slightly exceeded detectable levels in pears, but still constituted only a negligible proportion of the total residues. These results confirm that residues in these crops resulting from treatment with fenpropathrin are adequately determined by analysis for fenpropathrin alone.

Table 12. The occurrence of metabolites in fruits treated with fenpropathrin; residues in mg/kg.

Parent	PBacid	TMPA	Reference
Apples			
3.6	<0.02	<0.02	24
1.5	<0.02	<0.02	24
3.7	<0.02	<0.02	24

Parent	PBacid	TMPA	Reference
2.8	<0.02	<0.02	24
2.4	<0.02	<0.02	24
Pears			
1.0	<0.02	0.04	37
3.2	<0.02	0.07	37
1.1	<0.02	0.03	22
2.9	<0.02	0.06	22
1.8	<0.02	0.04	22
2.4	<0.02	0.04	22
Grapes			
3.1	<0.02	<0.02	25
1.1	<0.02	<0.02	25
1.1	<0.02	<0.02	25
2.6	<0.02	<0.02	25
1.1	<0.02	<0.02	25
1.4	<0.02	<0.02	25
1.0	<0.02	<0.02	25
5.6	<0.02	<0.02	25

Summary of degradation in crops. The above studies demonstrate that fenpropathrin itself 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 in both fruits and leaves did not differ greatly from those in animals.

This conclusion is supported by the data collected in some of the supervised field trials for PBacid and TMPA which were either below the limit of determination of the method or negligible compared with the levels of parent fenpropathrin. It is therefore considered that crop residues are described adequately by defining them as the parent product alone.

It is also evident that any uptake of residues from the soil is too slow for detectable residues to occur in suceeding crops, especially in view of the comparatively short persistence of the compound in soils as shown in the following section.

In soil

Fenpropathrin readily disappears from soil by two main mechanisms, biodegradation and photochemical degradation of surface deposits. It is relatively immobile because of its strong adsorption and its comparatively short life in normal agricultural soils. The nomenclature of the compounds involved in the degradation pathways is as in Figure 1 and the above text.

Degradation. In the first of the soil degradation studies, 5-6 g of a single soil were treated with fenpropathrin labelled either in the cyano group, the C-1 position of the cyclopropyl

ring or uniformly in the benzyl ring. The soils were then perfused with nutrient solutions buffered to pH 7.0 for 148 or 208 days depending on the label. Liberated carbon dioxide was collected periodically for estimation of radioactivity. The soils and perfusates were solvent-extracted at the end of the study and the extracts analyzed for radioactivity and fractionated by TLC. The unextracted activity was determined by combustion.

Table 13 shows the recoveries obtained from the three phases expressed as a percentage of the activity applied (average of two duplicate perfusions).

Table 13. Recovery of radioactivity in soil perfusion studies.

Phase	% of applied activity		
	CN label	Cyclopropyl label	Benzyl label
Carbon dioxide	36.5	19.5	4.7
Perfusate	9.0	10.5	8.0
Soil extract	14.0	32.5	34.0
Combusted soil	7.0	17.2	22.1
Total recovery	66.5	79.7	68.8

As will be seen, the production of labelled carbon dioxide was greatest from the cyano label and least from the benzyl, indicating the relative readiness of the different parts of the molecule to mineralize although the soil with the cyano label was perfused for 208 days compared with only 148 days for the other two labels. Much of the difference in the production of carbon dioxide was made up by the difference in the total amount of activity retained in the soils.

In the soil extracts from the cyclopropyl label, the parent compound was the main component with small amounts of tetramethylcyclopropanecarboxylic acid (TMPA) and unidentified polar compounds and, in one of the perfusions, the amide of fenpropathrin ($CONH_2$-fenp.). No amide was found from the cyano label. In the case of the benzyl label, the liberation of carbon dioxide was much slower and the rates were erratic. Neither $CONH_2$-fenp. nor PBacid was detected in the soils. In the perfusates, the main components isolated were the parent and unidentified hydrophilic compounds (Noble, 1976).

The degradation of fenpropathrin labelled in either the benzyl or cyclopropyl rings was further studied in a sandy clay soil (Brenes) or a clay (Los Palacios) from Spain or a sandy loam from the UK (Leiston). The soils were treated in glass jars at a rate of 2.86 mg/kg and kept in the dark at a temperature between 23 and 25°C. The moisture contents of the soils were maintained at their original values by the periodic addition of distilled water but in the case of Brenes soil the study was conducted at two moisture levels, 6 and 16%. An additional study using Brenes soil was set up to determine volatiles. Benzyl-labelled fenpropathrin was added to the soil at a rate of 2.5 mg/kg at a moisture content of 19.6%. The whole system was aerated continuously and volatiles and carbon dioxide were absorbed in traps.

In a separate study Leiston soil, in conical flasks, was treated at the rate of 2.5 mg/kg with benzyl- or cyclopropyl-labelled fenpropathrin and stored at 25°C under distilled water with occasional nitrogen purging to maintain anaerobic conditions.

In the aerobic study soils were sampled at 4, 8 and 16 weeks and in the volatiles study at 26 weeks. In the anaerobic study, the soils were sampled at intervals of 32, 60, 120 and 160 days. In all cases they were extracted with acetonitrile/water (7:3). In the volatiles

study, the aspirated air was sampled at intervals throughout the experiment. Unextracted residues were determined in the soils after the solvent extraction. Degradation products were identified by co-chromatography, the carboxylic acids after methylation.

In the aerobic soil after 16 weeks, by far the most important component of the residue was the parent compound but the proportion of the original ^{14}C remaining varied greatly, ranging from 66% in Brenes soil at 6% moisture to 10.2% in Leiston at 16% moisture. Although the moisture capacities of the soils were not stated it would appear that the main factor influencing the degree of degradation was the dryness of the soils i.e. the moisture content in relation to the moisture capacity. 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).

In the drier soils after 16 weeks the most prominent metabolite was PBacid in the case of the benzyl label and TMPA in the case of the cyclopropyl label. The other two metabolites found were $CONH_2$-fenp. and COOH-fenp., but these were scarcely detectable in the soils with high moisture (Brenes at 16% moisture and Leiston). In those two soils PBacid was also much lower but unextracted activity was much higher than in the drier soils, suggesting either entrapment in the soil organic matter or possibly mineralization and incorporation in the soil organic matter pool.

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.

The main effect of imposing anaerobic conditions on the soils was to slow the rate of ester hydrolysis to some extent and to impede the subsequent degradation of PBacid and TMPA which tended to accumulate. This indicates that their subsequent degradation in aerobic conditions is essentially oxidative (Roberts and Standen, 1976).

In reviewing evidence for the further degradation of metabolic products, Miyamoto concluded that PBacid, as derived from the degradation of fenpropathrin and other pyrethroids, is rapidly and completely degraded to CO_2 under aerobic conditions. There is somewhat comparable evidence for the degradation of TMPA although it is not quite so extensive as for PBacid. Anaerobic conditions impeded the degradation of both compounds (Miyamoto, 1980).

Two fresh Japanese soils, a sandy clay loam (Azuchi) and a light clay with 15% organic matter (Kodaira), were studied under three different conditions: natural aerobic and anaerobic soil, and autoclaved aerobic soil. The soils were treated with fenpropathrin labelled either in the benzyl ring or at C-1 in the cyclopropyl ring to produce a concentration of 1 mg/kg, and then incubated at 25°C. In the aerobic study with the unsterilized soil incubation was continued for 24 weeks, but in the other cases the incubation period was only 8 weeks. In the aerobic soils the moisture content was maintained at a moisture capacity of approximately 40%. Arrangements were made to trap liberated CO_2 in all cases.

Soil extracts (three extractions with methanol) were assayed at various times during the studies. Some of the soils were retained after extraction for examination of the unextracted activity.

It was found that under aerobic conditions the half-life of fenpropathrin was 11 and 17 days on the Azuchi and Kodaira soils respectively, and after 24 weeks the level of fenpropathrin had declined from 1 mg/kg to 0.025 and 0.040 mg/kg respectively (*see also* Mikami, 1983, for the basis for the calculation of these values). After 8 weeks approximately

0.85 mg/kg remained in the same two soils under anaerobic conditions and approximately 0.93 mg/kg under sterilized conditions. The corresponding figures for the natural aerobic soils after 8 weeks (mean of the two labels) were 0.05 and 0.10 mg/kg, so that anaerobic conditions retarded the degradation of fenpropathrin to a much greater degree than had been observed in anaerobic aquatic soil (Roberts and Standen, 1976). Sterilization also greatly retarded degradation, demonstrating the importance of biological processes.

Whilst 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 soils, both aerobic and anaerobic. In the aerobic soils with the cyclopropyl-labelled compound the main components of activity were the parent fenpropathrin, dephenyl-fenpropathrin (desph-fenp.), 4′-OH-fenpropathrin, and small amounts of $CONH_2$-fenp. and COOH-fenp. There were also indications of very small amounts of unidentified products. In the case of the benzyl label very low levels of PBacid were observed during the first 4 weeks. Unextractable residues at the end of the study reached 44-45% of the added activity 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.

Under anaerobic conditions there was a similar pattern of metabolites but in a much smaller proportion as compared with the parent. Correspondingly, bound activity was also at a much lower level. There was no evidence of accumulation of PBacid, as found by Roberts and Standen.

A major part of the lost activity in the aerobic soils was recovered as labelled CO_2 in fairly similar amounts from both labels: an average between the two of 42% in the Kodaira soil and 55% in the Azuchi.

It was shown that part of the bound activity in the soils could be liberated as CO_2 by subsequent incubation of the extracted soils with fresh untreated soil but the extent of this differed greatly between the two soils. Slightly more than 20% of the bound activity was liberated from the cyclopropyl-labelled Azuchi soil whereas the corresponding figure for the Kodaira soil was only about 3% (Mikami *et al.*, 1983a).

The aerobic part of the above study (Mikami *et al.*, 1983a) was repeated at shorter time intervals. The same soils and conditions were used. The incubation period was only 30 days and soils were extracted 1, 3, 7, 17 and 30 days after treatment.

The half-lives were 30 and 33 days in the Kodaira and Azuchi soils respectively, some 2-3 times as long as in the initial study. The same degradation products were found, i.e. CO_2, desph fenp. and small amounts of 4′-OH-fenp., $CONH_2$-fenp., COOH-fenp. and PBacid. Within the short time-scale of this study the dephenyl compound increased with time in both soils. The same was true of the 4′-OH-fenp., but much less so in the Azuchi soil. The authors observed that the appreciably longer degradation time was probably due to the fact that in this study the soils had been stored for a period of 8 months and had probably suffered a reduction in bioactivity (Mikami and Sakata, 1984).

A study of the fate of 1-cyclopropyl-labelled fenpropathrin was carried out at lower temperatures in a laboratory study in The Netherlands (Van Dijk and Vonk, 1989). The soils used were a humic sand taken from Wageningen and a loam soil from Lelystad which was calcareous. Samples of 50 g of the pre-incubated soils were placed in conical flasks and fenpropathrin was added to produce a final concentration in the soils of 2.5 mg/kg. The flasks were fitted with soda lime traps to assess the liberation of labelled CO_2 and incubated at 15°C in the dark for 41 weeks. Duplicate samples were taken at intervals and extracted with methanol.

The half-life of fenpropathrin was estimated to be 6 weeks in the loam soil but

longer than 36 weeks (the estimate was 40 weeks) in the humic sand, at which point some 57% remained. In the loam soil the main conversion products were CO_2 (about 45% after 41 weeks) and unextractable bound residues (28% after 41 weeks). After 36 weeks most of the extractable fraction consisted of unchanged fenpropathrin (average 6.8%) and minor amounts of 4′-OH-fenp. and desph-fenp. (average 0.8 and 0.4% respectively). The main conversion products in the humic sand after 36 weeks were carbon dioxide (3 and 24% of the applied activity for the duplicates) and unextractable residues (14 and 16%). Most of the extractable fraction consisted of unchanged fenpropathrin but there were small amounts of 4′-OH-fenp. and desph-fenp., averaging approximately 5 and 3% respectively of the applied dose.

In both soils it was evident that the maximum levels of these two metabolites had occurred before the 36th week and in some cases well before. The same was true of the unidentified metabolites whose values seem to have been highest at 6-13 weeks and fell from 3.3% at 13 weeks to 0.7% in the loam and from 3.2% at 6 weeks to 0.9% in the sand.

The authors commented that in their work most of the pesticides they had studied were degraded only slowly in the humic sand, and noted that the soil humidity in their studies was considerably lower than that employed by Mikami *et al*. (1983a). The temperature was also a good deal lower (15°C compared with 25°C in the Mikami study). They cite another of their studies (Vonk and van Dijk, 1988) in which the half-life of fenpropathrin in the loam soil at 10°C was found to be 30 weeks. This marked dependence on temperature in the loam soil was less evident in the sand.

A study of soil degradation was carried out according to the EPA protocol, Section 162-1 of the pesticide assessment guidelines. Fenpropathrin labelled in the benzyl ring 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 ì g/g soil; the measured concentration immediately after the addition was 10.2 ì g/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 unextracted activity.

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 unextractable residues for 17.8%. During the whole course of the study the maximum levels of the metabolites were 1.25% PBacid, 0.21% $CONH_2$-fenp., 0.55% desph-fenp., 0.19% 4′-OH-fenp. and 0.39% COOH-fenp., all in terms of the initial dose. In addition there were maxima of 0.07, 0.28, 0.34 and 0.16% of unidentified products. The ^{14}C mass balance for the whole period ranged from 98.7% to 107.1% with a mean of 102.4% 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. The authors considered that their results were in good accord with those of Mikami *et al*. (1983a) (Cranor, 1990).

The degradation of fenpropathrin in a loam soil from California was studied in aerobic conditions for 30 days followed by anaerobic conditions for 60 days. The fenpropathrin was labelled in the benzyl ring and was added to the soils to produce a nominal concentration of 10 mg/kg but the mean concentration after dosing was determined to be 12.1 mg/kg. The fenpropathrin was added to the pre-incubated soils which were then incubated at 25°C for a further 30 days. The soil moisture level was adjusted to approximately 75% of the field capacity and maintained at that level by periodic additions. The containers were equipped for the collection and determination of CO_2. After 30 days of aerobic incubation, anaerobic conditions were initiated by adding a small amount of glucose to the soils, covering them with water and flushing the flasks with nitrogen. The soils were extracted periodically with methanol and unextracted activity was determined by combustion analysis.

At the end of the aerobic phase, 85.1% of the activity remained as the parent compound. PBacid (2% of the initial dose), COOH-fenp. (1.3%) and 4′-OH fenp. (0.3%) were also found in the methanol extract. CO_2 was found to the extent of 0.6% of the applied dose. Desph-fenp. and $CONH_2$-fenp. were not detected. (These results were compared with those from another aerobic study which was continued for a year, in which 88% of the parent compound remained after the first 30 days and 8.9% was lost as CO_2. $CONH_2$-fenp. (0.12%), desph-fenp. (0.4%), a trace of 4′-OH-fenp (0.04%) and COOH-fenp. (0.26%) also occurred).

After the 60-day anaerobic phase, 66.0% of the initial dose remained as parent compound, 11.5% as PBacid, 6.4% as COOH-fenp. and 0.7% was liberated as CO_2. $CONH_2$-fenp. and 4′-OH-fenp. were present only as minor metabolites (<1.0%). Unextractable residues at this point were 8.0% of the applied dose and 15.7% of the applied activity was recovered in the supernatant water. The mean mass balance was 98.4 ± 2.8%.

Half-lives of fenpropathrin in the aerobic phase of the study were estimated to be 196 days and in the anaerobic phase 186 days.

Hence, as had been reported by Roberts and Standen in 1976, the main effect of imposing anaerobic aquatic conditions was to impede the degradation of metabolites, especially PBacid. It is not possible to infer the position regarding TMPA in this study owing to the position of the label (Daly and Williams, 1990).

Photodegradation. Studies were carried out in Japan with fenpropathrin labelled in the cyano group, the benzyl ring or the 1-carbon position of the cyclopropyl ring and applied to thin-layer soil plates prepared according to the procedure of Helling and Turner (Science, 162, 1968, 562-3). The three soils were Kodaira light clay, Katano sandy loam and Azuchi sandy clay loam. The fenpropathrin was applied at a rate of 1.1 ì g/cm^2 and the plates exposed to natural sunlight for 10 days during the month of September. The water content of the soils did not fall greatly during the study. Dark controls were run at the same time.

During the study plates were withdrawn at intervals and the soils extracted with methanol/water (5:1) for the determination of extractable activity and TLC fractionation. Unextractable residues were fractionated into activity associated with fulvic acid, humic acid and humins.

Under irradiation, the half-lives of the CN-labelled fenpropathrin were 1, 4 and 5 days in the Kodaira, Azuchi and Katano soils respectively. The fenpropathrin left in the soils at the end of the 14-day period (averages for the three labels in each soil) amounted to 5.1, 29.4 and 32.9% of the amounts applied. The corresponding figures in the dark controls were 74, 85 and 96%; there was insufficient degradation for half-lives to be estimated.

The main degradation product under irradiation with all three labels was $CONH_2$-fenp. 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. An exception was PBacid, especially on the Katano soil where it reached a maximum of 11.4% of the total applied activity after 7 days. On that soil "others" and unextracted residues were higher than on the other two; the unextracted activity was mainly associated with the fulvic acid fraction of the soil organic matter. Minor metabolites found were COOH-fenp., desph-fenp., Æ-carbamoyl-3-phenoxybenzyl alcohol (PM-amide) and 3-OH-Bacid. The last three did not occur in the dark controls.

The recovery of total activity in the dark controls was mostly just above 100% after 14 days, but gradually declined in the irradiated soils, presumably owing to mineralization of the labelled moiety. The losses shown in Table 14 were reported.

Table 14. Loss of activity (%) from irradiated soils after 14 days.

Soil	Loss of activity, %		
	Cyclopropyl label	CN label	Benzyl label
Kodaira	13.2	19.8	9.8
Azuchi	15.3	28.4	8.4
Katano	43.4	52.5	23.6

Clearly, loss was greatly enhanced by irradiation. It is evident that the nitrile carbon was the most susceptible followed closely by the 1-carbon of the cyclopropyl group. The benzyl group was evidently a more stable part of the molecule. It is clear that differences between the soils affected the rate of loss. The Katano soil was especially active owing, presumably, to the presence of photosensitizing substances. As shown in another part of this study, humic acid exerted a marked sensitizing effect when added to irradiated solutions of fenpropathrin in distilled water. It would seem likely that irradiation could play an important role in the degradation of surface deposits of fenpropathrin following spray applications which, in many cases, would be expected to remain on the soil surface for a large part of the season (Takahashi *et al*., 1983, 1985).

Adsorption. Attempts were made to determine adsorption coefficients by leaching treated soils but insufficient fenpropathrin passed through the columns for dependable estimates to be made. As an alternative approach, values may be deduced by using Briggs's equations linking the adsorption coefficient with the octanol-water partition coefficient (for fenpropathrin, $\log_{10} P_{ow} = 6.0$). The following Table was presented for two of Briggs's equations representing the lowest and highest values.

Table 15. Calculated values for adsorption coefficients of fenpropathrin.

Soil	Organic matter %	$\log_{10} P_{ow}$	K_d (Equation 2)	K_d (Equation 6)
Kodaira	15.3	6.0	500	2210
Azuchi	2.5	6.0	85	360
Katano	11.0	6.0	370	1590

Assuming that adsorption is confined essentially to the soil organic matter, the corresponding adsorption coefficient for organic matter (K_{om}) would have been (approximately) between 3300 (equation 2) and 14,400 (equation 6) (Mikami, 1983).

Leaching. A study was carried out with a sandy loam from the UK, packed into two glass columns and conditioned by slowly passing water through them until the effluent was clear. Fenpropathrin labelled in the benzyl or cyclopropyl ring was applied to the surfaces of the soil columns and the columns stored for 4 weeks. Two more columns were set up in the same way but not stored, and all four columns were leached at the rate of 0.25 ml/hour for 45 days, producing a total of 270 ml passing through each column, or a depth of about 17 cm. Samples of eluate that contained radioactivity were extracted with ethyl acetate, and extruded segments of soil taken at the end of the study were extracted with acetonitrile/water (7:3 v/v) for analysis.

The leachate from the stored column containing the cyclopropyl-labelled compound showed an appreciable level of activity amounting to 9.1% of that applied. This was shown by TLC to arise from approximately equal quantities of $CONH_2$-fenp. and TMPA.

In the soil columns, the major part of the radioactivity remained in the top 2 cm even in the stored soils. The total recoveries were 80-93% for the cyclopropyl and benzyl labels respectively in the unstored soils but these levels fell to 62 and 46% in the stored columns, indicating appreciable loss. The unextracted activity was between 41 and 47% except in the stored soil with the benzyl label; the low figure of 8% for this soil has no obvious explanation. The main metabolite in all cases was $CONH_2$-fenp., but except in the stored column with the cyclopropyl label the parent fenpropathrin constituted the major part of the residual activity.

It was concluded that neither fenpropathrin nor its degradation products were likely to leach through the soil under field conditions (Roberts, 1976).

A further study was carried out with fenpropathrin labelled either at the 1-cyclopropyl carbon or in the benzyl ring. There were 4 soils, Azuchi, Kodaira, Sapporo and Muko. The last had almost no organic matter or biological activity and consisted of 99% sand. The four soils were packed into columns to a height of 25 cm and the columns eluted with water until the eluate ran clear. More soil was treated with 1 mg/kg of the labelled fenpropathrin and placed on top of the columns either at once or after 4 weeks incubation at 25°C. During incubation provision was made for the determination of liberated CO_2. The columns were leached with a total of 1 litre of distilled water at a rate of 3 ml/hour for 14 days. From the dimensions of the columns, this would have been equivalent to a total depth of approximately 1.4 metres of water, a much greater depth than that in the Roberts study. At the end of the study the soil columns were divided into six segments of 5 cm each and the radioactivity was determined by combustion analysis. The treated soils and the top 5-cm sections were also extracted with methanol and the extract analysed.

In the eluates from the Kodaira, Azuchi and Sapporo soils radioactivity was negligible and no attempt was made to characterize it. There was a considerable level of activity in the eluate from the Muko soil. Where soil treated with cyclopropyl-labelled fenpropathrin without pre-incubation had been added to the column, 21.2% of the applied dose appeared in the eluate. The majority of the activity was from $CONH_2$-fenp. with small amounts from 4′-OH-fenp., desph.-fenp. and COOH-fenp., together with TMPA and others. There was very little fenpropathrin itself. In the columns containing pre-incubated soil treated with the cyclopropyl-labelled compound 37.6% of the applied dose appeared in the eluate. There was somewhat less $CONH_2$-fenp. (8.8%) but this was more than balanced by an increased amount of COOH-fenp. (17.5%) and the same complement of minor metabolites. In the case of the benzyl label, results were available only for the pre-incubated soil where the total activity in the eluate was 47.3% of that applied, with 8.7% as $CONH_2$-fenp. and 26.4% as COOH-fenp. A small amount of PBacid (3%) appeared (instead of TMPA of course).

Most of the activity in the soils remained in the treated layers, with minor amounts penetrating into the columns, mainly into the top 5 cm. In all cases the parent compound was the major component of the residues extracted with methanol and only small amounts of metabolites were present, even in the stored soils; $CONH_2$-fenp. was usually the most prominent. A considerable proportion of the activity remained unextractable in the pre-incubated soils, in one case reaching 35% (Kodaira soil with the cyclopropyl label), but in the Azuchi and Sapporo soils about 20% was more typical. The figure was much lower in the Muko sand (3-5%).

It was concluded that fenpropathrin and its metabolites showed only a very limited tendency to leach in normal soils (Mikami *et al.*, 1983b).

Conclusions. It is clear from these studies that fenpropathrin falling on soil will be degraded by a combination of photochemical and microbiological processes. It is unlikely that fenpropathrin would remain in the soil long enough to give rise to carry-over residues to affect succeeding crops.

The evidence is that metabolites do not accumulate in soil and that the degradation of fenpropathrin labelled in all 3 positions has been accompanied by the liberation of carbon dioxide, indicating that fragments observed to occur during the degradation process are themselves ultimately mineralized. There is no evidence of the accumulation of metabolites under aerobic conditions.

Fenpropathrin is strongly adsorbed by normal agricultural soils and studies in the laboratory using columns have shown that in such soils it is very resistant to leaching. The data give reassurance that when used as recommended, fenpropathrin will not cause contamination of groundwater in normal circumstances.

In storage and processing

Apples. Data for processed products are shown in Table 16. Residues of fenpropathrin in apples are essentially superficial as can be seen from the data on peeled apples in the French studies. As would be expected the extraction of apple juice leaves almost all of the residues in the solids.

Table 16. Residues of fenpropathrin in processed products: apples.

Country	Residues , mg/kg				
	Fruit		Pomace	Juice	Ref.
	raw	peeled	wet dry		
France	0.30	<0.01			10
France	0.36	<0.01			10
France	0.29	<0.01			10
France	0.31	<0.01			10
France	0.19	<0.01			10
France	0.20	<0.01			10
France	0.13	<0.01			10
France	0.13	<0.01			10
France	0.48	<0.01			10
France	0.49	<0.01			10
France	0.60	<0.01			10
France	0.16	<0.01			10
USA	3.6	3.4	34	0.10	71
USA	1.6	4.5	10.2	0.01	39

Pears. The position is similar to that of apples, except that the retention of residues by the pear solids would appear to be stronger than in the case of apples. The data are shown in Table 17.

Table 17. Residues of fenpropathrin in processed products: pears.

Country	Residues , mg/kg

	Fruit		Puree	Pears in	Ref.
	raw	peeled	washed	Syrup	
France	0.10	<0.01			8
France	0.05	<0.01			8
France	0.03	<0.01			8
France	0.65	<0.01			9
France	0.35	<0.01			9
France	0.24	<0.01			9
France	0.17	<0.01			9
France	0.42	<0.01			9
France	0.33	<0.01			9
France	0.13	<0.01			9
France	0.10	<0.01			9
France	0.19	<0.01			9
France	0.17	<0.01			9
France	0.19	<0.01			9
France	0.17	<0.01			9
USA	1.70		<0.01	<0.01	64
USA	1.25	0.01			73
USA	0.89	0.01	0.72		72

Grapes. As can be seen from Table 18 residues of fenpropathrin are retained by solids during grape juice extraction, as with apples and pears.

Table 18. Residues of fenpropathrin in processed products: grapes.

Country	Residues , mg/kg						Ref.
	Fruit	Raisins	Raisin	Pomace	Juice	Wine	
	raw	washed	waste	wet	dry		
France	0.06				n.d.		6
USA	0.37	0.45		0.49	2.5	0.03	26**
USA	0.75	1.5	0.13	0.90	4.9	0.09	26
USA	0.74	0.65	3.2	0.85	1.2	n.d.*	n.d. 26
USA	0.42	0.43					26
USA	3.1				0.01	n.d.	26
USA	2.6			2.8	10.5	0.01	26
USA	1.0	0.87					26
USA	1.4			0.99	2.6	n.d.	n.d. 26
USA	5.6			4.1	9.4	0.06	n.d. 26
USA	1.3				4.1	0.04	n.d. 26
USA	0.84	3.2	6.0	1.1	2.1	0.13	26

* n.d.= not detectable, <0.01 mg/kg
** The reference 26 (Fujie, 1992) includes the trials T-6077, T-6078, T-6412, T-6409, T-6413, T-6416, T-6728, T-6731, T-6829, T-6413, T-7545

Cotton seed. Three samples of seed from field trials were subjected to simulated laboratory processing as described below (ref. T-6427, 210-214 of Fujie, 1990a) and the residues surviving in various fractions were determined. It can be seen from Table 19 that residues in

soapstock were at about twice the level in the raw seed and that residues in the refined oil were in the region of three times those in the seed. Assuming a maximum level in raw cotton seed of 1 mg/kg, it can reasonably be concluded that residues will not exceed 2 mg/kg in soapstock and 3 mg/kg in refined oil.

Processing procedure. A Carver impact huller is used to obtain the fractions (kernels and hulls). The kernels are flaked in a Ferrell-Ross "flake-n-roll" to 0.008 of an inch thickness. The flakes are washed three times with hexane at a temperature of approximately 145°C. This extraction process takes 3 hours. The oil is recovered with a precision laboratory evaporator. During this process the oil reaches a maximum temperature of 75°C. Warm air is forced through the extractor to dissolve the cotton seed flakes. The oil is refined by the following steps:

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

Table 19. Residues of fenpropathrin in processed products: cotton seed processed in the USA. Fujie, 1990a (ref. 23).

Residues, mg/kg						Ref.
Seed	Meal	Crude oil	Refined oil	Hulls	Soap-stock	
0.02	0.01	0.06	0.06	0.02	0.01	T-6070
0.03	0.02	0.07	0.09	0.03	0.05	T-6071
1.2	0.09	2.3	2.6	1.0	1.6	T-6427

Stability of residues in stored analytical samples

Fenpropathrin. Samples of apples, cotton seed, grapes, oranges and pears were stored at -20°C for periods up to 12 months and analyzed for residues of fenpropathrin to determine the extent of loss during storage. The results are shown in Table 20.

Table 20. Effects of frozen storage on residues of fenpropathrin.

Interval (months)	% of initial level				
	Apples	Cotton seed	Grapes	Oranges	Pears
3	82	133		93	99
6	78	116	89	120	77
9	91		87		78
12	82	100	87	88	89

No clear trend emerges from these figures which fall within the normal spread of recoveries and it was concluded that there were no measurable losses from any of these items during one year of storage at -20°C. Similar conclusions were drawn from studies with grape juice,

grape pomace, raisins and raisin waste (Fujie, 1992).

Stability studies were also carried out on eggs and cattle kidneys over periods of 156 and 71 days respectively. Again there was no evidence of decline in the residue levels during storage (Fujie *et al*., 1986b).

It may reasonably be inferred that the same conclusions could be drawn for other food and feed items.

Metabolites. Samples of macerated oranges and pears were spiked with known amounts of either PBacid or TMPA and analyzed after 7.5 or 11.2 months of storage. The results were compared with recoveries from freshly spiked macerated samples of the same substrates. See Table 21.

Table 21. Storage stability of metabolites.

Fruit	Interval (months)	% recovery from spiked samples			
		Frozen		Fresh	
		TMPA	PBacid	TMPA	PBacid
Orange	7.5	72	77	92	82
		75	71		
	11.2	62	79	67	75
		64	78		
Pears	7.5	55	62	65	77
		62	66		
	11.2	57	70	72	76
		54	59		
Mean		63	70	74	78
Coefficient of variation		12%	11%	17%	4%

Hence the recoveries of TMPA and PBacid from fresh and frozen samples show no significant differences and demonstrate that both metabolites are stable in orange and pear macerates during the storage intervals of the study (Fujie, 1990c).

It is reasonable to infer a similar stability in other substrates.

Residues in the edible portion of food commodities

Crop by-products used for animal feed

The main commodities used for animal feeds are the fruit pomaces, cotton seed meal and soapstock. Residues in cotton seed meal and soapstock were much lower than in the fruit pomaces.

Fruit pomaces. Dry grape pomace (see Table 18) contained between 2 and 7 times the residue level in the original grapes. If the highest level in raw grapes is 5 mg/kg, the highest level to be expected in dry grape pomace would be 35 mg/kg. In the case of raisin waste,

the highest factor was again 7 so that a similar figure could be anticipated.

In the case of dry apple pomace the two sets of results in Table 16 suggest a maximum concentration factor of 9, so that residues in dry apple pomace would not be expected to exceed 45 mg/kg on the
basis of a maximum residue level in whole apples of 5 mg/kg.

METHODS OF RESIDUE ANALYSIS

Parent compound. A number of analytical procedures have been developed by Sumitomo. These depend on solvent extraction of the substrate, a clean-up by either silica gel or Florisil column chromatography and the determination of the extracted residue by GLC using electron capture detection. The main variations dictated by different substrates are concerned with extraction and clean-up procedures. Fruits and vegetables may be homogenized with water, shaken with acetone and extracted according to accepted procedures with dichloromethane, using sodium chloride to minimize emulsification. After drying with anhydrous sodium sulphate and clean-up by silica gel column chromatography, the solvent is evaporated at <40°C and the residue dissolved in acetone before estimation by GLC with EC detection. Other extraction procedures involve direct extraction of the homogenized material without suspension in water or homogenization with methanol instead of water.

In the case of cotton seed oil dissolution of the sample in n-hexane is followed by extraction with acetonitrile, which is removed by evaporation and the residue dissolved in acetone for measurement.

A somewhat similar procedure is recommended for animal fats, whereas in the case of meat or offal (kidney was specifically studied) the sample is homogenized in acetone and the suspension extracted with n-hexane. After drying with anhydrous sodium sulphate the solution is extracted with acetonitrile, the solvent evaporatedd and the residue redissolved in n hexane for estimation.

Milk is mixed with an equal volume of acetone and centrifuged. The combined supernatant layers are extracted with n-hexane, the extract is dried with anhydrous sodium sulphate and the solvent evaporated. The residue is redissolved in acetonitrile, cleaned up by silica gel chromatography, and dissolved in hexane before determination by GLC.

Soils are extracted with combined water/methanol and, after filtration, the extract is partitioned with dichloromethane. The solution is dried with anhydrous sodium sulphate and taken to dryness by rotary evaporation. The residue is re-dissolved in n-hexane containing 10% diethyl ether and cleaned up by silica gel chromatography. The solvent is removed by evaporation and the residue dissolved in acetone for estimation by GLC.

In the case of water samples large volumes, up to 10 l depending on the lower limit of determination required, are extracted with n-hexane and the extract dried with sodium sulphate and cleaned up by Florisil column chromatography.

The relationship between the amount of fenpropathrin and peak area is linear over the usual range of 0-0.8 ng of fenpropathrin with a lower level of determination of 0.04 ng. Recoveries from most substrates have been reported to be between 90 and 100% although this fell to 80% in water where very large volumes were involved. The lower limit of determination in most crop samples is 0.01 mg/kg although in some cases it is possible to achieve 0.005 mg/kg, depending on the success of the clean-up steps. In whole milk a lower limit of determination of 0.001 mg/kg is usual, whereas in water limits down to 0.001

µg/l have been achieved (Ohnishi and Suzuki, 1981, 1982a,b, 1983a,b,c; Ohnishi *et al*., 1987; Kadooka *et al*., 1991; Hirota, 1990).

The procedures for crop samples are basically similar to those adopted by the Shell Company, except that in their procedure the crop samples are extracted dry in the presence of anhydrous sodium sulphate and homogenized with a 1:1 mixture of acetone and petroleum spirit. The extract is dried and cleaned up by Florisil column chromatography, the solvent is evaporated and the solute dissolved in petroleum spirit before its analysis by GLC. It is recommended that a standard solution should be chromatographed with each two sample solutions to make certain that there is a continuous correction of small changes in the response of the equipment. The method also includes a TLC procedure for confirming the identity of the fenpropathrin (Anon, 1976b).

Methods developed in the USA are similar in principle to those developed in Japan. In summary, macerated crop samples (20g) are blended with 100g sodium sulphate and 150 ml acetone/hexane (1:2), re-extracted, and the extracts dried with sodium sulphate. The extracts are then shaken with water and partitioned with hexane. The resulting hexane extract is evaporated to dryness, and the residue redissolved in hexane for clean-up by silica gel chromatography. The column is eluted with 1:5 ether/hexane, the eluate evaporated to dryness and the residue redissolved in methanol. The methanol solution is further cleaned up on a C-18 "SepPak", the eluate is taken to dryness, and the residue redissolved in hexane for determination of fenpropathrin by gas chromatography with an electron capture detector.

The response of the system is linear with analytical standard solutions covering the range 0.06-0.12 µg/ml, but the linearity must be checked each day. The limit of determination of the method for crop samples is approximately 0.01 mg/kg for a 20-g sample which is equivalent to a concentration in the injected solution of 0.01 µg/ml. A fortified control sample should be analyzed with each set of unknown samples. Recoveries from fortified samples should be between 70 and 120%; recoveries outside these limits require a repeat of the analysis.

This method has been developed further to cover residues in oily samples such as cotton seed. The macerated sample (20g) is moistened with 20 ml water and blended with 100 g sodium sulphate and 200 ml acetone/hexane (1:2), re-extracted, and the extract concentrated to about 20 ml, made up to 50 ml with more hexane and partitioned into acetonitrile. The solution is evaporated to dryness and the residue redissolved in hexane for clean-up and measurement.

Oil samples are dissolved in hexane, partitioned into acetonitrile and the solution evaporated to dryness. The residue is redissolved in hexane and subjected to an alumina column clean-up. The fenpropathrin is eluted from the column with hexane containing 10% ether. The eluate is evaporated to dryness and the residue redissolved in hexane for GLC.

For soapstock, a 5-g sample is dissolved in water, diluted further in a separating funnel and extracted twice with dichloromethane, using phosphoric acid and sodium chloride to minimize emulsification. The combined extracts are taken to dryness, redissolved in hexane and partitioned with acetonitrile which in turn is evaporated and the residue redissolved in hexane for clean-up on an alumina column (Leary and Abell, 1986c; Fujie, 1990a).

The method also allows analysis of milk, eggs and animal tissues. The samples (homogenized as appropriate) are extracted with acetone/hexane (1:2), and the extract diluted with water. The lower aqueous layer is extracted with hexane. The hexane extracts are dried over sodium sulphate and the solvent removed. The residue is redissolved in

hexane and cleaned up by silica gel chromatography. With fatty samples, the hexane solutions are first extracted with acetonitrile then re-extracted with hexane before clean-up and subsequent determination by GLC with an electron capture detector (Fujie *et al.*, 1986b).

A further variation of the method was employed in the analysis of some of the grape samples, where a nitrogen-phosphorus flame ionization detector was used as an alternative to electron-capture (Fujie, 1992).

Metabolites. For the determination of the metabolites PBacid and TMPA, macerated crop samples are extracted with methanol/water, the pH adjusted to 8.3 and the solution extracted with hexane to remove parent fenpropathrin. It is then acidified to liberate the acids which are derivatized with pentafluorobenzyl bromide. An aliquot of the reaction mixture is then cleaned up by passing through a silica "SepPak" cartridge and analyzed by gas chromatography using a mass-selective detector.

Recoveries from crop samples averaged 69% for TMPA and 76% for PBacid but the acceptable recovery range was estimated to be 60-120% and thus wider than for fenpropathrin owing to the complexity of the procedure (Fujie, 1988).

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported to the Meeting.

Country	Commodity	MRL, mg/kg
Austria	Apple	1
	Cucumber	
	(incl. Squash, Summer)	0.1
	Egg plant	0.1
	Grapes	1
	Pepper, Sweet	0.1
	Pear	1
	Tomato	0.1
Belgium	Apple	0.5
	Cucumber	
	(incl. Squash,summer)	1
	Egg plant	1
	Pepper, Sweet	1
	Pear	0.5
	Tomato	1
France	Apple	0.5
	Grapes	0.5

Country	Commodity	MRL, mg/kg
	Peach	0.5
Germany	Dwarf French beans	1
	Climbing French beans	1
	Cucumber	0.2
	Tomato	1
Hungary	Apple	0.3
	Cucumber	0.1
	Grapes	0.2
	Pears	0.3
	Tomato	0.2
Italy	Apple	1
	Citrus fruits	1
	Cucumber	
	(incl. Squash, Summer)	1
	Grapes	1
	Peach	1
	Pear	1
	Tomato	1
Japan	Apple	1
	Citrus	
	peel	10
	fruits	2
	Cucumber	2
	Egg plant	2
	Pepper, Sweet	2
	Peach	2
	Pear	1
	Strawberry	2
	Tea, Green, Black	30
	Tomato	2
Netherlands	Apple	0.5
	Cucumber	

Country	Commodity	MRL, mg/kg
	(incl. Squash,Summer)	1
	Egg plant	1
	Pepper, Sweet	1
	Pear	0.5
	Tomato	1
Spain	Apple	0.1
	Cotton seed	0.05
	Egg plant	0.5
	Pear	0.1
	Tomato	0.5
Switzerland	Beans (Greenhouse)	0.5
	Beans (Field)	0.02
	Cucumber	0.02
	Pome fruits	0.02
	Stone fruits	0.02
USA	Apple	5*
	Cotton seed	1*
	Pear	5*

* proposed tolerances in USA

APPRAISAL

Residue data from supervised trials on apples, cotton seed, gherkins, grapes, pears and tomatoes were supplied to the Meeting. No data on cucumber were received.

The major biotransformation reactions of fenpropathrin in animals consist in oxidation at the methyl groups of the acid moiety and at the 2_ and 4_ positions of the alcohol moiety, cleavage of the ester linkage and conjugation of the resultant carboxylic acids and alcohols with glucuronic acid, sulphuric acid and glycine.

Studies in plants with radio-labelled fenpropathrin demonstrate that in fruit fenpropathrin itself is the primary component of the residues, whereas in leaves degradation products constitute the greater part of the residues. The major metabolic reaction of fenpropathrin in plants has been found to be the rupture of the ester linkage followed by oxidation to produce 3-phenoxybenzoic acid (PB acid) and the corresponding alcohol and aldehyde. From the acid side of the molecule, the main metabolite is 2,2,3,3-tetra-methylcyclopropanecarboxylic acid (TMPA) and this compound can give rise to 2-hydroxymethyl-2,3,3-trimethylcyclopropanecarboxylic acid (TMPA-CH_2OH) and 5-hydroxymethyl-6,6-dimethyl 3-oxabicyclo-[3.1.0]hexan-2-one (TMPA-CH_2OH lactone) by

subsequent hydroxylation. Also PB acid can be hydroxylated at the 4′ position and the parent molecule at the 2′ or 4′ position. The hydroxylated derivatives exist in both free and conjugated forms. Breakdown products in plants did not differ greatly from those in animals. The residues of the main metabolites PB acid and TMPA in samples from supervised field trials constituted only a negligible proportion of the total residues. It is therefore considered appropriate to define the residue in crops as the parent compound.

The fate of fenpropathrin in the soil will be influenced by a combination of photo degradation and microbiological processes. It is unlikely that fenpropathrin will remain in the soil long enough for residues to survive and affect succeeding crops. Metabolites do not accumulate in soil. Fenpropathrin is strongly adsorbed by soils, and when used as recommended will not contaminate ground water. Examination of plants grown on treated soils showed only extremely small uptake of radioactivity.

The residue data from supervised trials were evaluated as follows.

Apple. Results of 19 US trials with a maximum application rate of 0.45 kg ai/ha, a 14-day PHI and a maximum of 8 applications showed that the residues were below 5 mg/kg in whole fruit (minimum 0.06 mg/kg, maximum 4.5 mg/kg, estimated maximum residue level 5 mg/kg).

Pear. The maximum level observed in pears treated according to anticipated approved uses was 3.2 mg/kg in whole fruit in the State of Washington, USA, where the spray concentration was 0.024%, the application rate 0.9 kg ai/ha, the PHI 14 days and the crops were subjected to a total of 6 applications. In 15 supervised US trials within GAP based on 0.45 kg ai/ha, 0.012%, a 14-day PHI and 8 applications all residues were below 5 mg/kg (minimum 0.58 mg/kg, maximum 2.9 mg/kg; estimated maximum residue level 5 mg/kg).

Grapes. The maximum GAP was in US trials. There were 4 trials within GAP (0.45 kg ai/ha, a 21-day PHI and 4 applications; minimum 0.84 mg/kg, maximum 2.6 mg/kg; estimated maximum residue level 5 mg/kg) and 18 trials using 0.22 kg ai/ha, with a PHI of 21 days and also 4 applications. It is considered that residue levels from applications based on accepted use recommendations would normally fall below 5 mg/kg.

Gherkin. Residues in samples from 4 supervised German trials using an application rate of 0.08 kg ai/ha , a 3-day PHI and 3 applications did not exceed 0.1 mg/kg (minimum <0.01 mg/kg, maximum 0.1 mg/kg; estimated maximum residue level 0.2 mg/kg).

Peppers, Sweet. Residues from outdoors supervised trials based on 3 applications of 0.01% and a 0-1-day PHI in Japan and Spain ranged from 0.2 mg/kg to 1.2 mg/kg (estimated maximum residue level 1 mg/kg). Spanish residues (indoors, 7-day PHI) ranged from 0.04 to 0.38 mg/kg and for a 2-day PHI from 0.34 to 0.52 mg/kg.

Tomato. The highest levels were seen in four Japanese studies, because GAP in Japan allows an application rate of 0.25 kg ai/ha and a one-day PHI. One figure exceeded 1 mg/kg. Residue results of 5 outdoor and 8 indoor supervised trials in Germany with a lower application rate of 0.08 kg ai/ha show that 3 days after the last application residues were all below 0.6 mg/kg. (Outdoors: minimum <0.01 mg/kg, maximum 0.37 mg/kg. Indoors: minimum <0.01 mg/kg, maximum 0.46 mg/kg; estimated maximum residue level 1 mg/kg).

Egg plant. Residues from 4 Japanese trials based on 3 - 5 applications of 0.01% and a 1-day PHI were low (minimum 0.12 mg/kg, maximum 0.19 mg/kg; estimated maximum residue level 0.2 mg/kg).

Cotton seed. A well-known factor that can influence the level of residues in cotton seed is whether an appreciable number of bolls have opened at the time of the last application. If not, residues in the seed are usually very low but if there is direct contact between the

insecticide spray and the seed, residues can reach measurable levels. In considering the MRL needed it is important that it should be high enough to include cases where the last application was to plants with a comparatively high proportion of open bolls. It was possible to use 26 trials with an application rate of 0.22 kg ai/ha, 8 - 11 applications and a PHI of 18 - 22 days (minimum residues <0.01 mg/kg, maximum 1 mg/kg; estimated maximum residue level 1 mg/kg).

Residues in food of animal origin

Cattle. Residues in whole milk when a plateau level had been reached were approximately 0.15% of the level in the feed. If cows were fed on a diet consisting entirely of dried apple pomace at the postulated maximum residue level of 45 mg/kg (see processing of apples, below), it could be argued that the maximum level in milk would be 0.07 mg/l. Assuming that these residues would all be present in the fat and that the fat content of the milk would be 4%, such a level would be equivalent to 1.8 mg/kg in the milk fat. An animal transfer study showed levels in body fat to be approximately 1.4% of the level in the feed. Using the apple pomace figure of 45 mg/kg, it is reasonable to conclude that residues in meat fat would not exceed 0.6 mg/kg. Based on similar arguments and the data from the same studies, residues in meat (muscle) were about 0.08% of the feed level so that animals fed on apple pomace at 45 mg/kg would not be expected to have more than 0.05 mg/kg in muscle, kidney or liver.

Poultry. Poultry are unlikely to receive dietary items containing appreciable residues of fenpropathrin with the possible exception of cotton seed meal. With a maximum level of 1 mg/kg in raw cotton seed, it is unlikely that residues in meal would exceed 0.1 mg/kg. With a total feed level of 2.5 mg/kg, the level in fat reached only 0.02 mg/kg so that measurable residues would not be expected in the eggs, meat or edible offal of poultry fed on cotton seed meal.

In processing

In fruits the residues are essentially surface residues. As would be expected juice extraction leaves the great majority of the residues in the solids. In the case of dry apple pomace the data suggest a maximum concentration factor of 9, so that residues in dry apple pomace would not be expected to exceed 45 mg/kg on the basis of a maximum residue level in whole apples of 5 mg/kg.

As would be expected, raisins have higher residues than the raw grapes. The highest concentration factor in the trials is about 3. Using this factor and assuming that residues in raw grapes will not exceed 5 mg/kg, it would seem reasonable to estimate that residues in raisins would not exceed 15 mg/kg.

Dry grape pomace contained between 2 and 7 times the residue level in the original grapes. If the highest level in raw grapes is 5 mg/kg, the highest level to be expected in dry grape pomace would be 35 mg/kg.

Processing grape juice into wine appears to reduce residue levels still further and although strictly comparable data are only rarely available, residues of fenpropathrin have not been found above the limit of determination in wine, whereas in juice the highest level found was 0.06 mg/kg, which disappeared during vinification. In this particular case residues in the raw grapes were up to 5.6 mg/kg, so that even at this high level measurable residues did not survive in the wine.

As would be expected from the lipophilic nature of fenpropathrin, residues in oil obtained from cotton seed are higher than in the raw seed by roughly the inverse proportion of oil weight to seed weight. The residues in the meal ranged from 0.01 to 0.09

mg/kg. Residues in soapstock were about twice the level in the raw seed and residues in the refined oil were in the region of three times the seed level. Assuming a maximum level in raw cotton seed of 1 mg/kg, it can reasonably be concluded that residues in soapstock will not exceed 2 mg/kg and in oil 3 mg/kg.

Stability of stored analytical samples

In stability studies carried out on apples, pears, grapes, oranges, cotton seed, eggs and kidney of cattle over periods from 3-12 months there was no evidence of a decline in residue levels of fenpropathrin during storage at -20°C.

Methods of residue analysis

Methods of analysis used GLC with an EC detector after solvent extraction of the substrate and clean-up by either silica gel or Florisil column chromatography. The limit of determination in most crop samples is between 0.005 and 0.01 mg/kg.

RECOMMENDATIONS

On the basis of the residue data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing Maximum Residue Limits.

Definition of the residue: fenpropathrin (fat soluble)

Commodity		Recommended MRL (mg/kg)		PHI on which based, days
CCN	Name	New	Previous	
MO 0812	Cattle, Edible offal of	0.05	-	
MM 0812	Cattle meat	0.5 (fat)	-	
ML 0812	Cattle milk	0.1 F	-	
SO 0691	Cotton seed	1	-	18-22
OC 0691	Cotton seed oil, crude	3	-	
PE 0112	Eggs	0.01*	-	
VO 0440	Egg plant	0.2	-	1
VC 0425	Gherkin	0.2	-	3
FB 0269	Grapes	5	-	21
VO 0445	Peppers, Sweet	1	-	0-2
FP 0009	Pome fruit	5	-	14
PO 0111	Poultry, Edible offal of	0.01*	-	
PM 0111	Poultry meat	0.02 (fat)	-	
VO 0448	Tomato	1	-	3

* Limit of determination

FURTHER WORK OR INFORMATION

None.

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