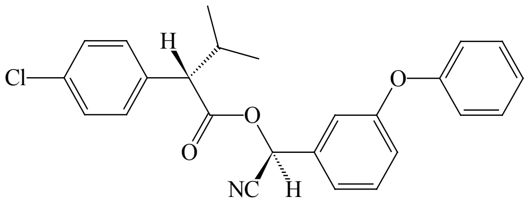
ESFENVALERATE (204)

First draft prepared by Denis Hamilton, Animal and Plant Health Service, Department of Primary Industries, Australia

IDENTITY

ISO common name:	esfenvalerate
Chemical name	
IUPAC:	(*S*)-α-cyano-3-phenoxybenzyl (*S*)-2-(4-chlorophenyl)-3-methylbutyrate
Chemical Abstracts:	[(*S*)-(*R**,*R**)]-cyano (3-phenoxyphenyl) methyl-4-chloro- α-(1-methylethyl) benzeneacetate
Company code nos.:	SAG 303, MO 70616, S-1844, S-5602Aα, DPX-GB800, DPX-YB656
CAS no.:	66230-04-4
CIPAC no.:	481
Minimum purity:	830 g/kg
Molecular formula:	$C_{25}H_{22}ClNO_3$
Molecular mass:	419.9
Structural formula:	



PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

Appearance:	white crystalline solid
Melting point:	59.1–60.1ºC
Boiling point:	not applicable
Relative density:	1.23 g/cm^3 at 26ºC
Vapour pressure:	1.17 x 10^{-9} Pa at 20ºC (99.9% pure)
Henry's law constant:	4.92 x 10^{-4} Pa m^3 mol^{-1}
Solubility in water:	<1µg/l (pH: 5.3) at 20ºC
Octanol/water partition coefficient:	log P_{ow} = 6.24 at 25ºC
Hydrolysis (sterile solution) (DT-50):	pH 5: approx. 120-130 days
	pH 7: not observable in 28 days
	pH 9: 64 days
Dissociation constant:	no dissociation
Quantum yield of direct photo-transformation in water at λ >290 nm:	$\Phi = 6.8 \times 10^{-3}$
Photostability (DT-50):	10 days (sunlight), 6 days (artificial light), in water
Flammability:	Non-flammable
Explosive properties:	Non-explosive
UV/visible absorption (max):	2.3 x 10^3 mol^{-1} cm^{-1} at 278 nm

Technical material

Appearance:	yellow-brown viscous liquid or solid at 23ºC
Minimum purity:	830 g/kg
Melting point:	43.3-54ºC
Boiling point:	151-167ºC
Stability:	Relatively stable to heat and light. Stable to hydrolysis at pH 5, 7, and 9 at 25ºC
Solubility in organic solvents:	n-hexane: 26 g/l methanol: 82 g/l in most other organic solvents: >500 g/l

Typical isomer composition of fenvalerate and technical esfenvalerate

	S,S-isomer	*R,S*-isomer	*S,R*-isomer	*R,R*-isomer
fenvalerate	23%	27%	27%	23%
technical esfenvalerate	84%	8%	7%	1%

FORMULATIONS

Esfenvalerate is available in the following formulations.

Emulsifiable concentrate (EC) formulations containing 25, 50, 100, 230, or 280 g/l esfenvalerate.

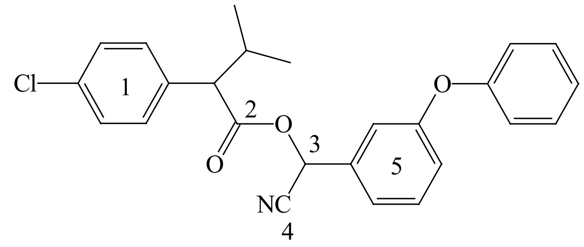
Emulsion, oil in water (EW) formulations containing 50 or 100 g/l esfenvalerate.

METABOLISM AND ENVIRONMENTAL FATE

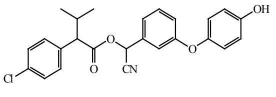
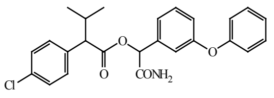
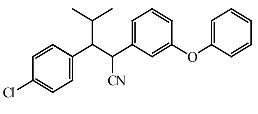
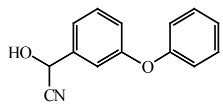
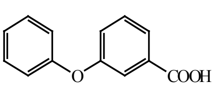
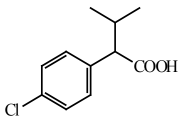
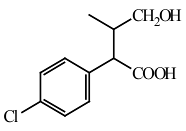
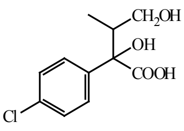
Fenvalerate and esfenvalerate were labelled with ^{14}C in various positions in the metabolism studies.

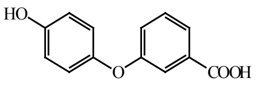
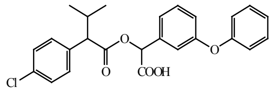
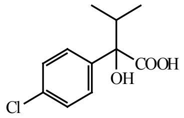
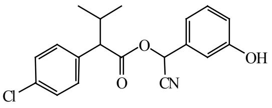
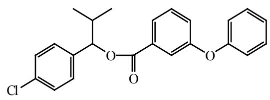
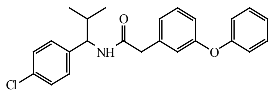
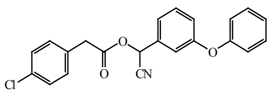
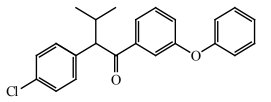
The positions are described as:

1: ^{14}C-chlorophenyl
2: ^{14}C-carbonyl
3: ^{14}C-benzylic
4: ^{14}C-cyano
5: ^{14}C-phenoxybenzyl



Metabolites are given various abbreviations and code numbers in the studies. Structures, abbreviations and codes are shown below.

 4'-OH-fen SD 48838 4'-OH-S5602	 CONH$_2$-fen SD 47117	 Dec-fen SD 54597	 PBalc-CN
 PBacid SD 44607	 Cl-V acid CPIA SD 44064	 3-OH-CPIA SD 53919	 2,3-OH-CPIA

 4'-OH-PBacid SD 46114	 COOH-fen	 SD 53065	 DE-Ph-S5602 SD 58086
 SD 53036	 SD 55886	 WL 45329	 SD 55888

***Note*: The names 3-OH-CPIA and 2,3-OH-CPIA are confusing because it is the 4-C methyl group, not the 3-C, CH_2 or CH, that is substituted, and in the dihydroxy compound the hydroxyl groups are not substituted adjacent carbons. The names are retained however because they have been widely used.**

Animal metabolism

The Meeting received studies of esfenvalerate and fenvalerate metabolism in rats and mice, and of fenvalerate metabolism in dairy cows and laying hens. The studies on rats and mice were evaluated by the WHO Core Assessment Group.

In a comparison of esfenvalerate and fenvalerate metabolism, Isobe *et al.* (1985) administered diets containing ^{14}C-chlorophenyl-labelled compounds to mice for 28 days. Two metabolites were identified in the livers and kidneys: CPIA and 3-OH-CPIA.

The following compounds were identified as metabolites of esfenvalerate in rats or mice, appearing in the excreta in amounts exceeding 5% of the dosed parent compound (Kaneko *et al.*, 1985): 4'-OH-fen, CPIA, 2,3-OH-CPIA, 4'-OH-PBacid (free + conjugated), PBacid (free + conjugated).

Cows. Tissue, milk and excreta residues were measured in lactating Guernsey dairy cows (treatment group of 5 animals, control group of 3, each animal weighing 410-640 kg) fed with a diet containing 78 ppm [^{14}C-chlorophenyl] and [^{14}C-phenoxybenzyl]fenvalerate in the feed for 21 days (Barber *et al.*, 1981; Lee, 1989a). The mean feed intake per day of dairy concentrate and alfalfa hay was 11.2-15.9 kg for each animal. Milk was collected each morning and evening and excreta were collected daily. Three animals were slaughtered 12 hours after the final dose for tissue collection. Two of the treated animals were placed on an untreated ration for an additional 10-20 days.

Fenvalerate rapidly reached a plateau level in milk, by about day 3 of feeding (Table 1). Approximately 90% of the ^{14}C in the milk was accounted for by fenvalerate itself. Almost all of the ^{14}C in the milk was present in the fat. A comparison of the ^{14}C measurement on body and milk fat with a measurement of fenvalerate by GLC shows that most of the ^{14}C was present as fenvalerate (Table 2). Fenvalerate also accounted for more than 90% of the total radioactive residues in the muscle, where the ^{14}C level on day 21 was approximately 0.25 mg/kg. Metabolites in the liver and kidney were consistent with those from rats, mice, plants and soil. Levels of ^{14}C in the liver and kidney were approximately 2 and 1.4 mg/kg respectively. The major metabolites in liver and kidney are shown in Table 3.

Table 1. Fenvalerate residues in milk resulting from a diet containing 79 ppm fenvalerate (Barber *et al.*, 1981; Lee, 1989a).

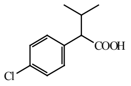
Days	Fenvalerate by GLC, mg/kg					
	Cow 1	Cow 2	Cow 3	Cow 4	Cow 5	Mean
1	0.07	0.15	0.04	0.13	0.07	0.09
3	0.35	0.58	0.38	0.53	0.41	0.45
5	0.41	0.43	0.44	0.44	0.36	0.42
7	0.45	0.50	0.48	0.40	0.33	0.43
9	0.50	0.52	0.46	0.52	0.42	0.48
11	0.52	0.48	0.48	0.54	0.36	0.48
13	0.47	0.43	0.39	0.61		0.48
15	0.56	0.53	0.40	0.54	0.34	0.47
17	0.75	0.51	0.49	0.56	0.33	0.53
19	0.51	0.53	0.52	0.51	0.35	0.48
21	0.44	0.59	0.43	0.44	0.32	0.44
22				0.33	0.23	0.28
23				0.12	0.10	0.11
24				0.08	0.05	0.07
25				0.05	0.03	0.04

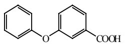
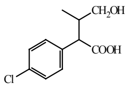
Table 2. Comparison of the total ^{14}C in milk and body fat and the fenvalerate content as measured by GLC in the dairy cow metabolism study (Lee, 1989a).

	^{14}C as fenvalerate, mg/kg	Fenvalerate by GLC, mg/kg
Cream day 7	7.2	6.8
Cream day 13	5.5	5.8
Cream day 21	5.2	4.6
SC fat cow 1 day 21	1.08	0.8
SC fat cow 2 day 21	1.33	1.2
SC fat cow 3 day 21	1.79	1.6
SC fat cow 4 day 31	2.23	2.0
SC fat cow 5 day 43	1.78	1.8
Mesenteric fat cow 1 day 21	1.69	1.8
Mesenteric fat cow 2 day 21	1.81	1.5
Mesenteric fat cow 3 day 21	3.36	3.4
Mesenteric fat cow 4 day 31	2.72	3.2
Mesenteric fat cow 5 day 43	2.39	2.2

Boyer and Lee (1981) identified the metabolites in kidney and liver from the dairy cow feeding study.

Table 3. Metabolites of fenvalerate in the kidney and liver of dairy cows resulting from a diet containing 78 ppm ^{14}C-chlorophenyl- and ^{14}C-phenoxybenzyl-labelled fenvalerate (Boyer and Lee, 1981).

Compound or fraction	% of total ^{14}C in sample	
	Kidney	Liver
Bound residues	10	
Water soluble, unidentified	10	4
Fenvalerate	17	
Organo-extractable, unidentified		3
SD 44064 	26	33
SD 44064 conjugates		13

Compound or fraction	% of total ^{14}C in sample	
	Kidney	Liver
SD 44607 	10	
SD 44607 conjugates		15
SD 53919 conjugates 		3

Hens. Separate groups of White Leghorn laying hens (10 birds per group) were dosed by capsule with [^{14}C-chlorophenyl]fenvalerate or [^{14}C-phenoxybenzyl]fenvalerate equivalent to approximately 158 ppm in the feed for 5 consecutive days (Potter, 1982). The birds were slaughtered 18 hours after the final dose for tissue collection and analysis. Eggs were collected each day. Body weights of the hens were 1.5-2.0 kg. Levels of radiolabel appearing in the tissues and eggs are shown in Table 4.

Fenvalerate was identified as the major component of the residue in fat and egg yolk, accounting for 81% and 85% of the radiolabel in fat and 52% and 70% in egg yolk from the chlorophenyl and phenoxybenzyl groups respectively. SD 44064 accounted for 8% of the label in egg yolk from the chlorophenyl group.

In the livers from the chlorophenyl-labelled group free and conjugated SD 44064 represented 38% of the total ^{14}C. In the livers from the phenoxybenzyl group 12% of the ^{14}C was associated with free and conjugated SD 44607, and 3% with conjugated SD 46114. Further efforts (Lee *et al.*, 1985b) to characterize the residue in liver indicated that approximately 50% of the radiolabel was tissue-bound.

Table 4. Levels of radiolabel in the tissues of hens dosed with fenvalerate for 5 consecutive days at the equivalent of 158 ppm in the feed (Potter, 1982).

Sample	^{14}C as fenvalerate, mg/kg	
	chlorophenyl label	phenoxybenzyl label
Liver	2.4	0.96
Fat	0.50	0.50
Egg yolks day 5	1.3	0.97
Egg whites	<0.2	<0.2
Dark meat	<0.2	<0.2
White meat	<0.2	<0.2

Akhtar *et al.* (1989) administered a single dose of 7.5 mg of [^{14}C-carbonyl]fenvalerate to laying hens after dosing for 3 days with unlabelled fenvalerate and examined the metabolism and distribution of the residue. About 85% of the dose was excreted within 24 hours. Metabolites identified in the excreta were 4'-OH-fen, CPIA, 2,3-OH-CPIA and 3-OH-CPIA.

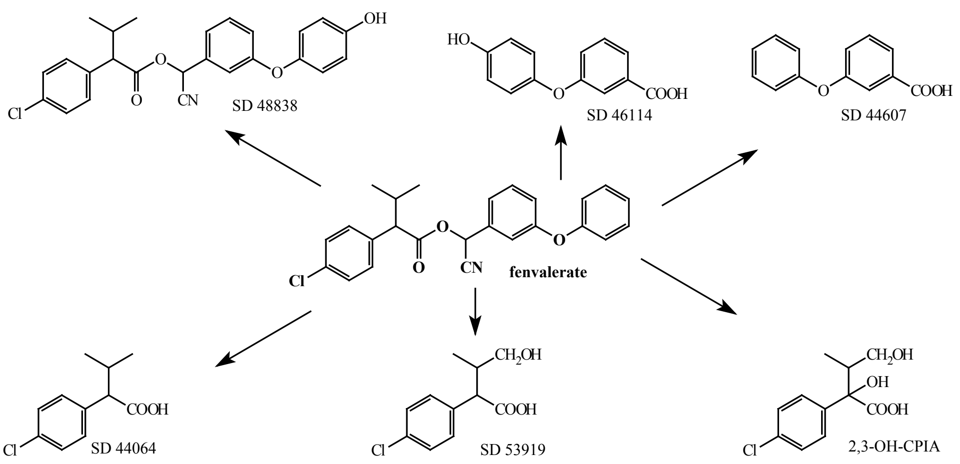


Figure 1. Animal metabolism of fenvalerate. Carboxyl compounds may be free or conjugated.

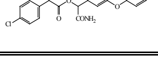
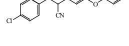
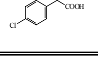
Plant metabolism

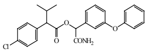
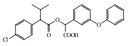
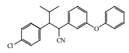
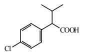
The Meeting received reports of studies of esfenvalerate metabolism in cabbages and fenvalerate metabolism in apple trees, cabbages, kidney beans, lettuce, soya beans, tomatoes and wheat.

Four-week old cabbage seedlings (Capitata variety) were treated evenly on the upper surfaces of 2 of the 5th-6th leaves at the rate of approximately 20 µg per 25 cm^2 (area of a single leaf) with either ^{14}C-chlorophenyl- or ^{14}C-phenoxybenzyl-labelled fenvalerate or esfenvalerate (Mikami *et al.*, 1985). Cabbages were harvested 24 and 48 days after treatment and examined for the nature and levels of the residue (Table 5).

The nature and amounts of metabolites (and photoproducts, e.g. Dec-fen) formed from fenvalerate and esfenvalerate were very similar. Most of the radiolabel remained with the treated leaves with less than 3% of the dose in other parts of the plant. No parent compound was found in untreated shoots. The rate of decrease of parent residues was very close for fenvalerate and esfenvalerate (18-20 days half-life). No αS/αR epimerization was observed in residues in cabbage treated with esfenvalerate. Four further metabolites (phenoxybenzoic acid, hydroxy-phenoxybenzoic acids and phenoxybenzyl alcohol) in free and conjugated forms were identified at low concentrations (0.1-4.5% of the applied dose).

Table 5. Nature of the residue in cabbages after treatment with ^{14}C-labelled fenvalerate and esfenvalerate (Mikami *et al.*, 1985).

Time after treatment, days	Fenvalerate or esfenvalerate	Residues as % of applied ^{14}C				
		III ($CONH_2$-fen) 	IV, (COOH-fen) free + conj 	V (Dec-fen) 	X, (CPIA) free + conj 	Bound ^{14}C
[^{14}C-chlorophenyl]fenvalerate						
24	41	1.1	1.2	1.8	7.2	2.2
48	19	1.3	1.4	2.2	5.0	1.1
[^{14}C-phenoxybenzyl]fenvalerate						
24	36	0.8	0.5	1.9		6.7
48	20	1.6	1.0	2.1		5.7
[^{14}C-chlorophenyl]esfenvalerate						
24	38	0.8	0.3	0.6	9.6	3.7

Time after treatment, days	Fenvalerate or esfenvalerate	Residues as % of applied ^{14}C				
		III ($CONH_2$-fen) 	IV, (COOH-fen) free + conj 	V (Dec-fen) 	X, (CPIA) free + conj 	Bound ^{14}C
48	17	1.0	0.3	0.1	7.3	4.4
[^{14}C-phenoxybenzyl]esfenvalerate						
24	32	0.7	0.8	0.9		3.9
48	17	1.4	0.4	0.5		1.2

Cabbage seedlings, 4 weeks old, were treated on the upper surfaces (approx. 17-19 µg per leaf) of the 5th and 6th leaves with [^{14}C-benzylic] or [^{14}C-cyano] (2*S*,α*R,S*)fenvalerate. At various intervals after treatment leaves and untreated shoots from the treated plants were harvested for analysis (Ohkawa *et al*., 1979). Most of the residue was in the surface rinse or was extractable and was mainly the parent compound (Table 6). A maximum of 4.1% of the dose was found in untreated parts of the plant, demonstrating little translocation. Minor amounts (<2% each) of metabolites were identified.

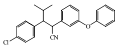
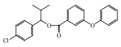
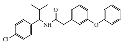
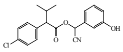
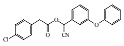
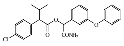
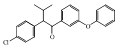
Table 6. Nature of the residue in cabbage leaves after treatment with ^{14}C-labelled (2*S*,α*R,S*)fenvalerate (Ohkawa *et al*. 1979).

Days after treatment	Residues as % of applied ^{14}C					
	Treated leaves					Plant parts other than treated leaves (shoots and roots)
	Surface wash	Fenvalerate in wash	Extractable ^{14}C	Fenvalerate in extract	Bound ^{14}C	Extractable + bound ^{14}C
[^{14}C-cyano](2S,α*R,S*)fenvalerate						
3	40	39	31	29	5.0	1.1
6	40	37	32	30	4.1	0.9
12	11	10	39	36	7.2	2.0
24	11	10	44	39	2.6	1.7
48	1.7	1.0	18	13	5.2	4.1
[^{14}C-benzylic](2S,α*R,S*)fenvalerate						
3	46	44	27	24	3.7	0.4
6	44	42	29	25	3.3	0.2
12	15	13	43	33	4.4	0.3
24	9.8	8.2	37	23	4.7	0.9
48	4.8	3.3	25	14	5.9	0.5

Two boxes (60×60 cm) of lettuce plants were treated with [^{14}C-chlorophenyl]fenvalerate and [^{14}C-phenoxybenzyl]fenvalerate at 10.8 mg/box (Roberts, 1977). They were treated a second time 14 days later and then harvested 12 days later. Fenvalerate constituted 81% (0.94 mg/kg) and 72% (0.80 mg/kg) of the total ^{14}C from the phenoxybenzyl and chlorophenyl labels respectively. CPIA at 0.03 mg/kg was the only other identified component. The remainder of the ^{14}C was in polar material or unextracted. Enzyme hydrolysis released small amounts of 3-phenoxybenzoic acid, 3-phenoxybenzyl alcohol, 3-phenoxybenzaldehyde and CPIA.

Compound leaves of tomato plants were treated separately with 250 µg of [^{14}C-chlorophenyl]fenvalerate or [^{14}C-phenoxybenzyl]fenvalerate (Ehmann, 1979a). The leaves were taken from the plants 32-48 days later, surface-rinsed with chloroform and then ground (mortar and pestle) and extracted with acetone. Most of the radiolabel was present in the surface rinse (96 and 97%). Transformation products in the rinse were identified by TLC and GC-MS. Fenvalerate was the major component of the residue (Table 7).

Table 7. Composition of the residue in the surface rinse of tomato leaves (Ehmann, 1979a) and soya bean leaves (Ehmann, 1979d) treated with radiolabelled fenvalerate.

		Tomato leaves		Soya bean leaves	
		^{14}C-chlorophenyl label	^{14}C-phenoxybenzyl label	^{14}C-chlorophenyl label	^{14}C-phenoxybenzyl label
fenvalerate		84%	79%	80%	84%
SD 54597		5.2%	6.7%	11.7%	8.5%
SD 53036		1.5%	6.5%	0.8%	0.5%
SD 55886		2.2%	1.5%	3.0%	1.3%
SD 58086		1.0%	0.5%	1.0%	1.5%
WL 45329		0.6%	0.2%	0.2%	0.1%
SD 47117		0.8%	1.5%		
SD 55888				0.2%	0.1%

Green tomato fruits were treated separately with 250 µg of [^{14}C-chloropheny] or [^{14}C-phenoxybenzyl]fenvalerate (Ehmann, 1979b). The ripe tomatoes were harvested 20 days after treatment for analysis. The intact tomatoes were subject to a surface rinse with chloroform. The surface-rinsed tomatoes were dipped into boiling water to assist separation of skin and pulp. Skins and pulp were extracted separately and successively with a number of solvent mixtures. Most of the radiolabel was in the surface rinse (88% from chlorophenyl label and 82% from the phenoxybenzyl label). Approximately 94% of the radiolabel in the surface rinse was fenvalerate, with 5-6% as the photodecarboxylated product SD 54597.

Developing soya bean pods were treated separately with 87 µg of [^{14}C-chlorophenyl] or [^{14}C-phenoxybenzyl]fenvalerate (Ehmann, 1979c). The mature pods were harvested 35 days after treatment for analysis. Seeds were carefully removed from the pods to avoid or minimise contamination from surface residue. The pods were thoroughly rinsed with acetone to yield the surface rinse, then macerated and extracted thoroughly with various solvent mixtures. 84 and 81% the applied radiolabel was recovered. Most of the radiolabel was found in the surface rinse; 97% and 94%, and 95% and 96% of this was fenvalerate and 1.2% and 0.7% was SD 55886. Other identified products, each present at less than 1%, in the surface rinse were SD 58086, SD 54597 and SD 53036. The level of radiolabel in the seeds was very low (marginally above background).

Leaves of soya bean plants were treated separately with 250 µg of [^{14}C-chlorophenyl] or [^{14}C-phenoxybenzyl]fenvalerate (Ehmann, 1979d). The leaves were harvested from the plants 34 days later, separated into senescent and non-senescing leaves and surface-rinsed with chloroform. Approximately 80% of the radiolabel was present in the surface rinse, with a further 12-15% extractable with organic solvents. Most of the radiolabel in the rinse was in fenvalerate; other components were similar to the identified components from the surface of tomato leaves (Table 7).

Leaves of outdoor apple trees (James Grieve variety) were treated on 3 occasions with either [^{14}C-chlorophenyl] or [^{14}C-phenoxybenzyl]fenvalerate at intervals of 25 and 37 days (Standen, 1978). Apple fruits were similarly treated, but only twice at an interval of 24 days. A total of 6.1 and 6.7 mg fenvalerate was applied to 100 leaves, and 4.8 and 4.9 mg fenvalerate to 100 apples on each tree. Leaves and apples were harvested 26 and 22 days respectively after the final treatments for analysis. In the apples approximately 98% of the radiolabel was in the peel and 87% of the phenoxybenzyl label and 93% of the chlorophenyl label in the whole apples was present in the peel as fenvalerate. Hydrophilic polar material and unextracted ^{14}C accounted for 6.6% and 3.8%. The parent compound accounted for 75% of the phenoxybenzyl label and 84% of the chlorophenyl label in the leaves. The residue consisted mainly of fenvalerate present on the surface.

Kidney bean seedlings, 14 days old, were treated on the upper surfaces of two primordial leaves (approx. 10 µg on 20 cm^2) with ^{14}C-carbonyl- and ^{14}C-benzylic-labelled (2*S*,α*R*,*S*)fenvalerate and [^{14}C-cyano]fenvalerate. At 30 and 60 days after treatment the seedlings were harvested (Ohkawa *et al.* 1979). Most of the residue was extractable and the majority was the parent compound. A maximum of 5.7% of the dose (no fenvalerate) was found in untreated parts of the plant, demonstrating no translocation of the parent compound. The major identified metabolites were conjugates (Table 8). Minor amounts of other related metabolites were also identified.

Table 8. Nature of the residue in bean leaf tissues after treatment with ^{14}C-labelled fenvalerate and (2*S*,α*R*,*S*)fenvalerate (Ohkawa *et al.* 1979).

	Residues as % of applied ^{14}C					
	Treated leaves					Plant parts other than treated leaves (shoots and roots)
Days after treatment	Extractable ^{14}C	Fenvalerate	Cl-Vacid-conj	PBalc-CN-conj	Bound ^{14}C	Extractable + bound ^{14}C
[^{14}C-cyano]fenvalerate						
30	73	70		2.8	3.7	3.8
60	62	58		2.8	4.6	4.8
[^{14}C-carbonyl] (2*S*,α*R*,*S*)fenvalerate						
30	84	81	8.2		2.7	4.7
60	79	75	4.6		4.6	5.7
[^{14}C-benzylic] (2*S*,α*R*,*S*)fenvalerate						
30	89	85		5.5	4.1	1.7
60	83	76		4.8	6.7	2.4

Two soils (a light clay and a sandy loam) were treated at 1 mg/kg with [^{14}C-benzylic] and [^{14}C-cyano](2*S*,α*R*,*S*)fenvalerate and incubated at 25°C in the dark for 14 days, when 14-day old kidney bean seedlings were transplanted into the soil and grown for 30 days at 25°C (Ohkawa *et al.* 1979). The ^{14}C content was measured in roots (140-360 µg/kg-some soil contamination), shoots (14-23 µg/kg) and pods and seeds (3-8 µg/kg). No parent compound was found in the shoots. The experiment demonstrated that there was little uptake of fenvalerate from soil or translocation to edible portions.

Spring wheat plants in pots, approximately 30 days after germination, were treated by atomiser spray over the entire foliage with either [^{14}C-chlorophenyl] or [^{14}C-phenoxybenzyl]fenvalerate at a rate approximating 1.1 kg ai/ha (Lee, 1985). The soil surface was covered during the treatment to minimise contamination of the soil. Treated plants were exposed to the environment for 77 days. Plants were sampled immediately and then at intervals up to 10 weeks after treatment. Harvested foliage was rinsed with hexane and methanol to obtain surface residues. Pulverised tissues were then extracted with acetone. After 10 weeks, 44% and 35% of the initial ^{14}C remained. At each sampling the majority of the ^{14}C was in the hexane rinse, i.e. in non-polar surface residues. The distribution and characterization of the residues are shown in Table 9. ^{14}C levels in the grain from mature plants were below reliable measurement (<0.01 mg/kg).

Table 9. Distribution of residues in wheat foliage after treatment with ^{14}C-labelled fenvalerate (Lee, 1985).

Interval after treatment, weeks	Residues as % of applied ^{14}C				
	Surface residue (hexane extract + MeOH extract)	Fenvalerate (hexane extract + MeOH extract)	SD 54597	Acetone extract of tissue	Unextractable
^{14}C-chlorophenyl					
0	99	98			
2	68	66		4	4
4	54	52		6	7
6	45	41	0.6	6	5

Interval after treatment, weeks	Residues as % of applied ^{14}C				
	Surface residue (hexane extract + MeOH extract)	Fenvalerate (hexane extract + MeOH extract)	SD 54597	Acetone extract of tissue	Unextractable
10	32	30	0.8	4	8
^{14}C-phenoxybenzyl					
0	99	98			
2	65	63		4	3
4	42	40		5	10
6	37	34	0.6	5	7
10	26	22	0.7	1	8

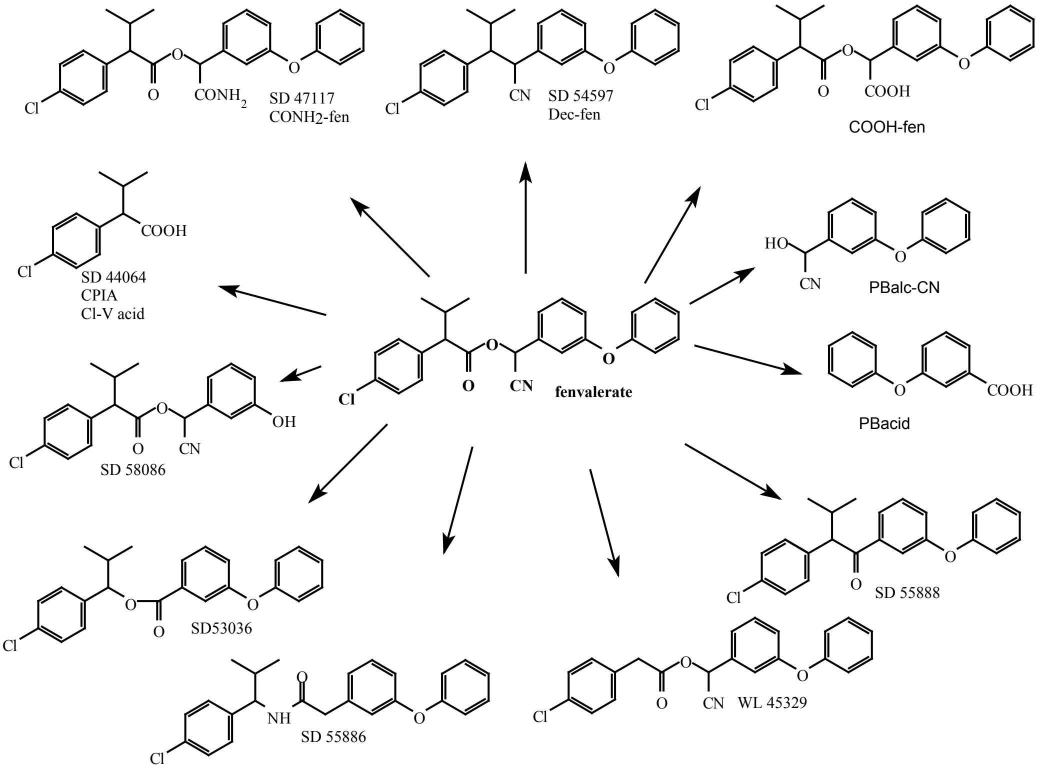


Figure 2. Plant metabolism of fenvalerate.

Environmental fate in soil

The Meeting received information on the soil and solution photolysis, aerobic soil degradation, field dissipation and soil adsorption of esfenvalerate, and on the soil photolysis and aerobic and anaerobic degradation, column leaching of aged residues, field dissipation and uptake during crop rotation of fenvalerate.

Photolysis. Katagi *et al.* (1985a) compared the photolysis of [^{14}C-chlorophenyl]fenvalerate and [^{14}C-chlorophenyl]esfenvalerate on a Sapporo clay loam (48% sand, 28% silt, 24% clay, 12% organic matter, pH 5.3) and a Chiba sandy clay loam (69% sand, 13% silt, 19% clay, 3.4% organic matter, pH 5.9). The soils were coated on glass plates to a thickness of 0.5 mm and the test compounds were evenly applied to the soil layer at 0.3-0.4 ng/cm^2. The plates were exposed to natural sunlight (July-August in Japan) for periods up to 30 days and then examined for remaining parent compound and photolysis products (Table 10).

The photolysis half-lives of fenvalerate and esfenvalerate were very similar (3-3.9 days calculated from the 0-10 days data). The disappearance rates were highest initially and then continually became slower during the 30 days of the study. The disappearance of the parent compound in the dark controls was substantial for the Chiba soil; after 30 days 29% and 27% of fenvalerate and esfenvalerate respectively had disappeared. Losses of the parent in the Sapporo dark control were less, with 71% of both fenvalerate and esfenvalerate remaining after 30 days. The major product of photolysis was the amide produced from the -CN group. Little isomerization occurred at either the α- or 2- positions.

Table 10. Soil surface photolysis of [^{14}C-chlorophenyl]esfenvalerate and [^{14}C-chlorophenyl]fenvalerate (Katagi *et al.,* 1985a).

	% of applied ^{14}C							Half-life (from 0-10 days data), days
	Days after treatment							
	0	1	3	5	10	20	30	
Fenvalerate on Sapporo clay loam								
Fenvalerate	92	54	26	18	8	3	2	3.0
CONH$_2$-fen	<0.1	7.5	16	18	19	18	17	
CPIA	0.7	2.8	3.1	3.6	4.5	3.3	4.0	
Bound ^{14}C	0.5	29	45	47	43	53	49	
Fenvalerate on Chiba sandy clay loam								
Fenvalerate	91	66	36	28	14	6	3	3.9
CONH$_2$-fen	<0.1	19	35	40	48	39	36	
CPIA	0.8	2.5	3.0	3.5	3.4	4.3	3.8	
Bound ^{14}C	2.7	5.2	11	12	12	12	13	
Esfenvalerate on Sapporo clay loam								
Esfenvalerate	94	50	24	16	9	3	2	3.1
CONH$_2$-fen	<0.1	7.3	16	18	20	17	13	
CPIA	1.5	2.6	3.5	3.5	4.1	4.0	4.5	
Bound ^{14}C	0.4	29	44	48	48	43	40	
Esfenvalerate on Chiba sandy clay loam								
Esfenvalerate	96	63	39	28	15	5	4	3.9
CONH$_2$-fen	<0.1	17	34	40	41	38	40	
CPIA	0.7	2.0	3.3	3.7	3.5	3.7	3.8	
Bound ^{14}C	0.1	5.5	11	11	11	13	13	

Katagi *et al.* (1985b) examined the photoisomerization of [^{14}C-phenoxybenzyl]esfenvalerate under UV irradiation ($\lambda > 290$ nm) in solution (benzene, hexane, methanol, aqueous buffers at pH 3.9, 7.2 and 9.9) and as a thin film on glass, silica gel and soil. The concentration of esfenvalerate in the solutions for irradiation was 2 mg/l. Surface layers were irradiated for 60 minutes, and solutions for 90 minutes. Estimated half-lives for the disappearance of esfenvalerate from irradiated thin films were silica gel >10 hours, glass 10 hours, soils 10, 4 and 7 hours. The results of photolysis in solution are shown in Table 11.

The main photoproduct from solution photolysis in benzene, methanol, hexane and buffered solutions was dec-fen. Virtually no epimerization was observed at either chiral position in the absence of photosensitizers. Triethylamine in benzene accelerated the epimerization of the α-*S* to the α-*R* isomer, but this occurred in the dark as well as under UV. A similar conversion occurred in the pH 9.9 buffer, in the dark as well as under UV. The authors concluded that the epimerization of esfenvalerate induced by sunlight would generally be minor.

Table 11. Disappearance of esfenvalerate and degree of photoisomerization during photolysis in aqueous and organic solutions and in the presence of photosensitizers (Katagi *et al.,* 1985b).

Conditions		% of esfenvalerate measured at time 0					Half-life, hours
		Irradiation periods, minutes					
		10	20	30	60	90	
Aq buffer, pH 3.9, esfenvalerate	UV	85	88	83	74	68	3.1
Aq buffer, pH 3.9, esfenvalerate	dark			90	93	91	
Aq buffer, pH 7.2, esfenvalerate	UV	95	94	90	83	80	4.7
Aq buffer, pH 7.2, *2S,αR* epimer	UV	0.8	1.0	1.1	0.9	0.6	
Aq buffer, pH 7.2, esfenvalerate	dark			94	97	94	
Aq buffer, pH 9.9, esfenvalerate	UV	94	85	91	86	58	2.3
Aq buffer, pH 9.9, *2S,αR* epimer	UV	5.7	6.7	2.3	1.3	15	
Aq buffer, pH 9.9, esfenvalerate	dark			89	83	90	
Aq buffer, pH 9.9, *2S,αR* epimer	dark			6.9	18	5.8	
Benzene, esfenvalerate	UV	77	82	76	71	58	2.4
Benzene + pyridine, esfenvalerate	UV	95	83	84	83		3.9
Benzene + pyridine, esfenvalerate	dark					95	
Benzene + triethylamine, esfenvalerate	UV	75	76	70	57	50	1.7
Benzene + triethylamine, *2S,αR* epimer	UV	17	6.7	18	17	19	
Benzene + triethylamine, esfenvalerate	dark					66	
Benzene + triethylamine, *2S,αR* epimer	dark					24	
Methanol, esfenvalerate	UV	96	92	89	78	68	2.8
Hexane, esfenvalerate	UV	95	92	88	80	74	3.5
Hexane + acetone, esfenvalerate	UV	88	88	88	83	76	4.7
Hexane + acetophenone, esfenvalerate	UV	95	94	92	91	89	10
Hexane + benzophenone, esfenvalerate	UV	92	90	85	86	83	6.7
Hexane + isobutyrophenone, esfenvalerate	UV	94	92	92	89	88	9.3
Hexane + benzil, esfenvalerate	UV	94	93	93	91	89	12
Hexane + naphthalene, esfenvalerate	UV	96	96	96	95	94	>15
Hexane + pyrene, esfenvalerate	UV	95	96	96	95	93	>15
Hexane + perylene, esfenvalerate	UV	95	93	92	89	83	6.6

Aerobic degradation. Itoh *et al* (1995) incubated [^{14}C-carbonyl]esfenvalerate and [^{14}C-carbonyl]fenvalerate at 0.51 and 1.5 mg/kg dry weight respectively in German soil 2.1 (92% sand, 4.4% silt, 3.5% clay, 0.7% organic carbon, pH 5.9), in German soil 2.2 (89% sand, 5.6% silt, 5.1% clay, 2.3% organic carbon, pH 5.8) and in a UK sandy loam (80% sand, 12% silt, 8.3% clay, 2.3% organic carbon, pH 5.3) under aerobic conditions at 20°C in darkness for 100 days. Recovery of ^{14}C, including volatiles, was in the range 92-110%. The results are shown in Table 12. Estimated half-lives of esfenvalerate were in the range 36-59 days and of fenvalerate 35 and 48 days. Initial disappearance rates were higher than the long-term rates. Mineralization of the carbonyl group was quite rapid, with half-lives of 42 to 85 days for esfenvalerate and 45 and 65 days for fenvalerate. The behaviour of esfenvalerate and fenvalerate was very similar. The configuration of esfenvalerate was not changed.

Table 12. Loss of parent compound and production of CO_2 during aerobic incubation of [^{14}C-carbonyl]esfenvalerate and [^{14}C-carbonyl]fenvalerate (Itoh *et al*., 1995).

	% of applied ^{14}C					Estimated half-life, days
	Days incubation					
	0	15	29	58	100	
Esfenvalerate, Soil 2.1, 50% water-holding capacity						
CO_2		16	29	46	58	85 [1]
Esfenvalerate	91	68	58	40	27	59
Bound ^{14}C	0.3	2.4	3.3	4	5.3	
Esfenvalerate, Soil 2.1, 80% water-holding capacity						
CO_2		22	38	58	72	59 [1]
Esfenvalerate	92	67	49	29	18	43
Bound ^{14}C	0.3	2.6	3.9	4.6	5.8	

	% of applied ^{14}C					
	Days incubation					
Esfenvalerate, Soil 2.2, 50% water-holding capacity						
CO_2		38	55	73	82	48 [1]
Esfenvalerate	92	51	35	21	15	40
Bound ^{14}C	1.1	2.9	3.4	3.6	3.3	
Esfenvalerate, Soil 2.2, 80% water-holding capacity						
CO_2		44	63	78	87	42 [1]
Esfenvalerate	91	48	30	18	12	36
Bound ^{14}C	1.1	3.6	3.8	4.1	3.8	
Fenvalerate, Soil 2.2, 50% water-holding capacity						
CO_2		25	40	58	70	65 [1]
Fenvalerate	94	69	49	29	22	48
Bound ^{14}C	0.3	2.6	3.3	3.7	3.6	
Fenvalerate, Soil 2.2, 80% water-holding capacity						
CO_2		35	53	72	83	45 [1]
Fenvalerate	94	60	34	20	13	35
Bound ^{14}C	1.1	3.8	4.5	5	4.8	
Esfenvalerate, Soil UK, 50% water-holding capacity						
days	0	14	28	60	101	
CO_2		18	33	53	65	71 [1]
Esfenvalerate	96	71	54	32	23	49
Bound ^{14}C	1.4	3.2	3.6	4.2	4.9	

[1] mineralization half life of the carbonyl group in fenvalerate or esfenvalerate.

Nambu and Yoshimura (1988) incubated [^{14}C-phenoxybenzyl]esfenvalerate at 1.0 mg/kg in a Kuki sandy loam (59% sand, 34% silt, 7% clay, 1.9% organic matter, pH 6.4), a Kodaira sandy loam (56% sand, 30% silt, 15% clay, 14% organic matter, pH 7.1), a Noichi sandy loam (55% sand, 26% silt, 19% clay, 3.3% organic matter, pH 7.0) and a Maebashi loamy sand (78% sand, 18% silt, 4.2% clay, 5.5% organic matter, pH 5.6) in duplicate under aerobic conditions at 15°C for 6 months. Recoveries of ^{14}C, including volatiles, were in the range 91-102%. The results are shown in Table 13.

The half-life for the disappearance of esfenvalerate in three of the soils was 80-90 days and in the Noichi sandy loam approximately 170 days. The half-life for mineralization ($^{14}CO_2$ evolution) was approximately 300 days in three of the soils and approximately 800 days in the Noichi sandy loam. In each case between 19 and 39% of the applied ^{14}C remained bound after 6 months, mostly in the humic acid and humin fractions.

Table 13. Aerobic degradation of [^{14}C-phenoxybenzyl]esfenvalerate incubated in 4 soils at 15°C for 6 months (Nambu and Yoshimura, 1988).

Compound	% of ^{14}C dose						
	Day 0	Day 14	Day 30	Day 60	Day 90	Day 120	Day 180
Kuki sandy loam, expt A							
Esfenvalerate	92	81	64	47	41	28	23
CO_2		12	16	25	32	34	36
$CONH_2$-fen	<0.1	0.3	0.8	0.8	0.7	0.6	0.4
PBacid	nd	1.0	1.5	1.3	1.0	0.9	0.5
4'-OH-fen	0.2	0.3	0.7	0.3	0.3	0.3	0.2
Bound ^{14}C	0.4	5.5	14	22	26	28	30
Kuki sandy loam, expt B							
Esfenvalerate	91	69	59	27	34	22	18
CO_2		11	18	35	41	43	47
$CONH_2$-fen	<0.1	0.8	1.2	0.8	0.3	0.6	0.5
PBacid	nd	1.8	1.7	1.1	0.7	0.8	0.5
4'-OH-fen	0.2	0.9	0.4	0.4	0.8	0.3	0.2

Compound	% of ^{14}C dose						
	Day 0	Day 14	Day 30	Day 60	Day 90	Day 120	Day 180
Bound ^{14}C	0.4	13	16	32	30	32	33
Kodaira sandy loam, expt A							
Esfenvalerate	91	70	44	33	27	26	20
CO$_2$		12	24	33	36	37	42
CONH$_2$-fen	<0.1	0.5	1.0	0.7	0.3	0.6	0.5
PBacid	nd	1.2	1.4	0.7	0.6	0.6	0.2
4'-OH-fen	0.2	0.3	0.3	0.3	<0.1	0.2	0.1
Bound ^{14}C	1.8	13	25	31	34	25	35
Kodaira sandy loam, expt B							
Esfenvalerate	89	60	45	29	26	19	17
CO$_2$		12	25	32	35	38	42
CONH$_2$-fen	<0.1	1.1	1.3	1.1	0.9	0.6	0.6
PBacid	nd	1.6	1.4	0.6	0.4	0.3	0.2
4'-OH-fen	0.1	0.6	0.4	0.2	0.2	0.1	0.1
Bound ^{14}C	1.7	19	24	32	34	33	39
Noichi sandy loam, expt A							
Esfenvalerate	93	87	81	76	68	58	51
CO$_2$		11	12	13	17	19	22
CONH$_2$-fen	<0.1	0.2	0.8	1.3	1.0	1.4	1.3
PBacid	nd	0.6	0.5	0.6	0.6	0.6	0.5
4'-OH-fen	0.2	0.2	0.4	0.4	0.5	0.5	0.4
Bound ^{14}C	0.5	2.3	5.0	8.7	13	15	19
Noichi sandy loam, expt B							
Esfenvalerate	91	83	76	67	62	50	40
CO$_2$		11	12	15	17	20	24
CONH$_2$-fen	0.1	0.7	1.4	1.9	1.1	1.8	1.6
PBacid	nd	0.5	0.7	0.8	0.6	0.5	0.4
4'-OH-fen	0.2	0.3	0.6	0.4	nd	0.3	0.4
Bound ^{14}C	0.5	3.8	7.3	13	16	20	24
Maebashi loamy sand, expt A							
Esfenvalerate	92	62	65	33	25	20	17
CO$_2$		13	17	38	42	45	48
CONH$_2$-fen	<0.1	2.0	1.0	0.9	0.6	0.6	0.6
PBacid	nd	1.0	0.6	0.5	0.2	0.2	0.2
4'-OH-fen	0.2	1.4	1.4	1.3	0.2	0.5	0.4
Bound ^{14}C	0.4	12	10	23	26	27	27
Maebashi loamy sand, expt B							
Esfenvalerate	91	81	71	56	46	37	28
CO$_2$		6.4	12	21	25	28	32
CONH$_2$-fen	<0.1	0.7	1.4	1.7	1.6	1.6	1.1
PBacid	nd	0.5	0.5	0.5	0.6	0.3	0.2
4'-OH-fen	0.2	1.3	1.4	1.0	1.3	0.5	0.4
Bound ^{14}C	0.4	4.7	8.6	15	20	21	29

Sakata *et al.* (1985) compared the degradation of [^{14}C-benzylic]esfenvalerate and its isomers under aerobic conditions at 25°C in an Azuchi sandy clay loam (63% sand, 17% silt, 21% clay, 0.4% organic matter and pH 5.5) and a Kodaira loam (62% sand, 31% silt, 8% clay, 13.2% organic matter and pH 4.4). The half-lives for degradation and mineralization of esfenvalerate were quite similar to those of its isomers (Table 14). The conditions did not cause epimerization.

The results suggest that the degradation rate of esfenvalerate is likely to be similar to that of fenvalerate. The nature and levels of degradation products were also similar among the four isomers, with their levels generally low; the highest level was 6.4% of the applied ^{14}C.

Table 14. Degradation of esfenvalerate and its isomers in two soils under aerobic conditions at 25°C (Sakata *et al.,* 1985).

Compound	Kodaira soil					Azuchi soil				
	% of ^{14}C dose				*half-life, weeks*	% of ^{14}C dose				*half-life, weeks*
	2 weeks	4 weeks	8 weeks	12 weeks		2 weeks	4 weeks	8 weeks	12 weeks	
esfenvalerate	67	52	21	13	*4.1*	82	68	54	45	*11.6*
2*S*, α*R*	81	76	48	32	*7.1*	84	77	63	54	*15.4*
2*R*, α*S*	53	27	16	12	*5.0*	52	34	20	14	*5.3*
2*R*, α*R*	79	68	46	36	*8.6*	69	51	30	24	*6.5*
Mineralization. Half-life is mineralization half-life.										
	% of ^{14}C dose appearing as CO_2				*half-life weeks*	% of ^{14}C dose appearing as CO_2				*half-life weeks*
esfenvalerate	11	24	45	57	*9.5*	6.7	14	26	36	*18*
2*S*, α*R*	3.5	6.6	17	24	*29*	3.5	7.3	14	21	*35*
2*R*, α*S*	19	37	52	57	*11*	25	40	54	58	*12*
2*R*, α*R*	4.3	11	20	25	*28*	13	24	38	45	*15*

Lee *et al.* (1985a) compared the aerobic degradation of [^{14}C-phenoxybenzyl]fenvalerate and esfenvalerate. The compounds were incubated at doses of 20 mg/kg fenvalerate and 5 mg/kg esfenvalerate in a silty loam soil (36% sand, 48% silt, 16% clay, 1.3% organic matter, pH 5.7) under aerobic conditions at 25°C in darkness for 90 days. Recoveries of ^{14}C, including volatiles, were in the range 96-103%. The results are shown in Table 15. The *S,R-* and *R,R-* isomers are more persistent than the other two and constitute a higher proportion of the aged residue. The degradation rate of the *S,S-* isomer in fenvalerate (half-life 95 days) is close to the degradation rate in esfenvalerate (half-life 74 days). Under these conditions the content of the *S,S-* isomer in the esfenvalerate residue was consistently 96-98% of the total, demonstrating that epimerization did not occur. After 90 days 14% of the fenvalerate and 21% of the esfenvalerate had been evolved as CO_2.

Table 15. Comparative aerobic soil degradation of ^{14}C-phenoxybenzyl-labelled fenvalerate and esfenvalerate (Lee *et al.,* 1985).

Day	Concentration, mg/kg					
	fenvalerate					esfenvalerate
	Total	*S,R-*	*R,S-*	*S,S-*	*R,R-*	*S,S-*
0	20	5.4	5.6	5.0	4.8	4.9
14	20	5.2	5.2	4.8	4.8	4.2
30	17	4.9	4.2	4.0	4.3	3.8
60	15	4.4	3.3	3.3	4.1	2.5
90	12	3.6	2.5	2.6	3.3	2.2
Half-life, days	117	156	76	95	176	74

Lee (1979a) incubated [^{14}C-chlorophenyl]fenvalerate at 5 mg/kg in a California sandy loam (82% sand, 11% silt, 7% clay, 1.1% organic matter, pH 7.3), a Louisiana commerce loam (22% sand, 71% silt, 7% clay, 0.24% organic matter, pH 5.7) and a Catlin silty clay loam (20% sand, 63% silt, 28% clay, 2.0% organic matter, pH 5.3) under aerobic conditions at 23°C for 90 days. In an anaerobic study the dosed soils were incubated aerobically for 30 days before waterlogging and establishment of anaerobic conditions. Lee (1979b) extended the aerobic study to 12 months. Recoveries of ^{14}C, including volatiles, were in the range 88-101%. The results are shown in Table 16.

The half-life of fenvalerate was 90 days and longer. Behaviour under aerobic and anaerobic conditions was similar. In 12 months, mineralization of the chlorophenyl group was 51%, 14% and 5.0% in the three soils.

Table 16. Aerobic and anaerobic degradation of [^{14}C-chlorophenyl]fenvalerate incubated in 3 soils (Lee, 1979a,b).

Compound	% of applied ^{14}C									
	Aerobic								*Anaerobic*	
	Day 0	Day 15	1 month	2 months	3 months	6 month	9 months	12 months	*Day 30*	*Day 60*
California sandy loam										
fenvalerate	99	87	71	54	50	27	18	17	*63*	*57*
CO_2		2.1	5.5	11.9	20	32	47	51	*6.1*	*4.5*
SD 48838		0.35	3.4	4.3	1.9	3.3	1.5	1.1	*4.4*	*1.0*
SD 44064		0.32	2.2	3.0	1.3	0.43	0.36	2.6	*4.2*	*3.0*
SD 47117			1.4	2.3	1.1	0.29	0.20	1.8	*1.1*	*0.75*
SD 53065			1.1	1.1	0.57	0.43	0.48	0.10	*0.75*	*0.22*
Commerce loam										
fenvalerate	98	92	80	80	78	62	57	32	*80*	*68*
CO_2		0.41	1.2	2.3	4.1	4.2	4.6	5.0	*0.55*	*1.7*
SD 48838		0.43	3.2	3.1	1.3	1.0	0.78	-	*1.6*	*1.1*
SD 44064		0.25	2.3	1.4	1.2	1.0	1.2	3.1	*2.8*	*2.5*
SD 47117		0.37	1.7	0.94	1.1	1.9	2.3	32	*1.3*	*1.1*
SD 53065			0.38	0.41	0.25	0.36	0.50	0.70	*0.76*	*0.36*
Silty clay loam										
fenvalerate	97	74	72	56	55	33	22	12	*65*	*57*
CO_2		0.53	1.6	3.3	7.7	9.4	12.8	14.3	*3.8*	*3.8*
SD 48838		2.5	2.1	2.8	1.1	1.4	0.89	0.16	*1.7*	*1.4*
SD 44064		1.3	2.5	2.6	1.5	1.2	0.77	1.7	*3.8*	*3.6*
SD 47117		0.37	0.56	0.93	1.1	0.98	1.2	10	*0.55*	*0.32*
SD 53065			0.35	0.40	0.74	0.36	0.25	0.89	*0.36*	*0.12*

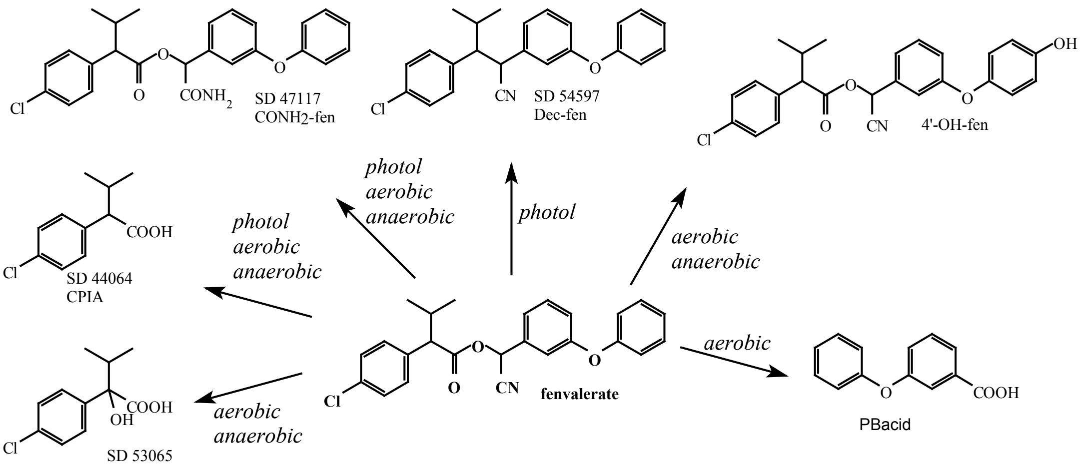


Figure 3. Fenvalerate, photolysis and degradation in soil.

The adsorption and desorption of [^{14}C-phenoxybenzyl]esfenvalerate on 6 soils at 20-25°C in 0.01M calcium chloride solution were measured using batch equilibration (Ohm, 2001). The study design followed US EPA and OECD guidelines. Polypropylene copolymer bottles were used to contain the test solutions and some problems were experienced with sorption to the vessel walls. The soil:solution ratio was 1:20 and the concentration of [^{14}C-phenoxybenzyl]esfenvalerate in the test solution 1 ng/ml. In preliminary tests very little desorption (2% and 6.7%) occurred and was not further evaluated. Esfenvalerate was stable under the test conditions. Adsorption K_d and K_{oc} values (Table 17) indicate that esfenvalerate will be highly immobile in soils.

Table 17. Adsorption of [^{14}C-phenoxybenzyl]esfenvalerate on 6 soils at 20-25°C in 0.01M calcium chloride solution (Ohm, 2001).

Soil	Soil properties						Adsorption	
	% organic carbon	CEC meq/100 g	% sand	% silt	% clay	pH	K_d ml/g	K_{oc} ml/g
Commerce silt loam	0.2	6.7	33	56	11	7.0	750	375,00
Donna sandy clay loam	0.5	15.6	54	25	20	8.1	700	140,00
Hanford sandy loam	0.7	9.0	75	18	7	8.5	600	85,70
Matapeake loam	1.2	6.8	35	47	18	5.2	1700	141,70
Drummer silty clay loam	2.6	26.3	8.4	61	31	4.8	15,50	596,20
Sassafras loamy sand	3.0	10.8	84	14	2	4.8	5,20	171,70

Jackson and Roberts (1976) examined the leaching of fresh and 4-weeks aged [^{14}C-phenoxybenzyl]fenvalerate residues on a sandy loam soil. The glass columns measured 45 mm in diameter and held 880 g soil to a depth of 320 mm. Water was applied to the top of the columns at the rate of 2 ml/hour for 45 days. Column eluates contained less than 2% of the applied radiolabel. Most of the ^{14}C remained in the top 2 cm of soil. The conclusions from this study are that fenvalerate and its soil degradation products have little tendency to be leached.

In a field dissipation study, esfenvalerate as an EC formulation was applied once at 2 field sites to bare soil in Kent and Cambridgeshire in the UK in July 1994 at a rate of 0.10 kg ai/ha (Burden, 1998). The soils were classified as sandy silt loam (pH 7.2, organic matter 1.9%) and clay (pH 7.1, 3.8% organic matter) respectively. At the Cambridgeshire site rainfall was 30% higher than average, but consistently heavy falls did not occur until 2 months after the study began, so did not affect samples collected earlier. At the Kent site rainfall was close to the yearly average. The 5×7 m plots were kept free of weeds by application of glyphosate. Soil cores (10 and 30 cm depth) were taken at intervals and (5 cm segments) examined for residues of esfenvalerate and its isomers and degradation products. The results are shown in Table 18.

No residues were detected below 5 cm. On day 28 the isomer composition of the residue was determined. The 2-*S*, α-*R* isomer was present at 0.01 mg/kg (the LOQ) in replicates 2 and 3 from the Kent site, and in the tank mix at approximately 11-13% of the total esfenvalerate; so its presence in the residue probably resulted from the application. The other isomers were not detected. Samples from both sites taken at 3, 6 and 9 months were analysed for the amide degradation product ($CONH_2$-fen) but it was not detected (LOQ <0.01 mg/kg).

Over the 12 months of the study at the two sites esfenvalerate decreased to 11% or less of the starting concentrations and its residues did not move down the soil profile.

A parallel field dissipation study was carried out in October 1994 at the same two sites with the same experimental parameters (Burden, 1997). The results were essentially the same as in the first study. The results are shown in Table 19. No epimerization was observed in samples taken on day 28. Esfenvalerate concentrations at the two sites decreased to 6% or less of the starting concentrations and the residues did not move down the soil profile.

Table 18. Field dissipation of esfenvalerate at two sites in the UK with application in summer at 0.10 kg ai/ha (Burden, 1998).

Interval	Esfenvalerate, mg/kg in 0-5 cm depth, expressed on soil dry weight					
	Sandy silt loam (Kent)			Clay (Cambridgeshire)		
	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3
0 day	0.12	0.16	0.15	0.21	0.072	0.087
3 days	0.14	0.14	0.10	0.18	0.069	0.059
7 days	0.15	0.15	0.087	0.090	0.080	0.074
2 weeks	0.064	0.087	0.066	0.097	0.079	0.021
4 weeks	0.084	0.054	0.066	0.036	0.039	0.042
2 months	0.037	0.026	0.047	0.070	0.078	0.038
3 months	0.027	0.018	0.016	0.056	0.052	0.039
6 months	0.009	0.017	0.008	<0.005	0.012	<0.005
9 months	0.009	0.019	0.009	0.010	<0.005	0.008
12 months	<0.005	<0.005	<0.005	0.006	<0.005	0.010
Estimated half-life, days	64	86	67	82	78	127

Table 19. Field dissipation of esfenvalerate at two sites in the UK with application in autumn at 0.10 kg ai/ha (Burden, 1997).

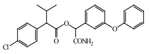
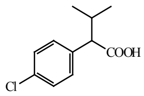
Interval	Esfenvalerate, mg/kg in 0-5 cm depth, expressed on soil dry weight					
	Sandy silt loam (Kent)			Clay (Cambridgeshire)		
	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3
0 day	0.16	0.20	0.17	0.22	0.21	0.28
3 days	0.088	0.095	0.12	0.090	0.13	0.14
7 days	0.11	0.12	0.082	0.33	0.18	0.30
2 weeks	0.076	0.068	0.095	0.14	0.16	0.13
4 weeks	0.062	0.032	0.083	0.078	0.12	0.13
2 months	0.044	0.066	0.078	0.11	0.065	0.093
3 months	0.046	0.015	0.057	0.060	0.046	0.062
6 months	0.026	0.019	0.044	0.037	0.035	0.029
9 months	<0.005	0.007	0.007	0.013	0.013	0.007
12 months	<0.005	<0.005	<0.005	<0.005	0.012	0.009
Estimated half-life, days	86	70	77	76	88	71

Schneiders (1989) reported the field dissipation of esfenvalerate at 3 sites in the USA (Table 20) and of fenvalerate at sites close to two of them (Table 21). At one site (Donna, TX) residues of esfenvalerate were at or below the LOQ even on day 0. At the Yuma AZ site, esfenvalerate was present in the surface layer at day 30, but was not detected below 75 mm. SD47117 ($CONH_2$-fen) did not exceed the LOQ (0.01 mg/kg) at any time. SD44064 (CPIA) was present in the surface layer at day 30. At the Montgomery AL site, esfenvalerate was present in the surface layer and in the 75-150 mm layer on day 0, but did not persist to day 30. Levels of the two degradation products were below the LOQ (0.01 mg/kg) at all times.

In the studies of fenvalerate dissipation at the other AL and AZ sites fenvalerate residues were higher and persisted above the LOQ longer, but its application rates were higher so the starting concentrations would be expected to be higher.

Table 20. Esfenvalerate residues in soil resulting from field dissipation in the USA, 1985 (Schneiders, 1989).

Locality	Soil	Cover crop	Application	Analyte and sample depth	Interval since final applicn, days	Residues, mg/kg	Study no.

			Rate g ai/ha	No.	Final applicn.				
Donna, TX	sandy clay loam	cotton	56	10	Aug	esfenvalerate 0-75 mm	0 30 90 323	0.01 <0.01 <0.01 <0.01	MO-RIR-24-210-85
						esfenvalerate 75-150 mm	0 30 90 323	<0.01 <0.01 <0.01 <0.01	
						esfenvalerate 150-300 mm	0 30 90 323	<0.01 <0.01 <0.01 <0.01	
						SD47117 0-75 mm 	0 30 90 323	<0.01 <0.01 <0.01 <0.01	
						SD47117 75-150 mm	0 30 90 323	<0.01 <0.01 <0.01 <0.01	
						SD47117 150-300 mm	0 30 90 323	<0.01 <0.01 <0.01 <0.01	
						SD44064 0-75 mm 	0 30 90 323	<0.01 <0.01 <0.01 <0.01	
						SD44064 75-150 mm	0 30 90 323	<0.01 <0.01 <0.01 <0.01	
						SD44064 150-300 mm	0 30 90 323	<0.01 <0.01 <0.01 <0.01	
Yuma, AZ	sandy clay loam	cotton	56	11	Oct	esfenvalerate 0-75 mm	0 30 90	0.06 0.03 0.08 <0.01 <0.01 (2)	MO-RIR-24-229-85
						esfenvalerate 75-150 mm	0 30 90	<0.01 (2) <0.01 (2) <0.01 (2)	
						esfenvalerate 150-300 mm	0 30 90	<0.01 (2) <0.01 (2) <0.01 (2)	
						SD47117 0-75 mm	0 30 90	<0.01 (2) <0.01 (2) <0.01 (2)	
						SD47117 75-150 mm	0 30 90	<0.01 (2) <0.01 (2) <0.01 (2)	
						SD47117 150-300 mm	0 30 90	<0.01 (2) <0.01 (2) <0.01 (2)	
						SD44064 0-75 mm	0 30 90	<0.01 (2) 0.04 <0.01 <0.01 (2)	

Locality	Soil	Cover crop	Application			Analyte and sample depth	Interval since final applicn, days	Residues, mg/kg	Study no.
			Rate g ai/ha	No.	Final applicn.				
						SD44064	0	<0.01 (2)	
						75-150 mm	30	<0.01 (2)	
							90	<0.01 (2)	
						SD44064	0	<0.01 (2)	
						150-300 mm	30	<0.01 (2)	
							90	<0.01 (2)	
Montgomery,	sandy	cotton	42	7	Sept	esfenvalerate	0	0.05 0.02	MO-RIR-24-
AL	clay					0-75 mm	30	<0.01 (2)	109-86
	loam						90	<0.01 (2)	
						esfenvalerate	0	0.02 <0.01	
						75-150 mm	30	<0.01 (2)	
							90	<0.01 (2)	
						esfenvalerate	0	<0.01 (2)	
						150-300 mm	30	<0.01 (2)	
							90	<0.01 (2)	
						SD47117	0	<0.01 (2)	
						0-75 mm	30	<0.01 (2)	
							90	<0.01 (2)	
						SD47117	0	<0.01 (2)	
						75-150 mm	30	<0.01 (2)	
							90	<0.01 (2)	
						SD47117	0	<0.01 (2)	
						150-300 mm	30	<0.01 (2)	
							90	<0.01 (2)	
						SD44064	0	<0.02 (2)	
						0-75 mm	30	<0.02 (2)	
							90	<0.02 (2)	
						SD44064	0	<0.02 (2)	
						75-150 mm	30	<0.02 (2)	
							90	<0.02 (2)	
						SD44064	0	<0.02 (2)	
						150-300 mm	30	<0.02 (2)	
							90	<0.02 (2)	

Table 21. Fenvalerate residues in soil resulting from field dissipation in the USA, 1978 (Schneiders, 1989).

Locality	Soil	Cover crop	Application			Analyte and sample depth	Interval since final applicn, days	Residues, mg/kg	Study no.
			Rate g ai/ha	No.	Final applicn				
Shorter, AL	sandy	cotton	220	15	Oct	fenvalerate	0	0.25	TIR-24-135-79
	loam					0-75 mm	15	0.19	
							21	0.27	
							30	0.03	
							45	0.14	
							90	0.09	
							180	<0.01	
						fenvalerate	0	0.08	
						75-150 mm	15	0.07	
							21	0.05	
							30	<0.01	
							45	<0.01	
							90	<0.01	
							180	<0.01	

Locality	Soil	Cover crop	Application			Analyte and sample depth	Interval since final applicn, days	Residues, mg/kg	Study no.
			Rate g ai/ha	No.	Final applicn				
						fenvalerate 150-225 mm	0	0.02	
							15	<0.01	
							21	<0.01	
							30	<0.01	
							45	<0.01	
							90	<0.01	
							180	<0.01	
Maricopa, AZ	sandy loam	cotton	220	15	Oct	fenvalerate 0-100 mm	0	0.19	TIR-24-140-79
							14	0.11	
							21	0.06	
							30	0.08	
							47	0.04	
							80	0.08	
							172	0.12	
						fenvalerate 100-200 mm	0	<0.01	
							14	0.02	
							21	0.05	
							30	<0.01	
							47	0.02	
							80	0.01	
							172	<0.02	
						fenvalerate 200-300 mm	0	<0.01	
							14	<0.01	
							21	0.03	
							30	<0.01	
							47	<0.01	
							80	<0.01	
							172	<0.01	

In a confined crop rotation study, a sandy loam soil was treated with [^{14}C-chlorophenyl]fenvalerate or [^{14}C-phenoxybenzyl]fenvalerate to provide initial concentrations of 0.22 mg/kg (Fan and Lee, 1980). Lettuce, beets and wheat were sown 30, 60 and 120 days after soil treatment, grown to maturity and examined for residues (Table 22). Residues did not result from the phenoxybenzyl label, and levels of ^{14}C from the chlorophenyl label were low. The highest level was 0.061 mg/kg as fenvalerate in wheat straw, which was investigated further. The majority (59%) was shown to be a conjugate of SD44064, probably a glucoside. Approximately 32% of the residue was incorporated into the cellulose fraction. Fenvalerate itself was not detected.

Lee *et al.* (1982) repeated the study, but with a much higher treatment rate, achieving an initial concentration of 2.3 mg/kg of fenvalerate in the soil, approximately equivalent to a 2.2 kg ai/ha application rate. Lettuce, beets and wheat were sown 30 and 120 days after soil treatment. Levels of ^{14}C in the harvested crops are shown in Table 23. Residues in lettuce were very low and there was no further identification. The residue in beet samples did not contain detectable fenvalerate. ^{14}C levels in wheat hulls and grain were too low for characterization. The level of fenvalerate in wheat straw (30 days, chlorophenyl label) was below the limit of detection (<0.01 mg/kg).

In summary, little residue was carried over, even from an exaggerated fenvalerate application rate, and no fenvalerate itself appeared in the rotation crops.

Table 22. Levels of radiolabel in crops at harvest resulting from sowing seeds into soil previously treated with labelled fenvalerate at 0.22 mg/kg in a confined crop rotation study (Fan and Lee, 1980).

Crop	^{14}C expressed as fenvalerate , mg/kg				
	30 days		60 days		120 days
	^{14}C-chlorophenyl	^{14}C-phenoxybenzyl	^{14}C-chlorophenyl	^{14}C-phenoxybenzyl	^{14}C-chlorophenyl
Lettuce	nd	nd	nd	nd	nd
Beet top	0.015	nd	0.018	nd	0.008
Beet root	0.024	nd	0.015	nd	nd
Wheat straw	0.013	nd	0.061	nd	0.032
Wheat bran	nd	nd	0.022	nd	0.009
Wheat grain	nd	nd	0.021	nd	0.009

nd: not detected

Table 23. Levels of radiolabel in crops at harvest resulting from sowing seeds into soil previously treated with labelled fenvalerate at 2.3 mg/kg in a confined crop rotation study (Lee *et al.*, 1982).

Crop	^{14}C expressed as fenvalerate, mg/kg		
	30 days	120 days	
	^{14}C-chlorophenyl	^{14}C-chlorophenyl	^{14}C-phenoxybenzyl
Lettuce	0.02 0.03	0.01 0.01	0.01 0.01
Beet leaf	0.18 0.10	0.02 0.02	0.02 0.02
Beet root	0.27 0.31	0.04 0.03	0.04 0.03
Wheat straw	0.29 0.33	0.04 0.05	0.05 <0.04
Wheat hulls	0.08 0.07	<0.04	<0.04
Wheat grain	0.04 <0.04	<0.04	<0.04

Environmental fate in water-sediment systems

The Meeting received information on the aqueous sterile hydrolysis of esfenvalerate and fenvalerate and the fate of esfenvalerate in water-sediment systems.

Hydrolysis rates were measured for [^{14}C-chlorophenyl]esfenvalerate and [^{14}C-chlorophenyl]fenvalerate at concentrations of approximately 50µg/l in sterile aqueous buffers (containing Tween 85) at pH 5, 7 and 9 at 25°C in the dark (Katagi *et al.*, 1985c). The results are shown in Table 24.

Hydrolysis rates at pH 5 and 7, especially of fenvalerate, were too low to be measurable in 28 days. At pH 9 the half-lives were similar: 80 and 64 days for fenvalerate and esfenvalerate respectively. Epimerization of esfenvalerate occurred at pH 7 and pH 9 at the α-position, and was faster than hydrolysis.

Table 24. Hydrolysis of fenvalerate and esfenvalerate at 25°C in aqueous buffers in the dark (Katagi *et al.*, 1985c).

Compound and PH	% of applied ^{14}C							Estimated half-life, days
	Incubation period, days							
	0	2	4	7	14	21	28	
FENVALERATE pH 5								
fenvalerate	86	83	82	86	89	77	93	
Dec-fen	1.1	2.2	1.2	1.8	1.2	2.6	2.2	
FENVALERATE pH 7								
fenvalerate	83	83	85	88	101	87	79	
Dec-fen	1.2	2.3	1.8	1.3	1.3	2.7	2.0	
CPIA					0.8	1.4	2.6	

Compound and PH	% of applied ^{14}C							Estimated half-life, days
	Incubation period, days							
	0	2	4	7	14	21	28	
FENVALERATE pH 9								
fenvalerate	96	90	90	91	90	79	72	80
Dec-fen	1.3	2.6	2.0	2.2	2.1	2.1	2.6	
CPIA	-	1.2	2.1	3.9	7.6	11	15	
ESFENVALERATE pH 5								approx. 120-130 [1]
esfenvalerate	103	96	97	99	94	87	86	
2*S*, α*R* epimer	0.8	0.9	0.9	0.8	2.6	1.0	1.2	
Dec-fen	1.2	1.3	1.9	2.2	1.1	3.0	2.0	
$CONH_2$-fen	0.2	0.1	0.4	0.1	<0.1	<0.1	<0.1	
ESFENVALERATE pH 7								
esfenvalerate	86	82	78	76	68	58	53	
2*S*, α*R* epimer	2.2	5.7	12	17	27	34	38	
Dec-fen	1.7	1.5	1.3	1.9	1.9	2.8	2.3	
CPIA	1.0	0.8	<0.1	0.4	1.5	0.1	4.0	
ESFENVALERATE pH 9								
esfenvalerate	71	48	46	48	41	38	27	64 [1]
2*S*, α*R* epimer	20	55	53	48	46	49	42	
Dec-fen	2.2	1.8	1.8	1.8	3.6	2.3	2.5	
CPIA	1.0	1.2	2.2	3.9	7.1	12	15	

[1] estimated half-life is for disappearance of esfenvalerate + 2*S*, α*R* epimer.

Lewis (1995) incubated [^{14}C-chlorophenyl]esfenvalerate in two sediment-water systems (2.5 cm sediment, 25 cm water depth) for 100 days in the dark at 10°C, with air passed through the surface of the water in such a way as not to disturb the water column. Dosing was equivalent to a field application rate of 50 g ai/ha. The two systems were mill stream pond (sand 20%, silt 53%, clay 27%, pH 7.6, organic carbon 5.8%, water conductivity 400-530 µS/cm at 25°C) and site B (sand 1.7%, silt 34%, clay 64%, pH 7.3, organic carbon 1.2%, water conductivity 1600 µS/cm at 25°C). The concentrations of the parent compound and products in the water and sediment were measured by two-dimensional TLC and HPLC. Table 25 summarizes the HPLC results. Recoveries of ^{14}C were in the range 83% to 101%. Very little volatile ^{14}C was evolved from the systems.

The main residue component in the water was CPIA, while esfenvalerate remained the main component in the sediment. The disappearance rates of esfenvalerate from both systems were similar, with half-lives of 54 and 68 days for mill stream and site B respectively.

Table 25. Degradation of [^{14}C-chlorophenyl]esfenvalerate during incubation in water-sediment systems for 100 days in the dark at 10°C (Lewis, 1995).

	% of applied ^{14}C			
	30 days water	30 days sediment	100 days water	100 days sediment
Mill stream pond				
esfenvalerate	3.8	53	2.3	26
CPIA	25	1.2	43	4.1
4'-OH-fen	0.42	3.6	0.48	2.6
DE-Ph-S5602	0.51	0.23	nd	nd
Site B				
esfenvalerate	7.9	56	2.7	25
CPIA	18	0.75	48	5.7
4'-OH-fen	0.70	3.7	0.29	2.4
DE-Ph-S5602	0.28	nd	nd	nd

nd: not detected

Takahashi and Oshima (1988) incubated [^{14}C-phenoxybenzyl]esfenvalerate and [^{14}C-chlorophenyl]esfenvalerate in water-sediment systems at 25°C for 18 weeks under aerobic conditions at a dose of 1 mg/kg dry sediment. The systems consisted of 20 g dry-weight sediment and 60 ml water (approx. 5 cm depth). The systems were Tondabayashi pond (sand 67%, silt 19%, clay 15%, organic matter 3.5%, pH of sediment 4.4, pH of water 6.9) and Onchi river (sand 94%, silt 2.0%, clay 4.5%, organic matter 0.9%, pH of sediment 5.9, pH of water 7.9).

Esfenvalerate was mainly attached to the sediment. Little if any epimerization to the 2*S*, α*R* isomer occurred, typically 0.5-3% of the dose. The main products were the carboxylic acids 2-(4-chlorophenyl)-3-methylbutyric acid (CPIA) and 3-phenoxybenzoic acid (PBacid), appearing in both sediment and water, which were both further degraded to CO_2. 30-50% mineralization occurred in 18 weeks. The half-lives of esfenvalerate were 74 and 79 days in the pond system and 54 and 58 days in the river system. The results are shown in Table 26.

Table 26. Degradation of [^{14}C-phenoxybenzyl]esfenvalerate and [^{14}C-chlorophenyl]esfenvalerate during incubation in aerobic water-sediment systems for 18 weeks in the dark at 25°C (Takahashi and Oshima, 1988).

	% of applied ^{14}C							
	Weeks of incubation							
	0	2	4	6	8	10	14	18
Tondabayashi pond								
[^{14}C-chlorophenyl]esfenvalerate								
CO_2		<0.1	0.1	1.0	4.5	9.6	21	32
water esfenvalerate		0.2	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
water CPIA		6.4	13	18	18	6.9	12	0.8
sediment esfenvalerate	97	74	66	58	51	46	35	28
sediment 2*S*, α*R* epimer	0.4	1.7	1.2	1.2	1.2	0.8	0.9	0.6
sediment CPIA	<0.1	5.8	8.9	11	12	8.0	8.8	2.3
Tondabayashi pond								
[^{14}C-phenoxybenzyl]esfenvalerate								
CO_2		0.3	3.3	9.6	17	24	34	45
water esfenvalerate		0.3	<0.1	<0.1	<0.1	<0.1	-	-
water PBacid		4.7	4.9	1.2	1.1	1.1	-	-
sediment esfenvalerate	94	74	67	59	52	46	37	30
sediment 2*S*, α*R* epimer	2.3	3.4	1.8	1.5	1.8	1.9	1.3	1.1
sediment PBacid	<0.1	6.4	8.9	6.9	6.3	5.0	2.8	2.2
Onchi river								
[^{14}C-chlorophenyl]esfenvalerate								
CO_2		0.6	3.5	11	16	21	29	40
water esfenvalerate		0.1	0.1	0.1	0.1	<0.1	<0.1	<0.1
water CPIA		13	18	7.2	8.5	7.1	1.6	0.2
sediment esfenvalerate	98	68	58	47	40	34	24	18
sediment 2*S*, α*R* epimer	0.5	3.3	2.7	2.6	2.9	3.0	2.5	1.9
sediment CPIA	<0.1	3.4	4.2	2.1	3.7	4.1	0.9	0.8
Onchi river								
[^{14}C-phenoxybenzyl]esfenvalerate								
CO_2		2.0	5.8	18	27	33	42	50
water esfenvalerate		0.1	<0.1	<0.1	<0.1	<0.1	-	-
water PBacid		9.3	13	0.4	5.2	0.1	-	-
sediment esfenvalerate	96	70	59	47	39	35	25	21
sediment 2*S*, α*R* epimer	1.4	3.9	3.5	3.7	5.3	3.6	2.4	1.9
sediment PBacid	<0.1	3.7	4.9	1.9	3.3	1.1	0.7	0.7

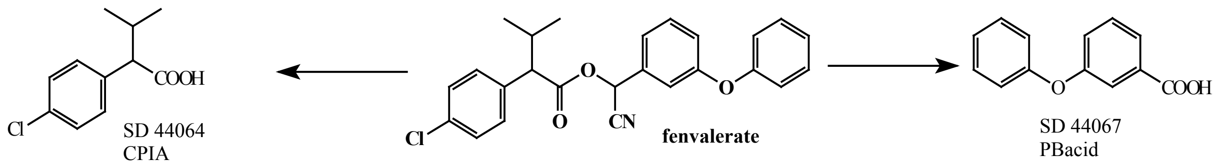


Figure 4. Fenvalerate, fate in water-sediment systems.

RESIDUE ANALYSIS

Analytical methods

The Meeting received information on methods of analysis for esfenvalerate residues in apple, barley ear, barley grain, barley straw, cabbage, cattle fat, cattle kidney, cattle liver, cattle muscle, egg white, egg yolk, hen fat and skin, hen liver, hen muscle, milk, peas, pea straw, pea pods, peach, potato, rape seed, rape seed oil, soil, tomato, tap water, wheat ears, wheat grain, wheat shoots, and wheat straw. Table 27 summarizes the validation results for each method.

In GLC methods for fenvalerate and esfenvalerate the (RS),(*S,R*) pair elutes before the (*S,S*),(*R,R*) pair. The ratio of the peak areas provides some information on the composition of the residue.

Croucher (1998a) described an analytical method for esfenvalerate residues in rape seed and rape seed oil. Rape seed is homogenized with acetone and the extract filtered, diluted with sodium chloride solution and extracted with hexane. Rape seed oil is dissolved in hexane. Residues in the extracts are partitioned into acetonitrile and, after addition of sodium chloride solution, into hexane for Florisil (rape seed) or silica SPE (oil) clean-up. The eluate is evaporated to dryness and the residue dissolved in toluene for analysis by GLC with an ECD. Although some difficulties were experienced with high recoveries from rape seed, the LOQ was accepted as 0.01 mg/kg.

Croucher (1998b) described a similar method for esfenvalerate residues in hen tissues and eggs, which are extracted with acetonitrile, the exact procedure depending on the substrate. Partition between acetonitrile and hexane is used for an initial clean-up and then SPE for the main clean-up. The analysis is completed as above. The LOQ for esfenvalerate residues in each was 0.01 mg/kg. A method for cattle tissues (Croucher, 1998c) is very similar to that for hen tissues. Milk is extracted by treatment with potassium oxalate, ethanol, ether and hexane followed by centrifugation to produce an organic layer, which may be evaporated and taken up in acetonitrile for clean-up and analysis.

Grolleau (1998) described DFG Method S 19 used for the determination of esfenvalerate residues in tomatoes, and provided validation data. Samples are homogenized with acetone and water, and homogenized again after addition of sodium chloride and ethyl acetate/cyclohexane. The homogenate is refrigerated for 30-40 minutes to allow the phases to separate. An aliquot of the organic phase is dried, filtered and evaporated to leave a residue that is taken up in a mixture of acetonitrile and toluene. The extract is cleaned up on an SPE cartridge ready for analysis by GLC with an ECD. The LOQ was 0.01 mg/kg.

Maestracci (1997e) validated an analytical method for a number of commodities down to an LOQ of 0.01 mg/kg: barley ears, barley grain, barley straw, cabbage, peas, pea straw, pea pods, peach, wheat ears, wheat grain and wheat straw. The sample is extracted with acetone, transferred to hexane and partitioned with acetonitrile as the initial clean-up. Clean-up is completed on a silica gel column, and the eluate analysed by GLC with an ECD. The LOQ was 0.01 mg/kg.

Weeren and Pelz (1998a) used DFG Method S 19, with modified extraction, for the determination of esfenvalerate residues in dry plant materials. Water is added before extraction in an amount that takes account of the existing water content, so that the acetone:water ratio remains constant. The method was validated with dry peas. Satisfactory recoveries were obtained down to the LOQ, 0.01 mg/kg. The method with modified extraction was also validated for potatoes (Weeren and Pelz, 1998b) and apples (Weeren and Pelz, 2000).

The same authors (1998c) used a modified extraction for high-lipid samples for DFG Method S 19. The sample is homogenized with acetonitrile, acetone and a synthetic calcium silicate, then filtered and an aliquot of the filtrate evaporated before proceeding with the remainder of the method. Rape seed was chosen as the representative material for validation. Satisfactory recoveries were obtained down to the LOQ, 0.01 mg/kg.

Benwell (1996a,b) validated an analytical procedure CHE/333/56-02R for esfenvalerate residues in wheat grain, straw, ears and shoots and barley grain and shoots. The homogenized sample is treated with water and then extracted with acetone. The extract is filtered, sodium chloride solution added and the residue partitioned into hexane. Grain and immature ear samples are cleaned up on a Florisil column, straw and immature shoot samples on a silica column followed by a diol solid phase cartridge. The eluates are evaporated and the residues taken up in a suitable volume of toluene for analysis by GLC with an ECD. Satisfactory recoveries were obtained down to the LOQ of 0.01 mg/kg.

Burden (1995) validated method CHE 333/40-01R for the determination of esfenvalerate residues in tap water. The water is extracted with dichloromethane, and the extract cleaned up with SPE cartridges and analysed by GLC-ECD. Satisfactory recoveries were obtained at 0.001-0.1 µg/l.

Mirbach and Huber (1991a-c) validated an analytical method for esfenvalerate residues in soils. Soil is extracted with ethyl acetate by shaking and sonification, the extract filtered and evaporated to dryness. The residue is taken up in toluene and, following clean-up on a Florisil column, the esfenvalerate is determined by GLC-NPD. Recoveries were acceptable at fortification levels of 0.02, 0.04 and 0.2 mg/kg.

Table 27. Analytical recoveries of esfenvalerate from various spiked substrates. Determinations are by GLC with an ECD.

Sample	Spike, mg/kg	n	Mean recovery, %	Recovery range, %	Ref
Apple	0.010	5	87	80-94	Weeren and Pelz, 2000
Apple	0.10	5	89	82-95	Weeren and Pelz, 2000
Barley ear	0.01-1.0	6	75	70-81	Maestracci, 1997e
Barley grain	0.01-1.0	9	86	72-97	Benwell, 1996b
Barley grain	0.01-0.10	4	93	91-95	Maestracci, 1997e
Barley straw	0.01-1.0	9	82	70-98	Benwell, 1996b
Barley straw	0.05-1.0	6	83	70-96	Maestracci, 1997e
Cabbage	0.01-0.10	6	94	87-100	Maestracci, 1997e
Cabbage	0.01-0.10	4	98	81-109	Maestracci, 1997e
Cattle fat	0.01	3	79	75-85	Croucher, 1998c
Cattle fat	0.1	3	79	74-84	Croucher, 1998c
Cattle fat	1	3	82	74-98	Croucher, 1998c
Cattle kidney	0.01	3	98	94-105	Croucher, 1998c
Cattle kidney	0.1	3	89	85-91	Croucher, 1998c
Cattle kidney	1	3	81	80-83	Croucher, 1998c
Cattle liver	0.01	3	90	86-97	Croucher, 1998c
Cattle liver	0.1	3	89	81-100	Croucher, 1998c
Cattle liver	1	3	77	73-79	Croucher, 1998c
Cattle muscle	0.01	3	79	74-86	Croucher, 1998c
Cattle muscle	0.1	3	87	85-88	Croucher, 1998c
Cattle muscle	1	3	81	77-86	Croucher, 1998c
Egg white	0.01	3	83	78-92	Croucher, 1998b
Egg white	0.1	3	90	82-95	Croucher, 1998b

Sample	Spike, mg/kg	n	Mean recovery, %	Recovery range, %	Ref
Egg white	1	3	77	71-81	Croucher, 1998b
Egg yolk	0.01	3	98	93-106	Croucher, 1998b
Egg yolk	0.1	3	82	74-91	Croucher, 1998b
Egg yolk	1	3	72	62-77	Croucher, 1998b
Hen fat and skin	0.01	3	114	107-122	Croucher, 1998b
Hen fat and skin	0.1	3	95	95-96	Croucher, 1998b
Hen fat and skin	1	3	99	94-104	Croucher, 1998b
Hen liver	0.01	5	100	82-115	Croucher, 1998b
Hen liver	0.1	3	93	91-96	Croucher, 1998b
Hen liver	1	3	90	88-94	Croucher, 1998b
Hen muscle	0.01	3	78	76-79	Croucher, 1998b
Hen muscle	0.1	3	79	76-82	Croucher, 1998b
Hen muscle	1	3	87	82-94	Croucher, 1998b
Milk	0.01	3	82	77-87	Croucher, 1998c
Milk	0.1	3	70	62-78	Croucher, 1998c
Milk	1	3	95	89-100	Croucher, 1998c
Pea grain	0.01-0.05	4	98	96-103	Maestracci, 1997e
Pea grain, dried	0.01-0.10	6	96	86-104	Maestracci, 1997e
Pea straw	0.05-0.50	6	91	76-106	Maestracci, 1997e
Pea, pod	0.01-0.10	6	90	76-100	Maestracci, 1997e
Pea, pod	0.01-0.05	4	84	78-88	Maestracci, 1997e
Peach	0.01-0.10	6	81	74-89	Maestracci, 1997e
Peas, dry	0.010	5	96	84-109	Weeren and Pelz, 1998a
Peas, dry	0.10	5	85	80-87	Weeren and Pelz, 1998a
Peas, dry	1.0	5	94	92-97	Weeren and Pelz, 1998a
Potato	0.010	5	98	92-103	Weeren and Pelz, 1998b
Potato	0.10	5	93	83-101	Weeren and Pelz, 1998b
Potato	1.0	5	96	81-104	Weeren and Pelz, 1998b
Rape seed	0.01	3	119	112-129	Croucher, 1998a
Rape seed	0.010	5	84	79-94	Weeren and Pelz, 1998c
Rape seed	0.1	3	111	99-118	Croucher, 1998a
Rape seed	0.10	5	100	94-108	Weeren and Pelz, 1998c
Rape seed	1	3	96	88-101	Croucher, 1998a
Rape seed	1.0	5	87	82-94	Weeren and Pelz, 1998c
Rape seed oil	0.01	3	76	69-81	Croucher, 1998a
Rape seed oil	0.1	3	83	74-90	Croucher, 1998a
Rape seed oil	1	3	84	79-88	Croucher, 1998a
Soil	0.02-0.2	10	90	69-102	Mirbach and Huber, 1991a
Soil	0.02-0.2	5	100	80-120	Mirbach and Huber, 1991b
Soil	0.02-0.2	10	87	63-110	Mirbach and Huber, 1991c
Tomato	0.01	5	78	73-82	Grolleau, 1998
Tomato	0.10	5	81	76-86	Grolleau, 1998
Tomato	1.0	5	80	74-88	Grolleau, 1998
Water, tap	0.001-0.1 µg/l	9	89	84-95	Burden, 1995
Wheat ear	0.01-1.0	6	87	70-108	Maestracci, 1997e
Wheat ears, immature	0.01-1.0	9	93	77-102	Benwell, 1996a
Wheat grain	0.01-1.0	9	101	88-107	Benwell, 1996a
Wheat grain	0.01-0.10	6	92	85-107	Maestracci, 1997e
Wheat shoots, immature	0.01-1.0	9	96	91-104	Benwell, 1996a
Wheat straw	0.01-1.0	9	100	89-109	Benwell, 1996a
Wheat straw	0.05-0.50	6	86	78-90	Maestracci, 1997e

Stability of residues in stored analytical samples

The Meeting received information on the stability of esfenvalerate residues during frozen storage of analytical samples of almonds, beef, blackberries, cabbage, corn silage, eggs, green beans, lettuce, milk, peaches, soil, soya beans, sugar beets, tomatoes, watermelons, wheat grain and wheat straw.

Schneiders and Orescan (1995) tested the freezer storage stability of esfenvalerate in soil and animal and plant commodities. The samples were finely chopped, fortified with esfenvalerate at 0.4 mg/kg and stored in glass jars at approximately -10°C for periods up to three years. At each analysis esfenvalerate was measured in two stored samples, one procedural recovery sample and one unfortified sample. Samples were analysed by three different methods for soil, for oily crops and fatty tissues, and for other crops. Extraction and clean-up were designed to match the substrate, with final analysis by GLC with an ECD.

No significant racemization of esfenvalerate was observed. The (*RS*),(*S,R*) pair is eluted before the (*S,S*) ,(*R,R)* pair by GLC, but was not observed in the stored samples.

Table 28. Freezer storage stability of esfenvalerate on finely chopped substrates spiked at 0.4 mg/kg and stored at approximately -10°C for periods up to three years (Schneiders and Orescan, 1995). Duplicate stored samples were analysed at each interval.

Storage, months	Stored sample, mg/kg	Procedural recovery, %	Storage, months	Stored sample, mg/kg	Procedural recovery, %	Storage, months	Stored sample, mg/kg	Procedural recovery, %
soil			beef			eggs		
0	0.40 0.37		0	0.46 0.34		0	0.31 0.39	
8.7	0.47 0.41	118	2.6	0.39 0.31	98	10.8	0.43 0.42	100
12.4	0.42 0.43	96	8.9	0.35 0.43	77	12.0	0.40 0.39	86
24.3	0.42 0.48	104	20.5	0.31 0.34	82	24.2	0.25 0.37	89
36.6	0.43 0.44	115	33.0	0.32 0.30	80	36.5	0.38 0.35	87
milk			almonds			blackberries		
0	0.40 0.39		0	0.30 0.27		0	0.43 0.48	
6.3	0.48 0.45	104	2.6	0.28 0.36	77	6.1	0.46 0.42	111
12.1	0.34 0.32	105	8.9	0.28 0.35	103	12.1	0.42 0.44	104
24.2	0.36 0.38	88	20.5	0.30 0.44	76	24.1	0.45 0.45	118
36.6	0.44 0.44	114	32.9	0.36 0.32	99	36.5	0.45 0.45	93
cabbage			corn silage			green beans		
0	0.44 0.41		0	0.41 0.46		0	0.44 0.40	
6.2	0.46 0.46	113	6.1	0.37 0.42	111	6.2	0.41 0.38	98
12.0	0.40 0.37	102	12.1	0.38 0.35	100	12.2	0.43 0.37	112
24.2	0.41 0.45	118	24.1	0.40 0.37	109	24.2	0.33 0.33	95
36.6	0.38 0.38	100	36.5	0.48 0.37	89	36.6	0.42 0.42	99
lettuce			peach			soya beans		
0	0.41 0.41		0	0.43 0.38		0	0.36 0.34	
6.2	0.50 0.38	113	6.1	0.45 0.48	104	2.6	0.47 0.37	100
12.2	0.38 0.40	108	12.1	0.41 0.42	106	8.9	0.34 0.28	95
24.2	0.48 0.41	99	24.1	0.34 0.36	100	20.5	0.32 0.29	97
36.6	0.42 0.39	106	36.5	0.42 0.39	102	32.9	0.38 0.31	76
sugar beets			tomatoes			watermelon		
0	0.46 0.41		0	0.45 0.47		0	0.48 0.45	
6.2	0.38 0.37	107	10.9	0.36 0.45	111	6.2	0.48 0.43	104
12.1	0.33 0.32	104	12.1	0.46 0.40	102	11.9	0.45 0.39	113
24.1	0.31 0.30	86	24.2	0.46 0.47	121	24.1	0.38 0.41	78
36.6	0.37 0.34	116	36.6	0.39 0.43	104	36.5	0.45 0.41	101

Benwell and Burden (1997) tested the stability of esfenvalerate in homogenized wheat grain and straw fortified at 0.25 mg/kg and stored in glass jars at approximately -18°C for periods up to two years. Sample sizes in the jars were 10 g for grain and 5 g for straw. Each set of analyses was of one stored sample, one sample for procedural recovery (at 0.25 mg/kg) and one unfortified sample. The analytical method was the same as that used for cereal trials in Europe. No measurements were made of possible epimerization during storage. The residues were stable during storage (Table 29).

Table 29. Stability of esfenvalerate in fortified wheat grain and wheat straw stored for 2 years in the dark at a nominal -18°C (Benwell and Burden, 1997).

Storage, months	Wheat grain		Wheat straw	
	Stored esfenvalerate, mg/kg	Procedural recovery, %	Stored esfenvalerate, mg/kg	Procedural recovery, %
0	0.24	99	0.25	102
1	0.24	83	0.25	86
3	0.19	99	0.23	83
6	0.22	104	0.24	103
12	0.21	91	0.22	87
18	0.21	79	0.22	85
24	0.26	102	0.24	95

Definition of the residue

Fenvalerate was introduced as a pesticide before esfenvalerate and residues for fenvalerate MRLs were usually defined as the sum of the fenvalerate isomers. In national systems esfenvalerate residues then conveniently fitted into the fenvalerate residue definition.

The residue of esfenvalerate should be defined as the (*S,S*) isomer only. However, separation of the (*S,S*) and (*R,R)* isomers would be analytically expensive and generally would serve little purpose because the level of the (*R,R)* isomer in esfenvalerate is typically only about 1%.

The hydrolysis studies suggest that epimerization of esfenvalerate is possible so that some of the (*S,S*) isomer could be converted to the (*S,R*) isomer and appear as such in the residue. In crop and animal residue situations epimerization is probably insignificant (<10%) and the (*S,R*) isomer (initially a 7% component of technical esfenvalerate) remains a minor component of the residue. The (*RS,SR*) pair elutes before the (*SS,RR*) pair in GLC analysis and, if the (*SR*) isomer is included, the *RS* should also be included because they are not separated by routine analytical methods.

It should be noted that most of the residue data for esfenvalerate are for the sum of all the isomers. A residue of the (*SS*) and (*RR*) isomers would generally be about 15% less than the sum of all the isomers, but in practice 15% makes little difference in comparison with inherent residue variation.

The FAO Manual (page 51) states that preferably no compound, metabolite or analyte should appear in more than one residue definition. It follows that, while a fenvalerate CXL is maintained for a commodity, the residues of esfenvalerate would be accommodated by the fenvalerate residue definition.

The Meeting agreed that the residue of esfenvalerate should be defined as the sum of the fenvalerate isomers.

Definition of esfenvalerate residue (for compliance with MRL and for estimation of dietary intake): sum of fenvalerate isomers.

The definition is worded to emphasise that all fenvalerate isomers are included, but the intention is that the definition is identical to that for fenvalerate.

The definition applies to plant and animal commodities. The residue is classed as fat-soluble.

USE PATTERN

Esfenvalerate has registrations in many countries as a broad-spectrum pyrethroid insecticide for a range of crops. It has uses on pome and stone fruits, small fruits and berries; root and tuber, bulb, leafy, Brassica, legume and fruiting vegetables; cereal grains and grasses, oilseeds and tree nuts. Esfenvalerate has registrations for the control of alfalfa looper, aphids, armyworms, beetles, cabbage looper, capsid bugs, carrot weevil, caterpillars, cherry fruit fly, chinch bugs, cloverworm, codling moth, Colorado potato beetle, corn borer, corn earworm, corn rootworms, cotton bollworm, cranberry weevil, cutworms, diamond back moth, European corn borer, grasshoppers, Heliothis, leafhopper, leafminers, leafrollers, moths, oriental fruit moth, pea aphid, pea weevil, peach tree borer, peach twig borer, pecan weevil, psylla, rootworm, San Jose scale, sawfly, spittlebug, stemborers, sucking insects, sugar cane borer, thrips, weevils and whitefly.

Information on registered uses was made available to the Meeting and is summarized in Table 30.

Table 30. Registered uses of esfenvalerate.

Crop	Country	Form	Application				PHI days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl	No.[1]	
Alfalfa	Spain	50 g/l EW	foliar	0.015	0.002-0.0075	1-2	15
Almonds	Chile	75 g/l EC	foliar		0.0005	1	21
Almonds	France	25 g/l EC	foliar	0.013	0.0013		15
Almonds	Mexico	110 g/l EC	foliar	0.088	0.004	2	21
Almonds	USA	84 g/l EC	foliar	0.056–0.11		(0.22)	21
Apple	Argentina	50 g/l EC	foliar	0.012	0.001		14
Apple	Bulgaria	12.5 g/l EC	foliar		0.0005		30
Apple	Bulgaria	50 g/l EC	foliar		0.001		14
Apple	Chile	75 g/l EC	foliar		0.0045	1	15
Apple	China	50 g/l EC	foliar		0.0017–0.0025		14
Apple	Denmark	50 g/l EW	foliar	0.005	0.0008-0.0024	1-2	42
Apple	France	25 g/l EC	foliar	0.013	0.0013		15
Apple	Korea	15 g/l EC	foliar	0.007		4	21
Apple	Mexico	110 g/l EC	foliar	0.088	0.004	2	21
Apple	USA	84 g/l EC	foliar	0.028–0.084		(0.59)	21
Apricot	Austria	50 g/l EC	foliar		0.0013-0.0015		14
Apricot	France	25 g/l EC	foliar	0.01–0.015	0.001– 0.0015		15
Artichoke	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	7
Artichoke	USA	84 g/l EC	foliar	0.034–0.056		(0.17)	1
Asparagus	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	14
Asparagus	France	25 g/l EC	foliar	0.013	0.0016- 0.003		
Barley	Australia	50 g/l EC	spraying	0.0065-0.017	-	-	7
Barley	Belgium	25 g/l EC	foliar	0.005			42
Barley	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	7
Barley	Germany	50 g/l EC	foliar	0.01–0.013	0.0017-0.007	1-2	35
Barley	Ireland	25 g/l EC	foliar	0.004	0.002	1-2	.
Barley	Italy	100 g/l EC	foliar	0.013–0.018	0.0016-0.003	1-2	21
Barley	Luxembourg	25 g/l EC	foliar	0.01–0.015	0.001-0.003	1-2	42
Barley	Spain	50 g/kg EW	foliar	0.015	0.003-0.0075	1-3	28
Barley	UK	25 g/l EC	foliar	0.003–0.005	0.0006-0.0025		
Beans, dry	Australia	50 g/l EC	foliar	0.007–0.017			14
Beans, dry	Brazil	25 g/l EC	foliar	0.010			14
Beans, dry	Spain	50 g/l EW	foliar	0.015	0.0015-0.0038	1-2	30
Beans, dry	USA	84 g/l EC	foliar	0.017–0.056		(0.22)	21

Crop	Country	Form	Application				PHI days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl	No.[1]	
Beans, broad	Australia	50 g/l EC	foliar	0.007–0.017			14
Beans, field	Austria	50 g/l EC	foliar	0.01	0.0017-0.005		42
Beans, field	Ireland	25 g/l EC	foliar	0.0075	0.0019	1–2	
Beans, field	Netherlands	25 g/l EC	foliar	0.005	0.0013	1-2	7
Beans, green	Australia	50 g/l EC	foliar	0.007–0.017			3
Beans, snap	USA	84 g/l EC	foliar	0.017–0.056		(0.22)	3
Beetroot	Denmark	50 g/l EW	foliar	0.01–0.015	0.0025-0.0075	1-2	42
Berry fruit	Austria	50 g/l EC	foliar		0.0013-0.0015		14
Blackcurrant	Poland	50 g/l EC	high vol	0.022	0.002-0.003	2	7
Blackcurrant	Poland	50 g/l EC	high vol	0.03	0.003-0.004	2	7
Blackberries	USA	84 g/l EC	foliar	0.028–0.056		(0.17)	7
Blueberry	USA	84 g/l EC	foliar	0.028–0.056		(0.22)	14
Boysenberries	USA	84 g/l EC	foliar	0.028–0.056		(0.17)	7
Brassica vegetables	El Salvador	100 g/l EC	foliar	0.028–0.056		2	3
Brassica vegetables	Guatemala	100 g/l EC	foliar	0.028–0.056		2	3
Brassica vegetables	Honduras	100 g/l EC	foliar	0.028–0.056		2	3
Brassica vegetables	Nicaragua	100 g/l EC	foliar	0.028–0.056		2	3
Broccoli	Australia	50 g/l EC	spraying	0.0095-0.019		-	2
Broccoli	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	3
Broccoli	Mexico	119 g/l EC	foliar	0.055		2	3
Broccoli	Netherlands	25 g/l EC	spraying	0.005		2	7
Broccoli	USA	84 g/l EC	foliar	0.017–0.056		(0.45)	3
Brussels sprouts	Australia	50 g/l EC	spraying	0.0095-0.019	0.00125	-	2
Brussels sprouts	Belgium	25 g/l EC	foliar	0.005			7
Brussels sprouts	Netherlands	25 g/l EC	spraying	0.005		2	7
Brussels sprouts	Spain	50 g/l EW	foliar	0.015	0.001-0.0038	1- 3	7
Bushberry group	Austria	50 g/l EC	foliar	0.013–0.015	0.0013- 0.0015		14
Bushberry group	Denmark	50 g/l EW	foliar	0.013- 0.015	0.001–0.0038	1-2	14
Cabbage	Belgium	25 g/l EC	foliar	0.005			7
Cabbage	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	3
Cabbage	China	50 g/l EC	foliar	0.008–0.015			3
Cabbage	Denmark	50 g/l EW	foliar	0.01–0.015	0.002–0.008		7
Cabbage	France	25 g/l EC	foliar	0.013	0.002–0.006		
Cabbage	Ireland	25 g/l EC	foliar	0.0038	0.0006-0.001	1-3	7
Cabbage	Luxembourg	25 g/l EC	foliar	0.005	0.0008-0.0025	1	7
Cabbage	Malaysia	25 g/l EC	foliar	0.035–0.050			7
Cabbage	Netherlands	25 g/l EC	foliar	0.005	0.0013	1-4	7
Cabbage	Portugal	25 g/l EC	foliar	0.015	0.0015	1-2	7
Cabbage	Spain	50 g/l EW	foliar	0.015	0.001-0.0038	1-3	7
Cabbage	USA	84 g/l EC	foliar	0.017–0.056		(0.45)	3
Cabbages	Australia	50 g/l EC	spraying	0.0095-0.019	0.00125	-	2
Cantaloupe	USA	84 g/l EC	foliar	0.034–0.056		(0.28)	3
Carrot	Denmark	50 g/l EW	foliar	0.015	0.0008-0.0025	1-3	14
Carrot	Finland	50 g/l EC	foliar	0.015-0.025			
Carrot	USA	84 g/l EC	foliar	0.034–0.056		(0.56)	7
Cauliflower	Belgium	25 g/l EC	foliar	0.005			7
Cauliflower	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	3
Cauliflower	Mexico	119 g/l EC	foliar	0.055		2	3
Cauliflower	Netherlands	25 g/l EC	spraying	0.005		2	7
Cauliflower	USA	84 g/l EC	foliar	0.017–0.056		(0.45)	3
Cauliflowers	Australia	50 g/l EC	spraying	0.0095-0.019	0.00125	-	2
Celery	Australia	50 g/l EC	spraying	-	0.0025	-	1
Celery	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	14
Cereals	Australia	50 g/l EC	spraying	0.0035	-	-	7
Cereals	Austria	50 g/l EC	foliar	0.0075-0.01	0.001- 0.005		0
Cereals	Denmark	50 g/l EW	foliar	0.01–0.015	0.0025- 0.008	1-2	30
Cereals	France	25 g/l EC	foliar	0.006-0.0075	0.001–0.004	1	
Cereals	Netherlands	25 g/l EC	foliar	0.005	0.0013	1	42

Crop	Country	Form	Application				PHI days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl	No.[1]	
Cereals	Portugal	25 g/l EC	foliar	0.015	0.0015	1-2	56
Cereals (barley, oats, rye, triticale, wheat)	Germany	50 g/l EC	spraying	0.01	0.003-0.005	1	
Cereals (barley, oats, rye, triticale, wheat)	Netherlands	25 g/l EC	spraying	0.005		2	14
Cherries	Chile	75 g/l EC	foliar		0.0005	1	5
Chickpeas	Australia	50 g/l EC	spraying	0.0065-0.025	-	-	14
Chinese broccoli	USA	84 g/l EC	foliar	0.017–0.056		(0.45)	3
Citrus	China	50 g/l EC	foliar		0.0006– 0.0007		21
Citrus	Korea	12.5 g/l EC	foliar	0.007		6	14
Citrus	Malaysia	25 g/l EC	foliar	0.016–0.025			7
Clover	Denmark	50 g/l EW	foliar	0.01-0.015	0.003-0.0075	1	
Clover	Spain	50 g/l EW	foliar	0.025–0.03	0.004–0.015	1 -2	
Collards	USA	84 g/l EC	foliar	0.034–0.056		(0.22)	7
Cotton	Australia	50 g/l EC	spraying	0.010-0.035	-	-	7
Cotton	Argentina	50 g/l EC	foliar	0.020–0.024	0.005 –0.006		21
Cotton	Brazil	15 g/l SC	foliar	0.007–0.030			7
Cotton	China	50 g/l EC	foliar	0.019–0.026			14
Cotton	Colombia	100 g/l EC	foliar	0.050			
Cotton	Costa Rica	100 g/l EC	foliar	0.029–0.036		2	21
Cotton	El Salvador	100 g/l EC	foliar	0.029–0.036		2	21
Cotton	Guatemala	100 g/l EC	foliar	0.029–0.036		2	21
Cotton	Greece	50 g/l EC	foliar	0.030			21
Cotton	Honduras	100 g/l EC	foliar	0.029–0.036		2	21
Cotton	Indonesia	25 g/l EC	foliar		0.005–0.010		
Cotton	Italy	100 g/l EC	foliar	0.013–0.05		1-3	
Cotton	Mexico	110 g/l	foliar	0.055		2	21
Cotton	Nicaragua	100 g/l EC	foliar	0.029–0.036		2	21
Cotton	Paraguay	25 g/l EC	foliar	0.019–0.025			21
Cotton	Paraguay	100 g/l EC	foliar	0.020–0.025			21
Cotton	Spain	50 g/l EW	foliar	0.025-0.03			30
Cotton	Spain	25 g/l EC	foliar	0.038			21
Cotton	Thailand	50 g/l EC	foliar		0.010		
Cotton	USA	84 g/l EC	foliar	0.022–0.056		(0.56)	21
Cucumber	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	3
Cucumber	Mexico	110 g/l EC	foliar	0.055		2	3
Cucumber	USA	84 g/l EC	foliar	0.034–0.056		(0.28)	3
Dewberries	USA	84 g/l EC	foliar	0.028–0.056		(0.17)	7
Dwarf snap beans	Netherlands	25 g/l EC	spraying	0.005		2	10
Egg plant	Spain	50 g/l EW	foliar	0.015	0.0015-0.0038	1-3	3
Egg plant	USA	84 g/l EC	foliar	0.034–0.056		(0.39)	7
Filberts	USA	84 g/l EC	foliar	0.056–0.11		(0.22)	21
Fodder beet	Netherlands	25 g/l EC	spraying	0.011		2	
Fodder rape	Australia	50 g/l EC	spraying	0.0065-0.025	-	-	2
Fruit trees	France	25 g/l EC	foliar	0.010-0.013			15
Fruiting vegetables	Austria	50 g/l EC	foliar	0.0075-0.0013			14
Garden pea	Poland	50 g/l EC	high vol	0.01	0.002-0.005	1	14
Garden peas	Australia	50 g/l EC	spraying	0.0065-0.025	-	-	14
Grain	Austria	50 g/l EC	foliar	0.0075-0.010			21
Grapes	Austria	50 g/l EC	foliar	0.015	0.0015		28
Grapes	Chile	75 g/l EC	foliar	0.01–0.02	0.001–0.002	2	21
Grapes	France	25 g/l EC	foliar	0.01–0.02	0.001-0.002		15
Grapes	Italy	100 g/l EC	foliar	0.018–0.03	0.001-0.0015	1 -2	14
Grapes	Luxembourg	25 g/l EC	foliar	0.02	0.07–0.08	1-3	15
Grapes	Spain	50 g/l EW	foliar	0.015	0.0019-0.0038	1-2	15
Honeydew	USA	84 g/l EC	foliar	0.034–0.056		(0.28)	3
Kale	Australia	50 g/l EC	spraying	0.0095-0.019	0.00125	-	2
Kidney beans	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	7

Crop	Country	Form	Application				PHI days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl	No.[1]	
Kohlrabi	Australia	50 g/l EC	spraying	0.0095-0.019	0.00125	-	2
Kohlrabi	Netherlands	25 g/l EC	spraying	0.005		2	7
Kohlrabi	USA	84 g/l EC	foliar	0.034-0.056		(0.45)	3
Leafy vegetables	Austria	50 g/l EC	foliar	0.0075-0.0013			14
Leeks	Denmark	50 g/l EW	foliar	0.015	0.003-0.008	1-3	14
Lentils	Australia	50 g/l EC	foliar	0.007–0.017			14
Lentils	Australia	50 g/l EC	spraying	0.0065-0.017	-	-	14
Lentils	Brazil	25 g/l EC	foliar	0.010			14
Lentils	Spain	50 g/l EW	foliar	0.015	0.0015-0.0038	1-2	30
Lentils	USA	84 g/l EC	foliar	0.017–0.056		(0.22)	21
Lettuce, Head	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	14
Lettuce, Head	USA	84 g/l EC	foliar	0.028 –0.056		(0.39)	7
Linseed	Australia	50 g/l EC	foliar	0.007–0.025			14
Linseed	France	25 g/l EC	foliar	0.01	0.0017- 0.005		
Loganberries	USA	84 g/l EC	foliar	0.028–0.056		(0.17)	7
Lucerne	Australia	50 g/l EC	spraying	0.0065-0.025	-	-	
Lupins	Australia	50 g/l EC	foliar	0.002–0.017			14
Maize	Argentina	50 g/l EC	foliar	0.014	0.009		21
Maize	Australia	50 g/l EC	spraying	0.015-0.025	-	-	7
Maize	Brazil	25 g/l EC	foliar	0.015–0.020			21
Maize	Chile	75 g/l EC	0.019			2	14
Maize	Costa Rica	100 g/l EC	foliar	0.029-0.056		2	21
Maize	Denmark	50 g/l EW	foliar	0.015	0.0038 0.008	1-2	45
Maize	El Salvador	100 g/l EC	foliar	0.029-0.056		2	21
Maize	Guatemala	100 g/l EC	foliar	0.029-0.056		2	21
Maize	Honduras	100 g/l EC	foliar	0.029-0.056		2	21
Maize	Nicaragua	100 g/l EC	foliar	0.029-0.056		2	21
Maize	Spain	50 g/l EW	foliar	0.015	0.0025-0.0075	1-2	45
Maize	USA	84 g/l EC	foliar	0.017–0.056		(0.28)	21
Melons	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	3
Melons	Costa Rica	100 g/l EC	foliar	0.028 -0.056		2	3
Melons	El Salvador	100 g/l EC	foliar	0.028 -0.056		2	3
Melons	Guatemala	100 g/l EC	foliar	0.028 -0.056		2	3
Melons	Honduras	100 g/l EC	foliar	0.028 -0.056		2	3
Melons	Mexico	110 g/l EC	foliar	0.055		2	3
Melons	Nicaragua	100 g/l EC	foliar	0.028 -0.056		2	3
Melons	USA	84 g/l EC	foliar	0.034–0.056		(0.28)	3
Mung beans	Australia	50 g/l EC	spraying	0.020-0.025	-	-	14
Muskmelon	USA	84 g/l EC	foliar	0.034–0.056		(0.28)	3
Mustard greens	USA	84 g/l EC	foliar	0.056		(0.22)	7
Navy beans	Australia	50 g/l EC	spraying	0.020-0.025	-	-	14
Nectarine	Chile	75 g/l EC	foliar		0.0005	1	5
Oats	Australia	50 g/l EC	spraying	0.0065-0.017	-	-	7
Oats	Spain	50 g/l EW	foliar	0.015	0.0025-0.0075	1	28
Onion	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	14
Onion	Denmark	50 g/l EW	foliar	0.015	0.003-0.008	1-3	14
Onion	Netherlands	25 g/l EC	foliar	0.005	0.0013	1-2	7
Peach	Austria	50 g/l EC	foliar		0.0013-0.0015		14
Peach	Chile	75 g/l EC	foliar		0.0005	1	5
Peach	France	25 g/l EC	foliar	0.01–0.015	0.001 –0.0015		15
Pear	Argentina	50 g/l EC	foliar	0.012	0.0008		14
Pear	Austria	50 g/l EC	foliar	0.013–0.015	0.0013-0.0015	1-2	14
Pear	Chile	75 g/l EC	foliar		0.0045	1	15
Pear	Denmark	50 g/l EW	foliar	0.013-0.015	0.0013- 0.003	1-2	14
Pear	France	25 g/l EC	foliar	0.01–0.015	0.0013- 0.0015		15
Pear	Korea	15 g/l EC	foliar	0.0002		6	7
Pear	USA	84 g/l EC	foliar	0.028–0.084		(0.42)	28
Pear (dormant)	USA	84 g/l EC	foliar	0.056-0.11		(0.22)	-

Crop	Country	Form	Application				PHI days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl	No.[1]	
Peas	Finland	50 g/l EC	foliar	0.015-0.020			F[2]
Peas, dry	Australia	50 g/l EC	foliar	0.007–0.017			14
Peas, dry	Brazil	25 g/l EC	foliar	0.010			14
Peas, dry	Spain	50 g/l EW	foliar	0.015	0.0015-0.0038	1-2	30
Peas, dry	USA	84 g/l EC	foliar	0.017–0.056		(0.22)	21
Peas, Field	Australia	50 g/l EC	foliar	0.004–0.017			14
Peas, Field	Austria	50 g/l EC	foliar	0.01	0.0017– 0.005		42
Peas, Field	France	25 g/l EC	foliar	0.01	0.0017-0.005		
Peas, Garden	Australia	50 g/l EC	foliar	0.007–0.025			14
Peas, Green	Chile	75 g/l EC	foliar	0.011–0.019	0.002	1	3
Peas, Green	Denmark	50 g/l EW	foliar	0.01-0.015	0.0017-0.0075	1-2	14
Peas, Green	France	25 g/l EC	foliar	0.01	0.0017- 0.005	1-2	
Peas, Green	Ireland	25 g/l EC	foliar	0.0075	0.002	1-3	
Peas, Green	Netherlands	25 g/l EC	foliar	0.005	0.0013	1-2	7
Peas, Green	USA	84 g/l EC	foliar	0.017–0.056		(0.11)	3
Peas, Pigeon	Australia	50 g/l EC	foliar	0.007–0.025			14
Pecans	USA	84 g/l EC	foliar	0.028–0.084		(0.34)	21
Pepper	Chile	75 g/l EC	foliar	0.011–0.019	0.002 0.003	1	7
Pepper	Costa Rica	100 g/l EC	foliar	0.029–0.036		2	7
Pepper	Indonesia	25 g/l EC	foliar		0.0006– 0.001		
Pepper	Malaysia	25 g/l EC	foliar	0.016–0.025			7
Pepper	Mexico	110 g/l EC	foliar	`0.055		2	7
Pepper	USA	84 g/l EC	foliar	0.034–0.056		(0.39)	7
Plums	Chile	75 g/l EC	foliar		0.0005	1	5
Pome fruit	Italy	100 g/l EC	foliar	0.027–0.2	0.0015-0.01	1-3	7
Pome fruit	Portugal	35 g/l EC	foliar	0.01–0.1	0.001–0.01	1-3	14
Pome fruit	Spain	50 g/l EW	foliar	0.01-0.015	0.0007-0.003	1-2	14
Potato	Austria	50 g/l EC	foliar	0.010			14
Potato	Belgium	25 g/l EC	foliar	0.005-0.0075			7
Potato	Denmark	50 g/l EW	foliar	0.01–0.015	0.0021-0.0042	1	14
Potato	France	25 g/l EC	foliar	0.013	0.0016-0.0030		8
Potato	Luxembourg	25 g/l EC	foliar	0.005	0.0008-0.0025	1-2	7
Potato	Mexico	110 g/l EC	foliar	0.055		2	7`
Potato	Portugal	25 g/l EC	foliar	0.015–0.020	0.0015-0.0020	1–3	14
Potato	Spain	50 g/l EW	foliar	0.015	0.0015-0.0038	1-2	15
Potato	USA	84 g/l EC	foliar	0.017–0.056		(0.39)	7
Potatoes	Netherlands	25 g/l EC	spraying	0.005		2	7
Protein crops	Austria	50 g/l EC	foliar	0.010			42
Pulses	Netherlands	25 g/l EC	spraying	0.005		2	7
Pumpkin	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	3
Pumpkin	USA	84 g/l EC	foliar	0.034–0.056		(0.28)	3
Radish	Finland	50 g/l EC	foliar	0.010-0.020			F[3]
Radishes	USA	84 g/l EC	foliar	0.034–0.056		(0.11)	7
Rape seed (incl. Canola)	Australia	50 g/l EC	spraying	0.0065-0.025	-	-	14
Rape seed	Austria	50 g/l EC	foliar	0.015			56
Rape seed	Belgium	25 g/l EC	foliar	0.0075			42
Rape seed	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	7
Rape seed	Denmark	50 g/l EW	foliar	0.01–0.015	0.003-0.0075	1	14
Rape seed	France	25 g/l EC	foliar	0.01–0.015	0.005-0.0075		
Rape seed	Germany	50 g/l EC	spraying	0.013	0.003-0.006	1	56
Rape seed	Germany	50 g/l EC	spraying	0.008	0.002-0.004	1	56
Rape seed	Germany	50 g/l EC	foliar	0.012	0.0016-0.003		8
Rape seed	Ireland	25 g/l EC	foliar	0.0075-0.013	0.004-0.006	1-3	
Rape seed	Italy	100 g/l EC	foliar	0.008 –0.023	0.001-0.0038	1 -2	30
Rape seed	Luxembourg	25 g/l EC	foliar	0.0075	0.001-0.0038	1	42
Rape seed	Spain	50 g/l EW	foliar	0.015	0.003-0.0075	1-2	42
Raspberries	USA	84 g/l EC	foliar	0.028–0.056		(0.17)	7

Crop	Country	Form	Application				PHI days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl	No.[1]	
Rice	Brazil	25 g/l EC	foliar	0.025			21
Rice	Costa Rica	100 g/l EC	foliar	0.012-0.015		2	21
Rice	El Salvador	100 g/l EC	foliar	0.012-0.015		2	21
Rice	Guatemala	100 g/l EC	foliar	0.012-0.015		2	21
Rice	Honduras	100 g/l EC	foliar	0.012-0.015		2	21
Rice	Nicaragua	100 g/l EC	foliar	0.012-0.015		2	21
Rice	Paraguay	25 g/l EC	foliar	0.019- 0.025			21
Rice	Paraguay	100 g/l EC	foliar	0.020–0.025			21
Rye	Italy	100 g/kg EC	foliar	0.013–0.018	0.0016-0.003	1-2	21
Rye	Spain	50 g/kg EW	foliar	0.015	0.003-0.0075	1	28
Safflower	Australia	50 g/l EC	foliar	0.007–0.017			14
Sorghum	Australia	50 g/l EC	spraying	0.0175-0.023	-	-	7
Sorghum	Spain	50 g/l EW	foliar	0.015	0.0025-0.0075	1-2	45
Sorghum	USA	84 g/l EC	foliar	0.017–0.056		(0.17)	21
Soya beans	Argentina	12 g/l EC	foliar	0.005–0.008			21
Soya beans	Argentina	75 g/l EC	foliar	0.003–0.006		2	28
Soya beans	Argentina	50 g/l EC	foliar	0.007-0.023	0.005–0.015		21
Soya beans	Australia	50 g/l EC	spraying	0.0065-0.025	-	-	14
Soya beans	Austria	50 g/l EC	foliar	0.01	0.0017-0.005		42
Soya beans	Brazil	25 g/l EC	foliar	0.008–0.010			21
Soya beans	Brazil	150 g/l SC	foliar	0.008–0.010			7
Soya beans	China	50 g/l EC	foliar	0.008–0.015			
Soya beans	Indonesia	25 g/l EC	foliar	0.003–0.005			
Soya beans	Mexico	110 g/l EC	foliar	0.055		2	15
Soya beans	USA	84 g/l EC	foliar	0.017–0.056		(0.22)	21
Spinach	Denmark	50 g/l EW	foliar	0.01–0.015	0.003–0.008	1	
Squash	Mexico	110 g/l EC	foliar	0.055		2	3
Squash	USA	84 g/l EC	foliar	0.034–0.056		(0.28)	3
Stone fruit	Austria	50 g/l EC	foliar	0.013–0.015	0.0013-0.0015		14
Stone fruit	USA	84 g/l EC	foliar	0.028–0.084		(0.42)	14
Sugar beet	Netherlands	25 g/l EC	spraying	0.011		2	
Sugar beets	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	7
Sugar beets	Denmark	50 g/l EW	foliar	0.01–0.015	0.0025–0.0075	1-2	30
Sugar beets	Italy	100 g/l EC	foliar	0.013–0.05	0.0016–0.008	1-2	21
Sugar beets	Spain	50 g/l EW	foliar	0.015	0.0025–0.005	1-2	30
Sugar beets	USA	84 g/l EC	foliar	0.034–0.056		(0.17)	21
Sugar cane	El Salvador	100 g/l EC	foliar	0.036		2	21
Sugar cane	Guatemala	100 g/l EC	foliar	0.036		2	21
Sugar cane	Honduras	100 g/l EC	foliar	0.036		2	21
Sugar cane	Nicaragua	100 g/l EC	foliar	0.036		2	21
Sugar cane	USA	84 g/l EC	foliar	0.034–0.056		(0.22)	21
Sunflower	Argentina	75 g/l EC	foliar	0.005- 0.006		2	28
Sunflower	Argentina	50 g/l EC	foliar	0.014	0.009		14
Sunflower	USA	25 g/l EC	foliar	0.008–0.056		(0.22)	28
Sunflowers	Australia	50 g/l EC	spraying	0.0065-0.025	-	-	14
Swede	Finland	50 g/l EC	foliar	0.010-0.020			S[3]
Sweet corn	Australia	50 g/l EC	spraying	0.015-0.025	-	-	7
Sweet corn	China	50 g/l EC	foliar	0.008–0.011			
Sweet corn	France	25 g/l EC	foliar	0.013	0.0016- 0.003		
Sweet corn	USA	84 g/l EC	foliar	0.017–0.056		(0.56)	1
Tomato	Brazil	25 g/l EC	foliar	0.018–0.019			4
Tomato	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	7
Tomato	Italy	100 g/l EC	foliar		0.003		7
Tomato	Malaysia	15 g/l EC	foliar	0.016–0.025		7	
Tomato	Mexico	110 g/l EC	foliar	.055		2	1
Tomato	Paraguay	25 g/l EC	foliar	0.019–0.030			4
Tomato	Paraguay	100 g/l EC	foliar	0.020–0.030			4
Tomato	Portugal	25 g/l EC	foliar	0.0175	0.0018	1-3	2

Crop	Country	Form	Application				PHI days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl	No.[1]	
Tomato	Spain	50 g/l EW	foliar	0.015			3
Tomato	Spain	25 g/l EC	foliar	0.038	0.0038		3
Tomato	USA	84 g/l EC	foliar	0.017–0.056		(0.56)	1
Tomatoes	Australia	50 g/l EC	spraying	0.0095-0.020	0.0013-0.0028	-	2
Triticale	Spain	50 g/kg EW	foliar	0.015	0.003-0.0075	1	28
Turnip	Finland	50 g/l EC	foliar	0.010-0.020			**Errore. Il segnalibro non è definito.**
Vines	Austria	50 g/l EC	foliar		0.0015		14
Walnuts	Chile	75 g/l EC	foliar		0.0005	1	21
Walnuts	USA	84 g/l EC	foliar	0.056–0.11		(0.22)	21
Watermelon	Chile	75 g/l EC	foliar	0.011–0.019	0.002–0.003	1	3
Watermelon	Mexico	110 g/l EC	foliar	0.055		2	3
Watermelon	USA	84 g/l EC	foliar	0.034–0.056		(0.28)	3
Wheat	Australia	50 g/l EC	spraying	0.0065-0.017	-	-	7
Wheat	Belgium	25 g/l EC	foliar	0.005			42
Wheat	Brazil	25 g/l EC	foliar	0.008–0.020			21
Wheat	Chile	75 g/l EC	foliar	0.011–0.019		1	7
Wheat	China	50 g/l EC	foliar	0.008–0.011			
Wheat	Germany	50 g/l EC	foliar	0.01–0.013	0.0017-0.007	1-2	35
Wheat	Ireland	25 g/l EC	foliar	0.004	0.002	1-2	
Wheat	Luxembourg	25 g/l EC	foliar	0.01–0.015	0.001-0.003	1- 2	42
Wheat	Spain	50 g/kg EW	foliar	0.015	0.003-0.0075	1- 2	28
Wheat	UK	25 g/l EC	foliar	0.003–0.005		1 -3	20
Youngberries	USA	84 g/l EC	foliar	0.028–0.056		(0.17)	7

[1] Figures in parentheses are kg ai/ha/season.

[2] Finland, application at flowering.

[3] Finland, application at seedling or sprouting-budding stage

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials in the following crops.

esfenvalerate	Table 32	tomatoes: France, Italy, Spain USA
esfenvalerate	Table 33	soya beans: USA
esfenvalerate	Table 34	wheat: France, Italy, Spain, UK.
esfenvalerate	Table 35	cotton seed: Greece, Spain, USA
esfenvalerate	Table 36	rape seed: France, Germany, Italy
esfenvalerate	Table 37	wheat straw and forage: France, Italy, Spain, UK
fenvalerate	Table 38	legume forage and fodder: USA
esfenvalerate	Table 39	legume forage and fodder: USA
esfenvalerate	Table 40	rape seed whole plant: Germany

Recent trials were generally well documented with full laboratory and field reports. Laboratory reports included method validation giving batch recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of sample storage were also provided. Although trials included control plots, no control data are recorded in the Tables except where residues in control samples exceeded the LOQ. The results are recorded unadjusted for recovery.

When residues were not detected they are shown as below the LOQ (e.g. <0.1 mg/kg). Residues, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residues from the trials conducted according to maximum GAP have been used for the estimation of maximum residues. These results are double underlined.

Conditions of the supervised residue trials are shown in Table 31. Most trial designs used unreplicated plots, but multiple residues recorded in the Tables refer to replicate plots. Field reports provided data on the sprayers used and their calibration, plot sizes, residue sample sizes and sampling dates. Rape seed trials in Germany and tomato trials in France in the 1980s were reported only in summary form, without field reports. Field sample sizes were not always provided in US trials from the 1970s and 80s.

Periods of freezer storage between sampling and analysis were recorded for all trials and were within the verified stability period of 2 years.

Table 31. Sprayers, plot sizes and field sample sizes in the supervised trials.

Crop	Country	Year	Sprayer	Plot size	Sample size	Compound
Cotton	Greece	1998-9	hand carried boom	77-115 m^2	1 kg	esfenvalerate
Cotton	Spain	1998-9	hand carried boom	76-114 m^2	1 kg	esfenvalerate
Cotton	USA	1984	CO_2 sprayer and tractor-mounted sprayer	35, 74 m^2	not stated	esfenvalerate
Legume forage	USA	1976-79	aircraft, CO_2 sprayer, tractor-mounted sprayer, hand carried sprayer	37 m^2 to 2.0 ha	not stated	fenvalerate
Legume forage	USA	1981-84	aircraft, CO_2 sprayer, helicopter, tractor-mounted sprayer, backpack	37 m^2 to 2.8 ha	not stated	esfenvalerate and fenvalerate
Rape seed	France	1998-99	hand carried boom	60, 75 m^2	0.5 kg min	esfenvalerate
Rape seed	Germany	1987-88	no information (no field reports)	25-100 m^2	1-2 kg and not stated	esfenvalerate
Rape seed	Italy	1998-99	hand carried boom	150 m^2	0.5 kg min	esfenvalerate
Soya bean	USA	1983	tractor mounted spray	100 m^2	not stated	esfenvalerate
Tomato	France	1986	no information (no field reports)	27 m^2	not stated	esfenvalerate
Tomato	Italy	1998-99	hand -gun knapsack, hand carried boom research sprayer	40-150 m^2	2 kg min	esfenvalerate
Tomato	Spain	1998-99	hand-gun knapsack research sprayer, hand carried boom research sprayer	40-125 m^2	2 kg min	esfenvalerate
Tomato	USA	1984-85	CO_2 sprayer, tractor-mounted sprayer	37-56 m^2	1 kg	esfenvalerate
Wheat	France	1995-98	hydraulic knapsack sprayer, hand carried boom	20-75 m^2	1-4 kg	esfenvalerate
Wheat	Italy	1995-96	knapsack motor sprayer	50-200 m^2	1 kg	esfenvalerate
Wheat	Spain	1998-99	hand carried boom	75 m^2	1 kg	esfenvalerate
Wheat	UK	1993	plot sprayer	30 m^2	1 kg	esfenvalerate

Summary information was provided by Poland on a trial with blackcurrants and a trial with garden peas.

Blackcurrants were treated once with esfenvalerate EC at 0.03 kg ai/ ha (2 g ai/hl and 1500 l water/ha) and harvested 7 days later, resulting in a residue of 0.16 mg/kg on the fruits.

Garden peas were treated once with esfenvalerate EC at 0.01 kg ai/ ha (3 g ai/hl and 400 l water/ha). The residue in the green plant 14 days after treatment was 0.08 mg/kg and the residues in the peas and straw 27 days after treatment were <0.005 and <0.01 mg/kg respectively.

Table 32. Esfenvalerate residues in tomatoes resulting from supervised trials in France, Italy, Spain and the USA.

Country, Year (Variety)	Application[1]					PHI days	Esfenvalerate, mg/kg	Report or Study no.
	Form	g ai/ha	g ai/hl	water, l/ha	no.			
France, 1986 (Carma)	EC	15	2.5	600	1	0 7 14	0.01 0.01 <0.01	S/FR/ER86/133 BEER.87.006
France, 1986 (Fantango)	EC	15	1.5	1000	1	0 7 14	0.02 0.01 0.01	S/FR/E86/426 BEER.87.006
France, 1986 (Pyros)	EC	15	1.5	1000	1	0 7 14	0.02 0.02 0.01	S/FR/E86/425 BEER.87.006
France, 1986 (Rio Grande)	EC	15	2.5	600	1	0 7 14	0.02 0.02 <0.01	S/FR/ER86/134 BEER.87.006
Italy, 1998, 820	EC	15	1.5	1000	3	0 1 3	0.05 0.02 0.01	EA980125 ITO1
Italy, 1998, 820	EC	15	1.5	1000	3	3	0.01	EA980125 ITO2
Italy, 1998, PS 176 Petoseed	EC	15	1.9 +1.9 +1.5	800 +800 +1000	g 3	0 1 3	0.01 0.02 0.04	EA980125 ITO3
Italy, 1998, PS 176 Petoseed	EC	15	1.9 +1.9 +1.5	800 +800 +1000	g 3	3	0.02	EA980125 ITO4
Italy, 1999, 121	EC	15	2.5	600	3	3	0.02	EA990128 IT02
Italy, 1999, 690	EC	15	2.5	600	3	3	0.02	EA990128 IT01
Italy, 1999, Madrila	EC	15	1.5	1000	g 3	3	0.02	EA990128 IT04
Italy, 1999, Optima	EC	15	1.5	1000	g 3	3	0.03	EA990128 IT03
Spain, 1998, Conflit	EC	15	2.5	600	3	0 1 3	0.03 0.03 0.02	EA980125 SPO1
Spain, 1998, Riogrande	EC	15	2.5	600	3	3	0.02	EA980125 SPO2
Spain, 1998, Royesta	EC	15	1.9	800	g 3	0 1 3	<0.01 0.01 0.02	EA980125 SPO3
Spain, 1998, Valenciano	EC	15	1.9	800	g 3	3	0.02	EA980125 SPO4

Country, Year (Variety)	Application[1]					PHI days	Esfenvalerate, mg/kg	Report or Study no.
	Form	g ai/ha	g ai/hl	water, l/ha	no.			
Spain, 1999, Bom	EC	15	2.1 +1.5 +1.5	700 +1000 +1000	g 3	3	0.02	EA990128 SP03
Spain, 1999, Bom	EC	15	2.1 +1.9 +1.5	700 +800 +1000	g 3	3	0.03	EA990128 SP04
Spain, 1999, Genaro	EC	15	2.1	700	3	3	<0.01	EA990128 SP01
Spain, 1999, Mia	EC	15	2.5 +2.5 +2.1	600 +600 +700	3	3	0.02	EA990128 SP02
USA (OH), 1984 (Better Boy)	EC	50		215	10	1	0.13 0.28	RIR-24-113-85
USA (TX), 1985 (Monte Grande)	EC	56		94	10	1	0.04 0.03	RIR-24-141-85
USA (DL), 1985 (FM 6203)	EC	56		230	10	1	0.12 0.11	RIR-24-149-85
USA (NC), 1985 (Homestead)	EC	56		560	4	1	0.14 0.13	RIR-24-166-85

[1] g: greenhouse or plastic tunnel

Table 33. Esfenvalerate and fenvalerate residues in soya beans resulting from supervised trials in the USA.

Location, Year (Variety)	Application				PHI days	compound	Residues, mg/kg	Report or Study no.
	Form	g ai/ha	water, l/ha	no.				
CA, 1983 (McCall)	EC	220	94	4	28	fenvalerate	0.07	LLR-41-0012
CA, 1983 (McCall)	EC	58	94	4	28	esfenvalerate	0.04 c 0.01	LLR-41-0012 (RIR-24-191-83)
GA, 1984 (Northrup King S69-96)	EC	52	47	4	21	esfenvalerate	0.02	RIR-24-202-84
AL, 1984 (Braxton)	EC	52	47	4	21	esfenvalerate	<0.01 (2)	RIR-24-227-84

c: sample from control plot

Table 34. Esfenvalerate residues in wheat resulting from supervised trials in France, Italy, Spain and the UK.

Country, Year (Variety)	Application					PHI days	Esfenvalerate, mg/kg [1]	Report or Study no.
	Form	g ai/ha	g ai/hl	water, l/ha	no.			
France (south), 1995 winter wheat (Soissons)	EC	7.5		333	2	42	<0.01	R&D/CRLD/ AN/fb/9616360
France (south), 1995 winter wheat (Soissons)	EC	7.5		333	2	0 3 10 42	ear 0.20 ear 0.16 ear 0.093 grain <0.01	R&D/CRLD/ AN/kd/9616357
France (south), 1996 winter wheat (Ami)	EC	7.4 +7.5		400	2	0 3 10 58	ear 0.24 ear 0.25 ear 0.11 grain <0.01	R&D/CRLD/ AN/vg/9715035
France (south), 1996 winter wheat (Soissons)	EC	7.5		400	2	62	<0.01	R&D/CRLD/ AN/kd/9715022
France (south), 1998 winter wheat (Orcale)	EC	15	5.0	310 +290	2	28	<0.01	EA980121 FR02
France (south), 1998 winter wheat (Soissons)	EC	15 +16	4.3	330 +360	2	13 27	ear 0.33 grain <0.01	EA980121 FR01
Italy, 1995 (Latino)	EC	18	3.6	500	1	21	0.03	EA950177 IIQ95/IT/02
Italy, 1995 (Nobel)	EC	18	3.6	500	1	0 7 14 20 28	ear 0.18 ear 0.10 ear 0.10 ear 0.08 grain <0.01	EA950177 IIQ95/IT/01
Italy, 1996 (Brasilia)	EC	18	3.6	500	1	0 7 14 21 28	ear 0.51 ear 0.64 ear 0.40 grain 0.02 grain 0.02	EA950177 IIQ96/IT/01
Italy, 1996 (Centauro)	EC	18	3.6	500	1	21	0.02	EA950177 IIQ96/IT/02
Spain, 1998 winter wheat (Gadul)	EC	15	5.0	290	2	15 28	ear 0.16 0.06 (immature grain + seed husk)	EA980121 SP01
Spain, 1998 winter wheat (Gadul)	EC	15	5.0	300	2	28	0.07 (immature grain + seed husk)	EA980121 SP02
Spain, 1999 winter wheat (Alcala)	EC	15	3.8	400	2	28	<0.01	EA990137 SP02
Spain, 1999 winter wheat (Estero)	EC	15	3.8	400	2	14 28	ear 0.55 grain <0.01	EA990137 SP01

Country, Year (Variety)	Application					PHI days	Esfenvalerate, mg/kg [1]	Report or Study no.
	Form	g ai/ha	g ai/hl	water, l/ha	no.			
UK, 1993 spring wheat (Promessa)	EC	5		200	1	34	<0.05	AS/2066/SL/1
UK, 1993 spring wheat (Promessa)	EC	5		200	1	21	<0.05	AS/2066/SL/3
UK, 1993 winter wheat (Mercia)	EC	5		200	1	50	<0.05	AS/2066/SL/2
UK, 1993 winter wheat (Riband)	EC	5		200	1	34	<0.05	AS/2066/SL/4

[1] Sample is grain unless stated otherwise.

Table 35. Esfenvalerate residues in cotton seed resulting from supervised trials in Greece, Spain and the USA.

Country, Year (Variety)	Application					PHI days	Esfenvalerate, mg/kg	Report or Study no.
	Form	g ai/ha	g ai/hl	water, l/ha	no.			
Greece, 1998 (Deltapine 20-Corona)	EC	30	3.8	800	3	28	0.01	EA980126 GR02
Greece, 1998 (Eva)	EC	30	3.8	800	3	29	0.04	EA980126 GR01
Greece, 1999 (Alegria)	EC	30	3.8	800	3	28	<0.01	EA990129 GR02
Greece, 1999 (Aria)	EC	30	3.8	800	3	28	<0.01	EA990129 GR01
Spain, 1998 (Nata)	EC	30	3.8	800	3	27	<0.01	EA980126 SP02
Spain, 1998 (Vulcano)	EC	30	5.0 +4.3 +3.8	600 +700 +800	3	29	<0.01	EA980126 SP01
Spain, 1999 (Crema 111)	EC	30	3.8	800	3	29	<0.01	EA990129 SP02
Spain, 1999 (Nata)	EC	30	4.3 +3.8 +3.8	700 +800 +800	3	29	<0.01	EA990129 SP01
USA (GA), 1984 (Stoneville 825)	EC	50		47	10	21	0.01 <0.01	RIR-24-198-84
USA (MS), 1984 (DES 422)	EC	50		94	10	30	<0.01 (2)	RIR-24-208-84

Table 36. Esfenvalerate residues in rape seed resulting from supervised trials in France, Germany and Italy.

Country, Year (Variety)	Application					PHI days	Esfenvalerate, mg/kg	Report or Study no.
	Form	g ai/ha	g ai/hl	water, l/ha	no.			
France (south), 1998 (Synergy)	EC	15	5	300	2	41	<0.01	EA980127 FR01
France (south), 1999 (Pronto)	EC	15	2.7 +3.0	550 +500	2	42	<0.01	EA990130 FR01
Germany, 1987	EC	12.5		400	1	50	<0.01	SMO 295 R76
Germany, 1987 (Jet Neuf)	EC	12.5		400	3	44	<0.01	SMO 295 R74
Germany, 1987 (Jet Neuf)	EC	12.5		400	3	56	<0.01	SMO 295 R75
Germany, 1987 (Jet Neuf)	EC	12.5			2	104	<0.01	SMO 295 R77[1]
Germany, 1988	EC	12.5		400	3	52	<0.01	CMK57 R47
Germany, 1988 (Cares)	EC	12.5		400	3	38	0.021	CMK57 R50
Germany, 1988 (Jet Neuf)	EC	12.5		400	3	43	<0.01	CMK57 R46
Germany, 1988 (Jet Neuf)	EC	12.5		400	3	54	<0.01	CMK57 R49
Italy, 1998 (Raphaela)	EC	15	2.5	600	2	42	<0.01	EA980127 IT01
Italy, 1999 (Oxipent)	EC	15	2.5	600	2	42	<0.01	EA990130 IT01

[1] No residues appeared on the foliage of the whole plant sampled on day 0, which suggests that the trial is invalid.

Table 37. Esfenvalerate residues in wheat straw and forage resulting from supervised trials in France, Italy, Spain and the UK.

Country, Year (Variety)	Application					PHI days	Esfenvalerate, mg/kg [1]	Report or Study no.
	Form	g ai/ha	g ai/hl	water, l/ha	no.			
France (south), 1995 winter wheat (Soissons)	EC	7.5		333	2	42	0.52	R&D/CRLD/ AN/fb/9616360
France (south), 1995 winter wheat (Soissons)	EC	7.5		333	2	42	0.42	R&D/CRLD/ AN/kd/9616357
France (south), 1996 winter wheat (Ami)	EC	7.4 +7.5		400	2	58	0.33	R&D/CRLD/ AN/vg/9715035
France (south), 1996 winter wheat (Soissons)	EC	7.5		400	2	62	0.32	R&D/CRLD/ AN/kd/9715022
France (south), 1998 winter wheat (Orcale)	EC	15	5.0	310 +290	2	28	0.56	EA980121 FR02

Country, Year (Variety)	Application					PHI days	Esfenvalerate, mg/kg [1]	Report or Study no.
	Form	g ai/ha	g ai/hl	water, l/ha	no.			
France (south), 1998 winter wheat (Soissons)	EC	15 +16	4.3	330 +360	2	0 27	green plants 0.52 straw 0.79	EA980121 FR01
Italy, 1995 (Latino)	EC	18	3.6	500	1	21	0.24	EA950177 IIQ95/IT/02
Italy, 1995 (Nobel)	EC	18	3.6	500	1	0 7 14 20 28	shoot 0.18 shoot 0.12 shoot 0.18 shoot 0.16 straw 0.19	EA950177 IIQ95/IT/01
Italy, 1996 (Brasilia)	EC	18	3.6	500	1	0 7 14 21 28	shoot 0.59 shoot 0.74 shoot 0.89 straw 0.76 straw 0.64	EA950177 IIQ96/IT/01
Italy, 1996 (Centauro)	EC	18	3.6	500	1	21	0.76	EA950177 IIQ96/IT/02
Spain, 1998 winter wheat (Gadul)	EC	15	5.0	290	2	0 28	green plants 0.39 straw 0.39	EA980121 SP01
Spain, 1998 winter wheat (Gadul)	EC	15	5.0	300	2	28	0.32	EA980121 SP02
Spain, 1999 winter wheat (Alcala)	EC	15	3.8	400	2	28	0.91	EA990137 SP02
Spain, 1999 winter wheat (Estero)	EC	15	3.8	400	2	0 28	green plant 0.78 straw 0.98	EA990137 SP01
UK, 1993 spring wheat (Promessa)	EC	5		200	1	34	<0.05 c 0.13	AS/2066/SL/1
UK, 1993 spring wheat (Promessa)	EC	5		200	1	21	<0.05 c 0.13	AS/2066/SL/3
UK, 1993 winter wheat (Mercia)	EC	5		200	1	50	<0.05	AS/2066/SL/2
UK, 1993 winter wheat (Riband)	EC	5		200	1	34	<0.05	AS/2066/SL/4

[1] sample is wheat straw unless stated otherwise.

c: control sample

Table 38. Fenvalerate residues in legume forage and fodder resulting from supervised trials in the USA. All EC formulations.

Location, Year (Variety)	Application			PHI	Sample	Fenvalerate mg/kg	Report or Study no.
	g ai/ha	water, l/ha	no.	days			
MN 1979 (Green Giant 359)	220	190	4	21	Pea vines	15 17 70 c 0.31	RIR-24-211-79
MN 1979 (Green Giant 359)	450	190	4	21	Pea vines	47 68 102 c 0.31	RIR-24-211-79
WI 1979 (Early Perfection)	220	300	4	10	Pea vines	3.5 4.4 c 0.03 0.03	RIR-24-218-79
WI 1979 (Early Perfection)	450	300	4	10	Pea vines	8.4 9.0 c 0.03 0.03	RIR-24-218-79
MN 1979 (Green Giant 531)	56	37	a 4	21	Pea vines	5.0 7.4 c 0.12	RIR-24-300-79
MN 1979 (Green Giant 531)	110	37	a 4	21	Pea vines	18 18 c 0.12	RIR-24-300-79
MN 1979 (Green Giant 531)	220	37	a 4	21	Pea vines	3.2 13 c 0.12	RIR-24-300-79
WA 1979 (Perfection)	220	230	4	21	Pea vines	8.7 8.7 c 0.03	RIR-24-383-79
WA 1979 (Perfection)	450	230	4	21	Pea vines	13 19 c 0.03	RIR-24-383-79
WA 1979 (Perfection)	220	47	a 4	21	Pea vines	4.0 4.9 c 0.03	RIR-24-383-79
NE 1976 (Great Northern)	110	230	4	21	Bean straw	11 12 c 0.05	RIR-24-258-76
NE 1976 (Great Northern)	220	230	4	21	Bean straw	22 0.05	RIR-24-258-76
ND 1979 (Navy-Fleetwood)	220	190	4	21	Bean hay	9 10 c 0.11	RIR-24-261-79
ND 1979 (Navy-Fleetwood)	450	190	4	21	Bean hay	20 23 c 0.11	RIR-24-261-79
CA 1979 (Light red kidney)	220	140	a 4	21	Bean vines	11 16	RIR-24-110-80
NY 1979 (California red kidney)	220	410	5	21	Bean vines	5.3 5.3	RIR-24-391-79
NY 1979 (California red kidney)	450	410	5	21	Bean vines	11 12	RIR-24-391-79
NE 1976 (Amsoy 71)	110	230	4	20	Soya bean straw	1.8 1.5	RIR-24-257-76
NE 1976 (Amsoy 71)	220	230	4	20	Soya bean straw	2.7	RIR-24-257-76

Location, Year (Variety)	Application			PHI	Sample	Fenvalerate mg/kg	Report or Study no.
	g ai/ha	water, l/ha	no.	days			
AR 1978 (Lee)	56	47	a 4	20	Soya bean forage	0.44 c 0.01	RIR-24-384-78
AR 1978 (Lee)	110	47	a 4	20	Soya bean forage	0.82 c 0.01	RIR-24-384-78
AR 1978 (Lee)	220	47	a 4	20	Soya bean forage	2.2 c 0.01	RIR-24-384-78
AR 1978 (Lee)	450	47	a 4	20	Soya bean forage	3.1 c 0.01	RIR-24-384-78
NE 1979 (Amsoy 71)	220	190	4	21	Whole soya bean plant	3.7 2.7	RIR-24-258-79
NE 1979 (Amsoy 71)	450	190	4	21	Whole soya bean plant	6.0 5.7	RIR-24-258-79
LA 1979 (Lee 74)	220	190	a 4	0 3 7 14 21 28	Whole soya bean plant	6.8 5.5 6.7 4.2 6.9 4.7 4.7 2.0 3.7 2.9 1.3 1.0	RIR-24-275-79
IN 1979 (Amsoy 71)	220	47	a 4	21	Whole soya bean plant	2.3 2.1 c 0.1	RIR-24-320-79
IN 1979 (Amsoy 71)	450	47	a 4	21	Whole soya bean plant	2.5 2.5 c 0.1	RIR-24-320-79
MS 1979 (Pickett 71)	220	56	4	21	Whole soya bean plant	2.2 2.8	RIR-24-333-79
MS 1979 (Pickett 71)	450	56	4	21	Whole soya bean plant	5.3 7.2	RIR-24-333-79
MS 1979 (Forrest)	220	94	4	21	Soya bean forage	4.2 c 0.02	RIR-24-389-79
IA 1979 (Williams)	220	94	4	21	Soya bean whole plant	5.9 8.5 c 0.02	RIR-24-133-80
SC 1981 (Centennial)	220	2.3 (oil)	a 4	21	Soya bean edible plant	6.7 9.2 c 0.30 0.09	RIR-24-324-81
AL 1982 (Bragg)	220	9.4 (water)	a 4	0 3 7 14 20	Soya bean forage	32 24 32 25 14 c 0.74	RIR-24-123-83

Location, Year (Variety)	Application			PHI	Sample	Fenvalerate mg/kg	Report or Study no.
	g ai/ha	water, l/ha	no.	days			
AL 1982 (Bragg)	220	2.3 (oil)	a 4	0 3 7 14 20	Soya bean forage	15 16 17 10 5.1 c 0.74	RIR-24-123-83
MS 1983 (Centennial)	220	2.3 (oil)	a 4	21	Soya bean forage	0.60 0.81	RIR-24-246-83
CA 1983 (McCall)	220	94	4	28	Whole soya bean plant	3.6 c 0.04	RIR-24-191-83
GA 1984 (Northrup King S69-96)	220	47	4	21	Soya bean hay	13 17	RIR-24-202-84
AL 1984 (Braxton)	220	47	4	21	Soya bean hay	4.9	RIR-24-227-84
CA 1983 (McCall)	220	94	4	28	Soya bean forage	3.6 c 0.04	LLR-41-0012

c: control sample

Table 39. Esfenvalerate residues in legume forage and fodder resulting from supervised trials in the USA. All EC formulations.

Location, Year (Variety)	Application			PHI	Sample	Esfenvalerate mg/kg	Report or Study no.
	g ai/ha	water, l/ha	no.	days			
CA, 1983 (McCall)	58	94	4	28	Whole soya bean plant	2.2 c 0.04	RIR-24-191-83
GA, 1984 (Northrup King S69-96)	50	47	4	21	Soya bean hay	9.1 10	RIR-24-202-84
AL, 1984 (Braxton)	50	47	4	21	Soya bean hay	2.4 3.1	RIR-24-227-84

Table 40. Esfenvalerate residues in whole rape plants resulting from supervised trials in Germany. All EC formulations.

Year (Variety)	Application			PHI days	Esfenvalerate, mg/kg	Report or Study no.
	g ai/ha	water, l/ha	no.			
1987 (Jet Neuf)	12.5	400	3	0 27	0.30 0.06	SMO 295 R74
1987 (Jet Neuf)	12.5	400	3	0 36	0.54 0.20	SMO 295 R75
1987	12.5	400	1	0 28	0.31 0.05	SMO 295 R76
1987 (Jet Neuf)	12.5		2	0 82	<0.01 <0.01	SMO 295 R77 [1]
1988 (Jet Neuf)	12.5	400	3	0 0 24	c 0.073 0.30 0.065	CMK57 R46
1988	12.5	400	3	0 38	0.25 0.018	CMK57 R47
1988 (Jet Neuf)	12.5	400	3	0 43	0.22 0.042	CMK57 R49
1988 (Cares)	12.5	400	3	0 0 30	c 0.021 0.12 0.043	CMK57 R50

[1] No residues appeared on the foliage of whole plants sampled on day 0, which suggests that the trial is invalid.
c: sample from untreated control plot.

Table 41. Residue interpretation table for esfenvalerate residues.

Crop	Country	Use pattern				Trial	Residue, mg/kg
		kg ai/ha	kg ai/hl	no.	PHI days		
Tomato	Spain GAP	0.015			3		
Tomato	Italy trial	0.015	0.0015	3	3	EA980125 ITO1	0.01
Tomato	Italy trial	0.015	0.0015	3	3	EA980125 ITO2	0.01
Tomato	Italy trial	0.015		3	3	EA980125 ITO3	0.04
Tomato	Italy trial	0.015		3	3	EA980125 ITO4	0.02
Tomato	Italy trial	0.015	0.0025	3	3	EA990128 IT02	0.02
Tomato	Italy trial	0.015	0.0025	3	3	EA990128 IT01	0.02
Tomato	Italy trial	0.015	0.0015	3	3	EA990128 IT04	0.02
Tomato	Italy trial	0.015	0.0015	3	3	EA990128 IT03	0.03
Tomato	Spain trial	0.015	0.0025	3	3	EA980125 SPO1	0.02
Tomato	Spain trial	0.015	0.0025	3	3	EA980125 SPO2	0.02
Tomato	Spain trial	0.015	0.0019	3	3	EA980125 SPO3	0.02
Tomato	Spain trial	0.015	0.0019	3	3	EA980125 SPO4	0.02
Tomato	Spain trial	0.015	0.0021	3	3	EA990128 SP03	0.02
Tomato	Spain trial	0.015	0.0021	3	3	EA990128 SP04	0.03
Tomato	Spain trial	0.015	0.0021	3	3	EA990128 SP01	<0.01
Tomato	Spain trial	0.015	0.0025	3	3	EA990128 SP02	0.02
Tomato	Italy GAP		0.003		7		
Tomato	France trial	0.015	0.0025	1	7	S/FR/ER86/133	0.01

Crop	Country	Use pattern				Trial	Residue, mg/kg
		kg ai/ha	kg ai/hl	no.	PHI days		
						BEER.87.006	
Tomato	France trial	0.015	0.0025	1	7	S/FR/ER86/134 BEER.87.006	0.02
Tomato	US GAP	0.056			1		
Tomato	US trial	0.05		10	1	RIR-24-113-85	0.28
Tomato	US trial	0.056		10	1	RIR-24-141-85	0.04
Tomato	US trial	0.056		10	1	RIR-24-149-85	0.12
Tomato	US trial	0.056		4	1	RIR-24-166-85	0.14
Soya beans	US GAP	0.056			21		
Soya beans	US trial	0.058		4	28	RIR-24-191-83	0.04
Soya beans	US trial	0.052		4	21	RIR-24-302-84	0.02
Soya beans	US trial	0.052		4	21	RIR-24-227-84	<0.01
Cereals	France GAP	0.0075	0.004	1			
Wheat	France trial	0.0075		2	42	R&D/CRLD/ AN/fb/9616360	<0.01
Wheat	France trial	0.0075		2	42	R&D/CRLD/ AN/kd/9616357	<0.01
Wheat	France trial	0.0075		2	58	R&D/CRLD/ AN/vg/9715035	<0.01
Wheat	France trial	0.0075		2	62	R&D/CRLD/ AN/kd/9715022	<0.01
Wheat	Spain GAP	0.015		2	28		
Wheat	France trial	0.015	0.005	2	28	EA980121 FR02	<0.01
Wheat	France trial	0.015	0.0043	2	27	EA980121 FR01	<0.01
Wheat	Italy trial	0.018	0.0036	1	21	EA950177 IIQ95/IT/02	0.03
Wheat	Italy trial	0.018	0.0036	1	28	EA950177 IIQ95/IT/01	<0.01
Wheat	Italy trial	0.018	0.0036	1	28	EA950177 IIQ96/IT/01	0.02
Wheat	Italy trial	0.018	0.0036	1	21	EA950177 IIQ96/IT/02	0.02
Wheat	Spain trial	0.015	0.0038	2	28	EA990137 SP02	<0.01
Wheat	Spain trial	0.015	0.0038	2	28	EA990137 SP01	<0.01
Cotton seed	Spain GAP	0.03			30		
Cotton seed	Greece trial	0.030	0.0038	3	28	EA980126 GR02	0.01
Cotton seed	Greece trial	0.030	0.0038	3	29	EA980126 GR01	0.04
Cotton seed	Greece trial	0.030	0.0038	3	28	EA990129 GR02	<0.01
Cotton seed	Greece trial	0.030	0.0038	3	28	EA990129 GR01	<0.01
Cotton seed	Spain trial	0.030	0.0038	3	27	EA980126 SP02	<0.01
Cotton seed	Spain trial	0.030	0.0038	3	29	EA980126 SP01	<0.01
Cotton seed	Spain trial	0.030	0.0038	3	29	EA990129 SP02	<0.01
Cotton seed	Spain trial	0.030	0.0038	3	29	EA990129 SP01	<0.01
Cotton seed	USA GAP	0.056			21		
Cotton seed	USA trial	0.050		10	21	RIR-24-198-84	0.01
Cotton seed	USA trial	0.050		10	30	RIR-24-208-84	<0.01
Rape seed	Germany GAP	0.013		1	56		
Rape seed	France trial	0.015	0.005	2	41	EA980127 FR01	<0.01
Rape seed	France trial	0.015	0.003	2	42	EA990130 FR01	<0.01
Rape seed	Germany trial	0.013		1	50	SMO 295 R76	<0.01
Rape seed	Germany trial	0.013		3	44	SMO 295 R74	<0.01
Rape seed	Germany trial	0.013		3	56	SMO 295 R75	<0.01
Rape seed	Germany trial	0.013		3	52	CMK57 R47	<0.01
Rape seed	Germany trial	0.013		3	43	CMK57 R46	<0.01
Rape seed	Germany trial	0.013		3	54	CMK57 R49	<0.01
Rape seed	Italy trial	0.013	0.003	2	42	EA980127 IT01	<0.01
Rape seed	Italy trial	0.013	0.003	2	42	EA990130 IT01	<0.01
Cereals	France GAP	0.0075	0.004	1			
Wheat straw	France trial	0.0075		2	42	R&D/CRLD/ AN/fb/9616360	0.52

Crop	Country	Use pattern				Trial	Residue, mg/kg
		kg ai/ha	kg ai/hl	no.	PHI days		
Wheat straw	France trial	0.0075		2	42	R&D/CRLD/ AN/kd/9616357	0.42
Wheat straw	France trial	0.0075		2	58	R&D/CRLD/ AN/vg/9715035	0.33
Wheat straw	France trial	0.0075		2	62	R&D/CRLD/ AN/kd/9715022	0.32
Wheat straw	Spain GAP	0.015		2	28		
Wheat straw	France trial	0.015	0.005	2	28	EA980121 FR02	0.56
Wheat straw	France trial	0.015	0.0043	2	27	EA980121 FR01	0.79
Wheat straw	Italy trial	0.018	0.0036	1	21	EA950177 IIQ95/IT/02	0.24
Wheat straw	Italy trial	0.018	0.0036	1	28	EA950177 IIQ95/IT/01	0.19
Wheat straw	Italy trial	0.018	0.0036	1	28	EA950177 IIQ96/IT/01	0.64
Wheat straw	Italy trial	0.018	0.0036	1	21	EA950177 IIQ96/IT/02	0.76
Wheat straw	Spain trial	0.015	0.005	2	28	EA980121 SP01	0.39
Wheat straw	Spain trial	0.015	0.005	2	28	EA980121 SP02	0.32
Wheat straw	Spain trial	0.015	0.0038	2	28	EA990137 SP02	0.91
Wheat straw	Spain trial	0.015	0.0038	2	28	EA990137 SP01	0.98

Livestock feeding trials

Feeding trials are described in the section on animal metabolism

FATE OF RESIDUES IN STORAGE AND PROCESSING

The Meeting received information on the effect of processing on residues of fenvalerate in tomatoes, soya beans and cotton seed and decided that the information could be used in support of esfenvalerate.

Spittler *et al.* (1984) processed tomatoes harvested 24 hours after the last of 13 applications of fenvalerate at 0.11 kg ai/ha in a trial in the USA (MA). The tomatoes (48 kg) were processed into juice and paste and sent to 5 laboratories for analysis. The residue level in chopped whole tomatoes (0.26 mg/kg) was reduced to 0.12 mg/kg in the paste, representing a processing factor of 0.46. Naidu (1990) reported that tomatoes in the USA (CA) had been treated with fenvalerate at 0.22 kg ai/ha in two trials in 1979 and the tomatoes had subsequently been processed into purée and pulp (Table 42). The processing factors for tomatoes to purée were 0.27 and 0.75, mean 0.51.

Barber and Jelatis (1979) treated soya bean plants with fenvalerate at 0.22 kg ai/ha and harvested the beans 48 days later for processing. Residues in the beans (<0.02 mg/kg) produced residues in the hulls (0.06 mg/kg), but not in the extracted meal or refined oil (<0.01 mg/kg).

Gilham and Woodbridge (1978) processed cotton seed from a cotton crop that had been treated with fenvalerate at 0.30 kg ai/ha and harvested 1 day later. Residues in the cotton seed without seed case (0.14 mg/kg) produced residues in the crude oil (0.16 mg/kg), neutral oil (0.23 mg/kg), bleached oil (0.22 mg/kg) and deodorised oil (0.18 mg/kg).

Cotton seed from supervised trials on fenvalerate in the USA was processed and residues measured in the processed commodities (Table 42). Low and undetectable residues made the trials of limited value.

Table 42. Fenvalerate residues in raw and processed commodities resulting from supervised trials in the USA. All EC formulations.

Crop, Location, Year (Variety)	Application			PHI days	Fenvalerate, mg/kg	Report or Study no.
	g ai/ha	water, l/ha	no.			
COTTON						
CA, 1975 (Deltapine)	220	47	a 8	6	seed <0.01 (2) hulls 0.02 soapstock 0.01 solvent-extracted meal <0.01 crude oil <0.01 refined, bleached and deodorised oil <0.01	RIR-24-249-75B
CA, 1975 (Deltapine)	450	47	a 8	6	seed 0.01 0.02 hulls <0.01 soapstock <0.01 solvent-extracted meal <0.01 crude oil <0.01 refined, bleached and deodorised oil <0.01	RIR-24-249-75B
TX, 1979 (TPSA 9070)	220	94	15	21	seed 0.04 meal 0.01 hulls 0.05 crude oil 0.27 0.32 refined oil 0.23 0.30 refined and bleached oil 0.23 soapstock <0.01 c crude oil 0.01 0.01 c refined oil 0.01 c refined and bleached oil 0.01	RIR-24-236-79
TOMATOES						
CA, 1979 (VF-198)	220	280	10	7	fruit 0.14 0.08 purée 0.03 wet pulp 0.10 dry pulp 0.78	RIR-24-204-79
CA, 1979 (GS-12)	220		a 10	7	fruit 0.06 0.10 purée 0.06 wet pulp 0.07 dry pulp 0.56	RIR-24-280-79

c: sample from control plot

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Monitoring data

The US Department of Agriculture Pesticide Data Program (PDP) collects residue data on a variety of fresh and processed fruits and vegetables, grain, and milk. It is designed to be statistically representative of the US food supply. Esfenvalerate is one of the compounds under surveillance.

Table 43. Results for esfenvalerate from the USDA Pesticide Data Program.

Year	No. of samples			Samples with residues in range, mg/kg					
	analysed	with residues	<LOD[1]	≤0.005	>0.005 ≤0.01	>0.01 ≤0.02	>0.02 ≤0.05	>0.05 ≤0.1	>0.1 ≤0.2
apple LOD 0.012-0.31 mg/kg									
1994	367	0	367						
1995	551	2	549				2		
1996	422	10	412				8	2	
apple juice LOD 0.015-0.31 mg/kg									
1996	130	0	130						
1997	546	2	544				2		
1998	555	0	555						
carrots LOD 0.012-0.31 mg/kg									
1994	309	0	309						
1995	561	2	559			2			
1996	404	0	404						
cucumber LOD 0.03-0.098 mg/kg									
1999	491	0	491						
grape juice LOD 0.02-0.098 mg/kg									
1998	528	0	528						
1999	552	0	552						
grapes LOD 0.012-0.085 mg/kg									
1994	352	0	352						
1995	547	0	547						
1996	420	0	420						
lettuce LOD 0.012-0.05 mg/kg									
1994	382	0	382						
1999	185	1	184						1
milk LOD 0.04-0.013 mg/kg									
1996	570	0	570						
1997	727	0	727						
1998	595	0	595						
peaches LOD 0.012-0.14 mg/kg									
1994	185	0	185						
1995	202	4	198			2	1	1	
1996	181	0	181						
canned peaches LOD 0.02-0.098 mg/kg									
1997	412	1	411				1		
potatoes LOD 0.005-0.05 mg/kg									
1994	375	0	375						
1995	569	0	569						
soya bean grain LOD 0.012 mg/kg									
1998	490	0	490						
tomatoes LOD 0.015-0.098 mg/kg									
1996	91	8	83				6	1	1
1997	402	4	398				4		
1998	395	0	395						
1999	209	9	200				9		
canned tomatoes LOD 0.002-0.038 mg/kg									
1999	287	3	284	1			2		

[1] LOD is limit of reporting.

NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was aware of the following national MRLs.

Country	MRL, mg/kg	Commodity
Argentina	0.05	soya bean
	1	sunflower
	10	pasture

Country	MRL, mg/kg	Commodity
Australia	Residue definition:	fenvalerate, sum of isomers
	0.02	edible offal (mammalian)
	0.05	sweet corn (corn-on-the-cob)
	0.2	cattle meat (in the fat), milks (in the fat), tomato
	0.5	goat meat (in the fat), legume vegetables, oilseed, pulses, sheep meat (in the fat)
	1	Brassica (cole or cabbage) vegetables, head cabbages, flowerhead brassicas, Brassica leafy vegetables
	2	celery, cereal grains
	5	wheat bran, unprocessed
	10	primary feed commodities [other than forage and straw and fodder (dry) of cereal grains]
	20	forage of cereal grains, straw and fodder (dry) of cereal grains
Austria	0.02	beans, bushberry, peas, stonefruit
	0.05	apple, pear, rape seed, soya bean
	0.1	grape
	0.02/0.05	fruiting vegetables
	0.2/0.05	cereals
Belgium	0.05	cabbages, wheat
	0.2	barley
Belorussia	0.01	cabbages
	0.02	barley
	0.1	mustard, peas, rape seed
Brazil	0.05	beans, coffee, corn, cotton seed, rice, soya bean, tomato, wheat
Chile	0.02	corn, potato
	0.05	cauliflower
	0.2	almond, artichoke, walnut
	0.5	cucumber
	1	melon, pepper, pumpkin, tomato, watermelon
	2	apple, broccoli, pear
	10	cabbages, cherries, nectarine, peach, plum
Denmark	0.02	carrot, leek, maize, onion, peas, soya bean, sugar beet
France	0.02	almond, apricot, asparagus, cucurbits, linseed, peach, sweet corn
Germany	Residue definition	Sum of (*R,R*) and (*S,S*) isomers[1]
	0.02 (*)	fat contained in poultry meat, preparations of poultry meat, poultry offal and fat, milk, milk products, eggs, other food of plant origin
	0.05	Brussels sprouts, head cabbage, hops, oil seed, pome fruit, rye, tea, tealike products, tomatoes, triticale, wheat
	0.1	grapes
	0.2	barley, oats, fat contained in meat, preparations of meat, offal and animal fats (except poultry)
Germany	Residue definition	Sum of (*R,S*) and (*S,R*) isomers[1]
	0.02 (*)	fat contained in poultry meat, preparations of poultry meat, poultry offal and fat, milk, milk products, eggs, other food of plant origin
	0.05	barley, hops, oats, oil seed, tea, tealike products, fat contained in meat, preparations of meat, offal and animal fats (except poultry)
Kazakhstan	0.01	cabbages
	0.1	mustard, rape seed
Korea	0.2	cucumber
	1	red pepper
	2	apple, citrus, pear
	3	Chinese cabbage
Mexico	0.02	potato
	0.05	soya bean
	0.2	almond, cotton seed
	0.5	cucumber
	1	melon, pepper, squash, tomato, watermelon
	2	apple, broccoli
Moldavia	0.01	cabbages
	0.02	barley
	0.1	mustard, peas, rape seed
Russia	0.01	cabbages
	0.02	barley

Country	MRL, mg/kg	Commodity
	0.1	mustard, peas, rape seed
Taiwan	0.5	fruiting vegetables
	2	leafy vegetables
Thailand	7	cotton seed, crucifers
Ukraine	0.01	cabbages, grape
	0.02	barley
	0.1	peas, rape seed
	5	hops
USA	0.03	eggs, whole, poultry liver, poultry meat
	0.3	poultry fat
	0.5	kiwifruit, sugar beet roots
	1	artichoke
	2	kohlrabi
	5	lettuce head, mustard greens, sugar beet tops
	7	sorghum grain
	10	sorghum fodder, sorghum forage
Uzbekistan	0.01	cabbages
	0.02	barley
	0.1	mustard, peas, rape seed

* lower limit of analytical determination.

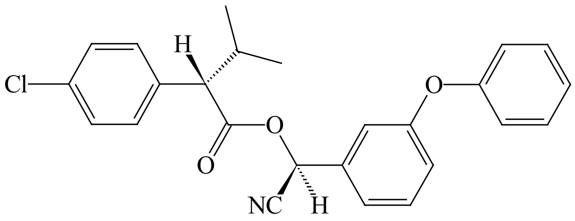
[1] In most countries the residue definition for both esfenvalerate and fenvalerate is the sum of all isomers. In the EU MRLs for esfenvalerate and fenvalerate are set separately for the sum of the (*R,R)* and (*S,S*) isomers and for the sum of (*R,S*) and (*S,R*) isomers.

APPRAISAL

Residue and analytical aspects of esfenvalerate were considered for the first time by the present Meeting.

It should be noted that fenvalerate (119) was first considered in 1979. Advice has been received that fenvalerate will be supported by the data submitter during the review process for esfenvalerate and possibly post-review (Annex 1 of CCPR Report 2002, ALINORM 03/24). The Meeting was informed that fenvalerate registrations are withdrawn in some European countries but will continue in other countries including Japan and USA.

Esfenvalerate is a broad-spectrum pyrethroid insecticide with uses on many crops.



Relation between fenvalerate and technical esfenvalerate - typical isomer compositions

	S,S-isomer	R,S-isomer	S,R-isomer	R,R-isomer
Fenvalerate	23%	27%	27%	23%
technical esfenvalerate	84%	8%	7%	1%

The Meeting received information on esfenvalerate metabolism and environmental fate, methods of residue analysis, freezer storage stability, national registered use patterns, supervised residue trials and national MRLs. Information on fenvalerate was also supplied on these topics in support of esfenvalerate.

Animal metabolism

The Meeting received metabolism studies for esfenvalerate and fenvalerate on rats and mice, and for fenvalerate on dairy cows and laying hens.

The following compounds were identified as metabolites of esfenvalerate in rats or mice, appearing in the excreta in amounts exceeding 5% of the dosed parent compound: 4'-OH-esfenvalerate; 2-(4-chlorophenyl)isovaleric acid (CPIA); 2-(4-chlorophenyl)-2-hydroxy-3-hydroxymethyl butanoic acid (2,3-OH-CPIA); 3-(4'-hydroxyphenoxy)benzoic acid (free + conjugated); 3-phenoxybenzoic acid (free + conjugated).

In a dairy cow metabolism study with [^{14}C]fenvalerate the residue rapidly reached a plateau in milk (by day 3). Approximately 90% of the ^{14}C in the milk was accounted for by fenvalerate itself and almost all of the ^{14}C in the milk was present in the fat. A comparison of the ^{14}C measurement on fat tissues and fat of milk with a fenvalerate measurement by GLC showed that most of the ^{14}C was present as fenvalerate itself. Carboxylic metabolites of fenvalerate and their conjugates were identified in the liver and kidney.

Fenvalerate was identified as the major component of the residue in fat comprising 81-85% of the radiolabel in fat from laying hens dosed with labelled fenvalerate. Fenvalerate residues were identified in the egg yolks.

Fenvalerate, and esfenvalerate as a component of fenvalerate, should be defined as a fat-soluble residue.

Plant metabolism

The Meeting received plant metabolism studies for esfenvalerate on cabbages and for fenvalerate on apple trees, cabbages, kidney bean, lettuce, soybean, tomato and wheat.

In a comparative study on cabbages it was found that the nature and amounts of transformation products formed from fenvalerate and esfenvalerate were very similar. Most of the applied radiolabel remained on the treated leaves with little translocation to other parts of the plant. No αS/αR epimerisation was observed for residues in cabbage treated with esfenvalerate. After 24 and 48 days the parent compound (fenvalerate or esfenvalerate) was still the major identified component of the remaining residue. The main identified metabolite was free and conjugated CPIA. Dec-fen (3-(4-chlorophenyl)-4-methyl-2-(3-phenoxyphenyl) pentanenitrile), a photolysis product, was identified as a minor component of the residue.

In the fenvalerate metabolism studies, fenvalerate was a surface residue and solvent extractable. Parent fenvalerate constituted the main identified component of the residue. A number of metabolites were identified including the photoproduct Dec-fen, which is unlikely to be an animal metabolite. Dec-fen constituted 5-10% of the residue on crop foliage.

Environmental fate

Soil

The Meeting received information on the behaviour and fate of esfenvalerate during soil and solution photolysis, aerobic soil metabolism and field dissipation. Information was also provided on the soil adsorption properties of esfenvalerate and on the behaviour and fate of fenvalerate during soil photolysis, aerobic and anaerobic soil metabolism, column leaching of aged residues, field dissipation and crop rotation.

Esfenvalerate is susceptible to soil surface photolysis (half-life 3-4 days). A study of photoisomerization of esfenvalerate in solution predicted that epimerization induced by sunlight will be generally minor.

Aerobic soil metabolism of fenvalerate and esfenvalerate occurred at much the same rates and their behaviour in the soil was generally comparable. The configuration of esfenvalerate was not converted to any other configuration, i.e. epimerization was not apparent. Esfenvalerate was the major part of the environmental residue. The behaviour of fenvalerate under aerobic and anaerobic conditions was similar.

Adsorption-desorption and leaching studies indicate that esfenvalerate will be highly immobile in soils. In field dissipation, the residues of esfenvalerate did not move down the soil profile and dissipated with half-lives of approximately 60-130 days.

In fenvalerate crop rotation studies, little of the residue carried over to the succeeding crop and none of the residue was fenvalerate itself. Part of the carry-over residue was identified as a conjugate of CPIA.

Water-sediment systems

The Meeting received information on the behaviour of esfenvalerate and fenvalerate during aqueous sterile hydrolysis and the fate of esfenvalerate in water-sediment systems.

Hydrolysis rates at pH 5 and 7 were too small to be measurable in 28 days. At pH 9 the half-lives were quite similar - 80 and 64 days for fenvalerate and esfenvalerate respectively. Epimerization of esfenvalerate occurred at pH 7 and pH 9 at the α-position. Epimerization was faster than hydrolysis. At pH 9 from day 2 through the rest of the experiment the level of [2S,αR] was slightly higher than or equal to the esfenvalerate level. At pH 7 the epimerization rate was slower but substantial. After 14 days at pH 7 the ratio of esfenvalerate to [2S,αR] epimer was 2.5.

CPIA was the most prevalent metabolite in water-sediment systems and became the major part of the residue to occur in the water phase.

Analytical methods

Samples in the field trials were analysed for esfenvalerate by solvent extraction, cleanup by solvent partition and column chromatography followed by GC-ECD measurement. Validation with an LOQ of 0.01 mg/kg was achieved for numerous commodities.

The RS,SR pair elutes before the SS,RR pair on GC analysis, so significant racemization of esfenvalerate to fenvalerate would be apparent as a changed peak ratio.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the stability of esfenvalerate residue samples during storage of analytical samples at freezer temperatures. Test data were provided on the following substrates: almonds, beef, blackberries, cabbage, corn silage, eggs, green beans, lettuce, milk, peach, soil, soybeans, sugar beets, tomatoes, watermelon, wheat grain and wheat straw.

Esfenvalerate residues were stable in storage at -10°C for the 2-3 years of the tests. No significant racemization of esfenvalerate was observed during storage. The RS,SR pair elutes before the SS,RR pair in the GC analysis, but was not observed in the stored samples.

Residue definition

Fenvalerate was introduced as a pesticide before esfenvalerate and residue limits for fenvalerate were usually defined as the sum of the fenvalerate isomers. In national systems esfenvalerate residues then conveniently fitted into the fenvalerate residue definition.

The residue definition for esfenvalerate should consist of the SS isomer only. However, separation of the SS and RR isomers would be analytically expensive and generally would serve little purpose because the level of RR isomer in esfenvalerate is typically only about 1%.

The hydrolysis studies suggest that epimerization of esfenvalerate is possible and that some of the SS isomer could be converted to SR isomer and appear as such as residues. In crop and animal residue situations epimerization probably is insignificant (<10%) and the SR isomer (initially a 7% component of technical esfenvalerate) remains a minor component of the residue. The RS,SR pair elutes before the SS,RR pair in the GC analysis and, if the SR isomer is included, the RS should also be included because they are not separated in routine analytical methods.

It should be noted that most of the residue data for esfenvalerate are recorded as the sum of all isomers. A residue of SS+RR isomers would generally be about 15% less than the sum of all isomers, but in practice 15% makes little difference in comparison with inherent residue variability.

The FAO Manual (page 51) states that preferably no compound, metabolite or analyte should appear in more than one residue definition. It follows that, while a fenvalerate CXL is maintained for the relevant commodity, the residues of esfenvalerate may be accommodated into the fenvalerate residue definition.

At least while fenvalerate MRLs are maintained, the residue definition for esfenvalerate as "fenvalerate, sum of all isomers" might be a practical solution.

The Meeting agreed that the residue definition for esfenvalerate would be the sum of fenvalerate isomers.

Definition of esfenvalerate residue (for compliance with MRL and for estimation of dietary intake): sum of fenvalerate isomers.

The residue definition is worded to emphasise that all fenvalerate isomers are included, but the intention is that the residue definition is identical to that for fenvalerate.

The definition applies to plant and animal commodities. The residue is classed as fat-soluble.

Results of supervised trials

Supervised trials were available for the use of esfenvalerate on tomatoes, soybeans, wheat, cotton seed and rapeseed.

Supervised residue trials for fenvalerate were also provided but were not used because the fenvalerate application rate did not match the esfenvalerate GAP application rate.

Tomato. Italian GAP permits the use of esfenvalerate on tomatoes at a spray concentration of 0.003 kg ai/hl with harvest 7 days later. In two French trials in line with Italian GAP the residues were 0.01 and 0.02 mg/kg.

Spanish GAP allows the use of esfenvalerate on tomatoes at a rate of 0.015 kg ai/ha and harvest 3 days later. Residues from 8 Italian and 8 Spanish trials with conditions matching Spanish GAP were: <0.01, 0.01 (2), 0.02 (10), 0.03 (2) and 0.04 mg/kg.

In USA esfenvalerate may be used on tomatoes at 0.056 kg ai/ha with harvest permitted 1 day later. In four US trials with conditions matching US GAP the esfenvalerate residues were: 0.04, 0.12, 0.14 and 0.28 mg/kg.

The data populations from European and US trials appear to be different and should not be combined. The number of tomato trials (4) from the higher population was insufficient to make a recommendation so the recommendations are based on the European trials. There are 18 trials with highest and median values of 0.04 and 0.02 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for esfenvalerate in tomatoes of 0.1, 0.02 and 0.04 mg/kg, respectively.

Esfenvalerate residues complying with the estimated maximum residue level of 0.1 mg/kg would not exceed the current fenvalerate MRL of 1 mg/kg for tomatoes.

Soybeans. In the USA esfenvalerate may be used on soybeans at 0.056 kg ai/ha and with harvest 21 days after the final application. In 3 US trials with the GAP application rate and PHIs of 21 and 28 days the esfenvalerate residues were: <0.01, 0.02 and 0.04 mg/kg.

The number of trials was insufficient for an MRL recommendation.

Esfenvalerate residues from these trials in line with US GAP did not exceed the current fenvalerate MRL of 0.1 mg/kg for soya bean.

Wheat. In France esfenvalerate is registered for use on cereals at 0.0075 kg ai/ha. No PHI is specified. In four French trials on wheat with application rate 0.0075 kg ai/ha and PHI 42-62 days the residues in wheat grain were all below LOQ (0.01 mg/kg).

Esfenvalerate may be used on wheat in Spain with 2 applications at 0.015 kg ai/ha and harvest 28 days after the second application. In 2 Spanish trials, 4 Italian trials and 2 French trials with conditions matching Spanish GAP esfenvalerate residue levels were: <0.01 (5), 0.02 (2) and 0.03 mg/kg. Harvest of two Italian trials was 21 days after treatment, which was considered sufficiently close to the prescribed 28 days to be valid.

Residues in wheat from a trial matching UK GAP (3 applications of 0.005 kg ai/ha and 20 days PHI), except that there was only 1 application instead of 3, were <0.05 mg/kg. The trial data were not used because the LOQ (0.05 mg/kg) was substantially higher than the LOQ (0.01 mg/kg) for the other trials.

In summary, residues in the 12 trials matching GAP were: <0.01 (9), 0.02 (2) and 0.03 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for esfenvalerate in wheat of 0.05, 0.01 and 0.03 mg/kg, respectively.

Esfenvalerate residues complying with the estimated maximum residue level of 0.05 mg/kg would not exceed the current fenvalerate MRL of 2 mg/kg for cereal grains.

Cotton seed. Esfenvalerate is registered for use on cotton in Spain at 0.03 kg ai/ha with a 30 day PHI. In four Greek trials with conditions matching Spanish GAP the residues on cotton seed were: <0.01 (2), 0.01 and 0.04 mg/kg. In four Spanish trials also with conditions matching Spanish GAP the residues in cotton seed were: <0.01 (4) mg/kg.

In USA esfenvalerate is registered for use on cotton at 0.056 kg ai/ha with a 21 days PHI. Esfenvalerate residues on cotton seed were <0.01 and 0.01 mg/kg in two US trials where the application rate was 0.050 kg ai/ha and the intervals to harvest were 30 and 21 days.

The residue data from US and Europe appear to be from the same population. In summary the residues from the 10 cotton seed trials are, in rank order, median underlined: <0.01 (7), 0.01 (2), 0.04 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for esfenvalerate in cotton seed of 0.05, 0.01 and 0.04 mg/kg, respectively.

Esfenvalerate residues complying with the estimated maximum residue level of 0.05 mg/kg would not exceed the current fenvalerate MRL of 0.2 mg/kg for cotton seed.

Rapeseed. Esfenvalerate may be used on rapeseed in Germany with one application at 0.013 kg ai/ha with a 56 days PHI. Residues in rapeseed were below LOQ (0.01 mg/kg) in rapeseed from 6 trials in Germany (1-3 applications of 0.013 kg ai/ha and 43-56 days PHI) 2 trials in France (2 applications of 0.015 kg ai/ha, 41-42 days PHI) and 2 Italian trials (2 applications of 0.013 kg ai/ha and 42 days PHI).

Although all residues were below LOQ there was no evidence that the residue levels were essentially zero; STMR and HR were therefore recommended at the LOQ..

The Meeting estimated a maximum residue level, an STMR value and an HR value for esfenvalerate in rapeseed of 0.01*, 0.01 and 0.01 mg/kg, respectively.

Wheat straw and forage. The twelve trials that produced wheat data also produced wheat straw data. Two additional trials from Spain produced straw data within GAP. The esfenvalerate residues in the 14 trials in rank order, median underlined, are: 0.19, 0.24, 0.32, 0.32, 0.33, 0.39, 0.42, 0.52, 0.56, 0.64, 0.76, 0.79, 0.91 and 0.98.

The Meeting estimated a maximum residue level and an STMR value for esfenvalerate in wheat straw and fodder of 2 and 0.47 mg/kg, respectively.

Soybean hay. Residue data were provided for soybean hay and whole soybean plant from the 3 US soybean trials already considered. The number of trials was insufficient for an MRL recommendation.

Rapeseed whole plant. Rapeseed whole plant residue data were provided from the German trials already considered for rapeseed. If the permitted interval between treatment and cutting for forage is the same as for rapeseed harvest (56 days) the conditions of the trials do not match GAP and the trials cannot be evaluated.

Processing

The Meeting received processing information for residues of fenvalerate in tomatoes, soybeans and cotton seed and decided that the information could be used in support of esfenvalerate.

The cotton seed and soybean data were of limited value because of 'non-detect' values and some inconsistency. In one cotton seed study the crop was harvested only 1 day after treatment so the residues may not have been representative of 21-30-day old residues as required by current GAP.

The processing factor for tomatoes to paste was 0.46 and for tomatoes to puree 0.51. The Meeting applied the processing factors to the tomato STMR (0.02) to produce STMR-Ps of 0.01 mg/kg for tomato paste and tomato puree.

Farm animal dietary burden

The Meeting estimated the farm animal dietary burdens for esfenvalerate.

Maximum farm animal dietary burden estimation

Commodity	group	residue mg/kg	basis	% dry matter	residue, on dry wt mg/kg	*Choose diets, %*			Residue contribution, mg/kg		
						Beef cattle	*Dairy cattle*	*Poultry*	Beef cattle	Dairy cattle	Poultry
Cotton seed	SO	0.05	MRL	88	0.056	*25*			0.014		
Wheat straw and fodder	AS	2	MRL	88	2.3	*25*	*60*		0.57	1.4	
Wheat	GC	0.05	MRL	89	0.056	*50*	*40*	*80*	0.028	0.023	0.045
					TOTAL	*100*	*100*	*80*			
						Maximum dietary burden			**0.61**	**1.6**	**0.045**

STMR farm animal dietary burden estimation

Commodity	group	residue mg/kg	basis	% dry matter	residue, on dry wt mg/kg	*Choose diets, %*			Residue contribution, mg/kg		
						Beef cattle	*Dairy cattle*	*Poultry*	Beef cattle	Dairy cattle	Poultry
Cotton seed	SO	0.01	MRL	88	0.011	*25*			0.003		
Wheat straw and fodder	AS	0.47	STMR	88	0.53	*25*	*60*		0.13	0.32	
Wheat	GC	0.01	STMR	89	0.011	*50*	*40*	*80*	0.006	0.005	0.009
					TOTAL	*100*	*100*	*80*			
						STMR dietary burden			**0.14**	**0.32**	**0.009**

The esfenvalerate dietary burdens for animal commodity MRL and STMR estimation (residue levels in animal feeds expressed on dry weight) are: beef cattle 0.61 and 0.14 mg/kg, dairy cattle 1.6 and 0.32 mg/kg and poultry 0.045 and 0.009 mg/kg.

Farm animal feeding studies

The dairy cow feeding study with [^{14}C]fenvalerate was designed to provide residue transfer information as well as metabolism information. The level of fenvalerate in the animal diet was 79 ppm. Approximate levels of ^{14}C and % as fenvalerate were: fat 1-3 mg/kg (90%+), milk 0.47 mg/kg (90%+), muscle 0.25 mg/kg (90%), liver 2 mg/kg (<1%) and kidney 1.4 mg/kg (17%).

White Leghorn laying hens were dosed with [^{14}C]fenvalerate at the equivalent of 158 ppm in the feed in a metabolism study that also provided information on residue levels in tissues and eggs. Approximate levels of ^{14}C and % as fenvalerate were: fat 0.5 mg/kg (81-85%), egg yolk 1-1.3 mg/kg (52-70%), liver 1-2.4 mg/kg (insignificant %), muscle <0.2 mg/kg, egg whites <0.2 mg/kg.

Animal commodity maximum residue levels

The feeding levels in the fenvalerate metabolism studies (cow 79 ppm and hen 158 ppm) were so much higher than the maximum dietary burdens for esfenvalerate (cow 1.6 mg/kg and hen 0.045 mg/kg) that it is not reasonable to make calculations. It is reasonable to conclude that the residues will be ‘much less’ than in the feeding studies and probably mostly below LOQ.

The Meeting noted that the residues of esfenvalerate in mammalian products arising from the farm animal diet would not exceed the MRLs already established for fenvalerate for:

- meat (from mammals other than marine mammals) 1 mg/kg (fat); and
- edible offal (mammalian) 0.02 mg/kg; and
- milks 0.1 mg/kg F..

The Meeting estimated maximum residue levels of 0.01* mg/kg for poultry meat (fat), poultry offal and eggs. In the absence of more definitive information the Meeting decided to estimate STMR and HR values at the LOQ for poultry meat, poultry edible offal, poultry fat and eggs.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the maximum residues listed below are suitable for establishing maximum residue limits and for IEDI assessment. Relevant fenvalerate CXLs are included for information.

Definition of the residue (for compliance with MRLs and for estimation of dietary intake): sum of fenvalerate isomers. The residue is fat-soluble.

CCN	Commodity Name	MRL, mg/kg	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg	Fenvalerate CXL, mg/kg
SO 0691	Cotton seed	0.05	0.01	0.04	0.2
MO 0105	Edible offal (mammalian)				0.02
PE 0112	Eggs	0.01*	0.01	0.01	
MM 0095	Meat (from mammals other than marine mammals)				1 (fat)
ML 0106	Milks				0.1 F
PM 0110	Poultry meat	0.01* (fat)	0.01	0.01	
PO 0111	Poultry, Edible offal of	0.01*	0.01	0.01	
SO 0495	Rape seed	0.01*	0.01	0.01	
VD 0541	Soya bean (dry)				0.1
VO 0448	Tomato	0.1	0.02	0.04	1
	Tomato paste		0.01		

CCN	Commodity Name	MRL, mg/kg	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg	Fenvalerate CXL, mg/kg
	Tomato purée		0.01		
GC 0654	Wheat	0.05	0.01	0.03	2 (cereal grains)
AS 0654	Wheat straw and fodder, dry	2	0.47		

* the MRL is estimated at or about the LOQ

DIETARY RISK ASSESSMENT

Long-term intake

The Meeting decided to treat esfenvalerate and fenvalerate together for the purposes of dietary risk assessment because the residues consist of the same components but in different proportions.

Fenvalerate has not been recently evaluated so STMRs and HRs are not available. The TMDIs for fenvalerate for the five GEMS/Food regional diets were in the range 50-70% of the ADI, 0.02 mg/kg bw/day (Annex 3).

Esfenvalerate IEDIs for the five GEMS/Food regional diets for the crop and farm animal commodities where STMRs are available were <1% of the ADI, 0.02 mg/kg bw/day (Annex 3).

When esfenvalerate IEDIs were added to the fenvalerate TMDIs the estimated intakes for the five GEMS/Food regional diets were in the range 50-70% of the ADI (Annex 3).

The Meeting concluded that the long-term intake of residues of esfenvalerate resulting from its uses that have been considered by JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short term Intake (IESTI) for esfenvalerate was calculated for 6 food commodities [(and their processed fractions)] for which maximum residue levels were estimated and for which consumption data were available. The results are shown in Annex 4.

The IESTI represented 0-3% of the acute RfD for the general population and 0-10% of the acute RfD for children. The Meeting concluded that the short-term intake of residues of esfenvalerate, resulting from its uses that have been considered by the JMPR, is unlikely to present a public health concern.

REFERENCES

Akhtar, M.H., Trenholm, H.L. and Hamilton, R.M.G. 1989. Metabolism of fenvalerate in laying hens. *J. Agric. Food Chem.*, 37, 190-196.

Barber, G.F. and Jelatis, J.C. 1979, Residue levels of SD 43775 in soybeans (and processed products) receiving four applications of SD 43775, a Mississippi study. Project 61567.32, Report TIR-24-413-78. Report AR-91-0406. Shell Development Co, UK. Unpublished.

Barber, G.F., Wendt, M.B. and Winter, W.T. 1981. Determination of SD 43775 residues in tissues, blood plasma, urine and feces of cows fed 78 ppm ^{14}C-SD 43775. Report RIR-24-617-80-B. Project 61567.20. Shell Development Co, USA. Du Pont Report AMR-1501-89. Unpublished.

Behmke, F.D. and Lee, P.W. 1991. Magnitude of fenvalerate residues in cottonseed after Pydrin® and Asan® insecticide applications. Report AMR-1881-90, includes trials RIR-24-198-84, RIR-24-208-84. RIR-24-249-75B, RIR-24-236-79. E.I. duPont de Nemours and Company, USA.. Report LLR-0270, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Benwell, L. 1996a. Esfenvalerate: the validation of the analytical method for the determination of the residue in wheat grain and straw (Method CHE 333/56-02R). Corning Hazleton. Report No. 333/56-1012. Report

LLA-0071, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Benwell, L. 1996b. Esfenvalerate: the validation of the analytical method for the determination of the residue in barley grain and straw (Method CHE 333/56-02R). Corning Hazleton. Report No. 333/57-1012. Report LLA-0072, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Benwell, L. and Burden, A. 1997. Esfenvalerate: the evaluation of residues stability in wheat grain and straw under deep freeze storage conditions. Report 16/312-1016, Covance Laboratories, UK. Cyanamid, UK. Report LLR-0274, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Bosio, P.G. 1986. Residues of esfenvalerate in tomatos from France treated with Sumialpha. -1986 trials-Shell Chemie France. Report BEER.87.006. Report LLR-0188, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Boyer, A.C. and Lee, P.W. 1981. Identification of SD 43775 and its metabolites in cow kidney and liver tissue after feeding carbon-14 labeled SD 43775. Regulatory Information Record RIR-22-003-81. Shell Development Co, USA. Du Pont Report AMR-1501-89. Unpublished.

Burden, A.N. 1995. Esfenvalerate: the validation of the analytical method for the determination of residues in water. (Method CLE 333/40-01R). Corning Hazleton. Report No. 333/40-1012. Report LLA-51-0068, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Burden, A.N. 1997. ‘Sumi-alpha’ (containing 25 g esfenvalerate/l): dissipation from two field soils (autumn application). Report 333/15-1012b, Covance, UK. Report LLR-0272, Sumitomo Chemical Co, Ltd, Japan. Unpublished.

Burden, A.N. 1998. ‘Sumi-alpha’ (containing 25 g esfenvalerate/l): dissipation from two field soils (summer application). Report 333/15-1012a, Covance, UK. Report LLR-0271, Sumitomo Chemical Co, Ltd, Japan. Unpublished.

Croucher, A. 1998a. Esfenvalerate: validation of an analytical method for the determination of residue in rapeseed and rapeseed oil. (Method CLE 333/116-04R). Report CLE 333/116-D2140, Covance Laboratories, UK.. Report LLA-0082, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Croucher, A. 1998b. Esfenvalerate: validation of an analytical method for the determination of residue in hen (Method CLE 333/107-04R). Covance Laboratories. Report No. CLE 333/107-D2140. Report LLA-0083, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Croucher, A. 1998c. Esfenvalerate: validation of an analytical method for the determination of residue in cattle tissues (Method CLE 333/108-04R). Covance Laboratories. Report No. CLE 333/108-D2140. Report LLA-0084, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Edmunds, J.W., Macdonald, I.A. and Gillis, N.A. 1988. The determination of concentration of esfenvalerate in rape. Huntingdon Research Centre, UK, Project SMO 295. Report LLR-81-0034, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Ehmann, A. 1979a. The fate of ^{14}C-SD 43775 (fenvalerate) applied to tomato leaves. Report TIR-22-116-79, Shell Research Ltd. Report AM-91-0115, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Ehmann, A. 1979b. The fate of ^{14}C-SD 43775 (fenvalerate) applied to immature tomato fruits-a range-finding study. Report TIR-22-104-79, Shell Research Ltd. Report AM-91-0114, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Ehmann, A. 1979c. The fate of [^{14}C] SD 43775 (fenvalerate) applied to developing pods of soybean plants. Report TIR-22-112-79, Shell Research Ltd. Report AM-91-0117, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Ehmann, A. 1979d. The fate of [^{14}C] SD 43775 (fenvalerate) applied to the leaves of soybean plants. Report TIR-22-111-79, Shell Research Ltd. Report AM-91-0116, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Fan, H.Y. and Lee, P.W. 1980. A 30, 60, and 120-day rotation crop study using carbon-14 labeled-chlorophenyl and phenoxyphenyl-SD 43775. Report TIR-22-004-80, Shell Development Company. Report AM-01-0122, Sumitomo Chemical Co, Ltd, Japan. Unpublished.

Farrell, K.J. 1995. Sumi-alpha esfenvalerate: The determination of residues in wheat and spring wheat. Report 4054. Project 9582/94097/83 (UK), includes trials AS/2066/SL/1, AS/2066/SL/2, AS/2066/SL/3, AS/2066/SL/4. Gosport. Report LLR-51-0241, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Gilham, J.A. and Woodbridge, A.P. 1978. Residues of WL 43775 in products from processed cottonseed from Colombia. Project SBL/8/78/I/AC 505. Report BLGR.0076.78. Report AR-81-0423. Shell Research, Ltd, UK. Unpublished.

Gillis, N.A., Macdonald, I.A. and Barnes, I. 1989. The determination of residual concentrations of esfenvalerate in rape seed and plant. Huntingdon Project CMK 57, Document 303FX-532-1601. Shell Agrar GmbH & Co. Report LLR-91-0100, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Grolleau, G. 1997. Magnitude of the residue of esfenvalerate in wheat raw agricultural commodity. Italy 1995 and 1996. European Agricultural Services. Study EA950177. Corning Hazleton ref 333/63. Report LLR-0244, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Grolleau, G. 1998. Validation of the modified DFG method S 19 for the determination of esfenvalerate in field specimens of water containing plant material (tomato). Study EAS/98-058, Defitraces, France. Report LLA-0093, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Grolleau, G. 1999a. Magnitude of the residue of esfenvalerate in cotton raw agricultural commodity. Greece and Spain 1998. European Agricultural Services. Study EA980126. Dr Specht and Partner, project SUM-

9806. Report LLR-0286, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Grolleau, G. 1999b. Magnitude of the residue of esfenvalerate in field and greenhouse tomato raw agricultural commodity. Italy and Spain 1998. Study EA980125. European Agricultural Services. Study EAS/98-059, Defitraces. Report LLR-0285, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Grolleau, G. 1999c. Magnitude of the residue of esfenvalerate in rapeseed raw agricultural commodity. Southern France and Italy 1998. European Agricultural Services. Study EA980127. Dr Specht & Partner, Project SUM-9807. Report LLR-0287, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Grolleau, G. 1999d. Magnitude of the residue of esfenvalerate in winter wheat raw agricultural commodity. southern France and Spain 1998. European Agricultural Services, Study EA980121. Dr Specht and Partner, project SUM-9805. Report LLR-0281, Sumitomo Chemical Co. Ltd. Unpublished.

Grolleau, G. 2000a. Magnitude of the residue of esfenvalerate in cotton raw agricultural commodity. Greece and Spain 1999. European Agricultural Services. Study EA990129. Dr Specht and Partner, project SUM-9914. Report LLR-0295, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Grolleau, G. 2000b. Magnitude of the residue of esfenvalerate in field and greenhouse tomato raw agricultural commodity. Italy and Spain 1999. Study EA90128. European Agricultural Services. Study EAS/99-101, Defitraces. Report LLR-0302, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Grolleau, G. 2000c. Magnitude of the residue of esfenvalerate in rapeseed raw agricultural commodity. Southern France and Italy 1999. European Agricultural Services. Study EA990130. Dr Specht & Partner, Project SUM-9915. Report LLR-0296, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Grolleau, G. 2000d. Magnitude of the residue of esfenvalerate in winter wheat raw agricultural commodity. Spain 1999. European Agricultural Services. Study EA990137. Dr Specht and Partner, project SUM-9913. Report LLR-0294, Sumitomo Chemical Co. Ltd Unpublished.

Isobe, N., Kaneko, H., Yanagita, S., Saito, K., Ohe, A., Yoshitake, A. and Miyamoto, J., 1985. Comparative metabolism of esfenvalerate and fenvalerate in rats and mice. 2. 28-Days dietary administration in mice. Report LLM-50-0008, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Itoh, K., Kodaka, R., Kumada, K, Nambu, K. and Kato, T. 1995. Aerobic soil metabolism of esfenvalerate and fenvalerate in European soils. Study SOI9300A, report LLM-50-0039. Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Jackson, C. and Roberts, T.R. 1976. The leaching behaviour of WL 43775 in laboratory soil columns. Report WKGR.0130.76. Project WK 3/I/AC 304. Shell Research Limited. Document AM-61, ref -0071. Sumitomo Chemical Co, Ltd, Japan. Unpublished.

Kaneko, H., Isobe, N., Shiba, K., Saito, K., Yanagita, S., Kitamura, N., Ohe, A., Yoshitake, A. and Miyamoto, J. 1985. Comparative metabolism of esfenvalerate and fenvalerate in rats and mice. 1. Single or 10 consecutive oral administration. Report LLM-50-0007, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Katagi, T., Mikami, N., Matsuda, T. and Miyamoto, J. 1985a. Photodegradation of fenvalerate and esfenvalerate on soils. Document LLM-50, ref -0005. Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Katagi, T., Mikami, N., Matsuda, T. and Miyamoto, J. 1985b. Photoisomerization of esfenvalerate in solid and liquid phases. Document LLM-50, ref -0004. Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Katagi, T., Mikami, N., Matsuda, T. and Miyamoto, J. 1985c. Hydrolysis of fenvalerate and esfenvalerate in buffered aqueous solutions. Document LLM-50, ref -0006. Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Lee, P.W. 1979a. Aerobic and anaerobic soil metabolism of ^{14}C-chlorophenyl-SD 43775 (fenvalerate). Shell Development Company. Report TIR-22-106-79. Report AM-91-0113, Sumitomo Chemical Co, Ltd, Japan. Unpublished.

Lee, P.W. 1979b. Twelve months aerobic soil metabolism of ^{14}C-chlorophenyl SD 43775 (fenvalerate). Shell Development Company, report TIR-22-122-79. Report AM-91-0123, Sumitomo Chemical Co, Ltd, Japan. Unpublished.

Lee, P.W. 1985. Application of ^{14}C-SD 43775 formulation. Study RIR-22-001-85. Report AM-0211, Sumitomo Chemical Co. Ltd, Japan. Shell Development Co. Unpublished.

Lee, P.W. 1989a. Chemical nature, magnitude and isomeric composition of fenvalerate residues in ruminants (dairy cow) milk, meat and meat by-products via dietary exposure. Report AMR-1501-89. E. I. du Pont de Nemours and Company, Inc., USA.. Report AM-91-0182, Sumitomo Chemical Co. Ltd, Japan. Unpublished

Lee. P.W. 1989b. Chemical nature and magnitude of fenvalerate residues in poultry, egg, meat, and meat by-products via dietary exposure. Report AMR-1461-89. E. I. du Pont de Nemours and Company, Inc., USA. Report AM-91-0181, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Lee, P.W. and Behmke, F.D. 1990. Magnitude of fenvalerate residues in dried foliage, vines, and hay of legume vegetables after Pydrin® and Asana® insecticide applications. Report AMR-1668-90. E.I.du Pont de Nemours and Company, USA. Includes trials RIR-24-110-80, RIR-24-123-83, RIR-24-133-80, RIR-24-191-83, RIR-24-202-84, RIR-24-211-79, RIR-24-218-79, RIR-24-227-84, RIR-24-246-83, RIR-24-257-76, RIR-24-258-76, RIR-24-258-79, RIR-24-261-79, RIR-24-275-79, RIR-24-300-79, RIR-24-320-79, RIR-24-324-81, RIR-24-333-79, RIR-24-383-79, RIR-24-384-78, RIR-24-389-79, RIR-24-391-79. Report LLR-0313, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Lee, P.W., Stearns, S.M. and Powell, W.R. 1982. A 30- and 120-day rotation crop study using ^{14}C-SD 43775 following a single soil treatment as a dosage rate of 2 lb ai/acre. Report RIR-22-004-82, Shell Development Company. Report AM-21-0152, Sumitomo Chemical Co, Ltd, Japan. Unpublished.

Lee, P.W., Stearns, S.M. and Powell, W.R. 1985a. Comparative aerobic soil metabolism of SD 43775 (Racemic) and SD 47443 (A-alpha.). Study MO-RIR-22-011-85. Shell Development Company. Report LLM-51-0029. Sumitomo Chemical Co, Ltd, Japan. Unpublished.

Lee, P.W., Stearns, S.M. and Powell, W.R. 1985b. Quantitative characterization of SD 43775 derived residues in the chicken liver tissues. Report RIR-22-004-85, Shell Development Company. Unpublished.

Lewis, C.J. 1995. (^{14}C)-Esfenvalerate: biodegradation in natural water-sediment systems at 10°C. Report 333/14-1015, Corning Hazleton (Europe). Report LLM-0040, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Maestracci, M. 1997a. Esfenvalerate formulation EXP60804A (EC). Trial France 1995. Residues in winter wheat (grain and straw). Philagro France. Report No. R&D/CRLD/AN/fb/961630. Report LLR-0254, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Maestracci, M. 1997b. .Esfenvalerate formulation EXP60804A (EC). Trial France 1995. Residues in winter wheat–decline study. Philagro France. Report R&D/CRLD/AN/kd/9616357. Report LLR-0255, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Maestracci, M. 1997c. Esfenvalerate formulation EXP60804A (EC). Trial France 1996. Residues in winter wheat (grain, straw). Philagro France. Report R&D/CRLD/AN/kd/9715022. Study 96-684. Report LLR-0262, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Maestracci, M. 1997d. Esfenvalerate formulation EXP60804A (EC). Trial France 1996. Residues in winter wheat–decline study. Philagro France. Report R&D/CRLD/AN/vg/9715035. Report LLR-0263, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Maestracci, M., 1997e. Validation of the assay method relative to the residues of esfenvalerate in winter wheat (gran, ear, straw), pea (pod and grain), dried pea (grain and straw), winter barley (grain, ear, straw), peach and cabbage samples (Method Code LLA-60-0023). Adme Bioanalysis Study code RPA/ESF/95102K. Report LLA-0080, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Mikami, N., Matsuda, T. and Miyamoto, J. 1985. Metabolic fate of fenvalerate and esfenvalerate in cabbages. Document LLM-20, ref -0002. Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Mirbach, M.J. and Huber, H.P. 1991a. Determination of the residues of esfenvalerate in soil (FRG-0068) (field and analytical phase reports). Project 275297, RCC Umweltchemie AG, Switzerland. Report LLR-11-0111, Sumitomo Chemical Co, Ltd, Japan. Unpublished.

Mirbach, M.J. and Huber, H.P. 1991b. Determination of the residues of esfenvalerate in soil (FRG-0079) (field and analytical phase reports). Project 275365, RCC Umweltchemie AG, Switzerland. Report LLR-11-0110, Sumitomo Chemical Co, Ltd, Japan. Unpublished.

Mirbach, M.J. and Huber, H.P. 1991c. Determination of the residues of esfenvalerate in soil (FRG-0067) (field and analytical phase reports). Project 275286, RCC Umweltchemie AG, Switzerland. Report LLR-11-0163, Sumitomo Chemical Co, Ltd, Japan. Unpublished.

Naidu, M.V. 1990. Magnitude of fenvalerate residues in tomatoes after Pydrin® and Asana® insecticide application. Report AMR-1788-90, includes trials RIR-24-204-79, RIR-24-280-79, RIR-24-141-85, RIR-24-113-85, RIR-24-149-85, RIR-24-166-85. E.I.du Pont de Nemours and Company, USA Unpublished.

Nambu, K. and Yoshimura, J. 1988. Degradation of esfenvalerate in soils under upland conditions at 15°C. Study SOI87001. Report LLM-80-0030, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Ohkawa, H., Nambu, K. and Mikami, N. 1979. Metabolic fate of fenvalerate (Sumicidin®) in bean plants and cabbage. Document AM-90, ref -0097. Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Ohm, M.B. 2001. Adsorption / desorption of [^{14}C]-esfenvalerate at a single concentration in six soils. Report DuPont-3438. E. I. Du Pont de Nemours and Company. Report LLM-0046, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Potter, J.C. 1982. Characterization and identification of significant residues of SD 43775 in fat, egg yolks, and liver from laying hens following oral administration of SD 43775 (p-chlorophenyl label) or SD 43775 (phenoxyohenyl label) in capsules for 5 days. Report RIR-22-007-82, Shell Development Company. Unpublished.

Roberts, T.R. 1977. The metabolism of the pyrethroid insecticide WL 43775 (Belmark, fenvalerate) in lettuce and soil under outdoor conditions. Report BLGR.0028.77, Shell Research Limited. Report AM-71-0053, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Sakata, S., Mikami, N., Matsuda, T. and Miyamoto, J. 1985. Degradation of esfenvalerate and its isomers in soil. Report LLM-30-0032, Sumitomo Chemical Co., Ltd, Japan. Unpublished.

Schneiders, G.E. 1989. Field soil dissipation studies with Asana® (esfenvalerate 0.66 lb/gal EC) and PYDRIN® (fenvalerate 2.4 lb/gal EC) insecticides. Dupont report AMR-1555-89. Includes MO-RIR-24-109-86, MO-RIR-24-210-85, MO-RIR-24-229-85, TIR-24-135-79, TIR-24-140-79. Report LLR-91-0090, Sumitomo Chemical Co, Ltd, Japan. Unpublished.

Schneiders, G.E. and Orescan, D.B. 1995. Freezer storage stability of esfenvalerate in various matrices. report AMR 1912-90. E.I. Du Pont de Nemours and Company, USA. Unpublished.

Skelsey, J.J. and Barber, G.F. 1983. Residue levels of MO 70616 and SD 43775 in soybeans receiving four applications of MO 70616 or SD 43775, a California study. Report AMR-1907-90. E.I.duPont de Nemours and Company, USA. Sumitomo Chemical Co., Ltd, Japan, Report LLR-41-0012. Unpublished.

Spittler, T., Argauer, R.J., Lisk, D.J., Mumma, R.O., Winnett, G. and Ferro, D.N. 1984. Gas chromatographic determination of fenvalerate insecticide residues in processed tomato products and by-products. *J. Assoc. Off. Anal. Chem.*, 67, 824-826.

Standen, M.E. 1978. The metabolism of the pyrethroid insecticide Sumicidin (WL 43775) in apple fruit and foliage under outdoor conditions. Document AM-71, ref -0052. Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Takahashi, N., and Ohshima, M. 1988. Degradation of esfenvalerate in sediment-water systems. Sumitomo Chemical Co., Ltd, Japan, Report LLM-80-0024. Unpublished.

Weeren, R.D. and Pelz, S. 1998a. Validation of DFG Method S 19 with modified extraction for the determination of residues of esfenvalerate in field samples of cereals and other dry plant material (dry peas). Report SUM-9802V, Dr Specht & Partner, Germany. Report LLA-0091, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Weeren, R.D. and Pelz, S. 1998b. Validation of DFG Method S 19 with modified extraction for the determination of residues of esfenvalerate in field samples of starch containing plant material (potatoes). Report SUM-9803V, Dr Specht & Partner, Germany. Report LLA-0090, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Weeren, R.D. and Pelz, S. 1998c. Validation of DFG Method S 19 with DFG Cleanup Method 5 for the determination of residues of esfenvalerate in field samples of fat containing plant material (rapeseed). Report SUM-9801V, Dr Specht & Partner, Germany. Report LLA-0089, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Weeren, R.D. and Pelz, S. 2000. Validation of DFG Method S 19 with modified extraction for the determination of residues of esfenvalerate in samples of fruits with high acid content (apple). Report SUM-0005V, Dr Specht & Partner, Germany. Report LLA-0097, Sumitomo Chemical Co. Ltd, Japan. Unpublished.

Cross-index of study and/or report numbers with references.

3438: Ohm, 2001
4054: Farrell, 1995
61567.2: Barber *et al.*, 1981
61567.32: Barber and Jelatis, 1979
275286: Mirbach and Huber, 1991c
275297: Mirbach and Huber, 1991a
275365: Mirbach and Huber, 1991b
16/312-1016: Benwell, and Burden, 1997
303FX-532-1601: Gillis *et al.*, 1989
333/14-1015: Lewis, 1995
333/15-1012a: Burden, 1998
333/15-1012b: Burden, 1997
333/40-1012: Burden, 1995
333/57-1012: Benwell, 1996b
333/63: Grolleau, 1997
9582/94097/83: Farrell, 1995
96-684: Maestracci, 1997c
AM-01-0122: Fan and Lee, 1980
AM-0211: Lee, 1985
AM-21-0152: Lee *et al.*, 1982
AM-61, ref-0071: Jackson and Roberts, 1976
AM-71, ref -0052: Standen, 1978
AM-71-0053: Roberts, 1977
AM-90, ref -0097: Ohkawa *et al.* 1979
AM-91-0113: Lee, 1979a
AM-91-0114: Ehmann, 1979b
AM-91-0115: Ehmann,1979a
AM-91-0116: Ehmann, 1979d
AM-91-0117: Ehmann, 1979c
AM-91-0123: Lee, 1979b
AM-91-0181: Lee, 1989b
AM-91-0182: Lee, 1989a
AMR-1461-89: Lee, 1989b
AMR-1501-89: Barber *et al.* 1981
AMR-1501-89: Boyer and Lee, 1981
AMR-1501-89. E. I: Lee, 1989a
AMR-1555-89: Schneiders, 1989
AMR-1668-90: Lee and Behmke,1990
AMR-1788-90: Naidu, 1990
AMR-1881-90: Behmke and Lee, 1991
AMR-1907-90: Skelsey and Barber, 1983
AMR 1912-90: Schneiders and Orescan, 1995
AR-81-0423: Gilham and Woodbridge, 1978
AR-91-0406: Barber and Jelatis, 1979
AS/2066/SL/1: Farrell, 1995
AS/2066/SL/2: Farrell, 1995
AS/2066/SL/3: Farrell, 1995
AS/2066/SL/4: Farrell, 1995
BEER.87.006: Bosio, 1986
BLGR.0028.77, Limited: Roberts, 1977
BLGR.0076.78: Gilham and Woodbridge, 1978
CLE 333/107-04R): Croucher 1998b
CLE 333/107-D2140: Croucher 1998b
CLE 333/108-04R): Croucher, 1998c
CLE 333/108-D2140: Croucher, 1998c
CLE 333/116-04R): Croucher, 1998a
CLE 333/116-D2140: Croucher, 1998a
CLE 333/40-01R): Burden, 1995
CMK 57: Gillis *et al.*, 1989
Code RPA/ESF/95102K: Maestracci, 1997e
EA90128: Grolleau, 2000b
EA950177: Grolleau, 1997
EA980121: Grolleau, 1999d
EA980125: Grolleau, 1999b
EA980126: Grolleau, 1999a
EA980127: Grolleau, 1999c
EA990129: Grolleau, 2000a
EA990130: Grolleau, 2000c
EA990137: Grolleau, 2000d
EAS/98-058: Grolleau, 1998
EAS/98-059: Grolleau, 1999b
EAS/99-101: Grolleau, 2000b
LLA-0071: Benwell, 1996a. 333/56-1012
LLA-0072: Benwell, 1996b
LLA-0080: Maestracci, 1997e
LLA-0082: Croucher, 1998a
LLA-0083: Croucher 1998b
LLA-0084: Croucher, 1998c
LLA-0089: Weeren and Pelz, 1998c
LLA-0090: Weeren and Pelz, 1998b
LLA-0091: Weeren and Pelz, 1998a
LLA-0093: Grolleau, 1998
LLA-0097: Weeren and Pelz, 2000
LLA-51-0068: Burden, 1995
LLA-60-0023): Maestracci, 1997e

LLM-0040: Lewis, 1995
LLM-0046: Ohm, 2001
LLM-20, ref -0002: Mikami *et al*., 1985
LLM-30-0032: Sakata *et al*. 1985
LLM-50, ref -0004: Katagi *et al*. 1985b
LLM-50, ref -0005: Katagi *et al*. 1985a
LLM-50, ref -0006: Katagi *et al*., 1985c
LLM-50-0007: Kaneko *et al*., 1985
LLM-50-0008: Isobe *et al*., 1985
LLM-50-0039: Itoh *et al*., 1995
LLM-51-0029: Lee *et al*., 1985a
LLM-80-0024: Takahashi and Ohshima, 1988
LLM-80-0030: Nambu and Yoshimura, 1988
LLR-0188: Bosio, 1986
LLR-0244: Grolleau, 1997
LLR-0254: Maestracci,1997a
LLR-0255: Maestracci, 1997b
LLR-0262: Maestracci, 1997c
LLR-0263: Maestracci, 1997d
LLR-0270: Behmke and Lee, 1991
LLR-0271: Burden, 1998
LLR-0272: Burden, 1997b
LLR-0274: Benwell, and Burden, 1997
LLR-0281: Grolleau, 1999d
LLR-0285: Grolleau, 1999b
LLR-0286: Grolleau, 1999a
LLR-0287: Grolleau, 1999c
LLR-0294: Grolleau, 2000d
LLR-0295: Grolleau, 2000a
LLR-0296: Grolleau, 2000c
LLR-0302: Grolleau, 2000b
LLR-0313: Lee and Behmke,1990
LLR-11-0110: Mirbach and Huber, 1991b
LLR-11-0111: Mirbach and Huber, 1991a
LLR-11-0163: Mirbach and Huber, 1991c
LLR-41-0012: Skelsey and Barber, 1983
LLR-51-0241: Farrell, 1995
LLR-81-0034: Edmunds *et al*., 1988
LLR-91-0090: Schneiders, 1989
LLR-91-0100: Gillis *et al*., 1989
MO-RIR-22-011-85: Lee *et al*., 1985a
MO-RIR-24-109-86: Schneiders, 1989
MO-RIR-24-210-85: Schneiders, 1989
MO-RIR-24-229-85: Schneiders, 1989
R&D/CRLD/AN/fb/961630: Maestracci,1997a
R&D/CRLD/AN/kd/9616357: Maestracci, 1997b
R&D/CRLD/AN/kd/9715022: Maestracci, 1997c
R&D/CRLD/AN/vg/9715035: Maestracci, 1997d
RIR-22-001-85: Lee, 1985
RIR-22-003-81: Boyer and Lee, 1981
RIR-22-004-82: Lee *et al*., 1982
RIR-22-004-85: Lee *et al*. ,1985b
RIR-22-007-82: Potter, 1982
RIR-24-110-80: Lee and Behmke,1990
RIR-24-113-85: Naidu, 1990
RIR-24-123-83: Lee and Behmke,1990
RIR-24-133-80: Lee and Behmke,1990
RIR-24-141-85: Naidu, 1990
RIR-24-149-85: Naidu, 1990
RIR-24-166-85: Naidu, 1990
RIR-24-191-83: Lee and Behmke,1990
RIR-24-198-84: Behmke and Lee, 1991
RIR-24-202-84: Lee and Behmke,1990
RIR-24-204-79: Naidu, 1990.
RIR-24-208-84: Behmke and Lee, 1991
RIR-24-211-79: Lee and Behmke,1990
RIR-24-218-79: Lee and Behmke,1990
RIR-24-227-84: Lee and Behmke,1990
RIR-24-236-79: Behmke and Lee, 1991
RIR-24-246-83: Lee and Behmke,1990
RIR-24-249-75B: Behmke and Lee, 1991
RIR-24-257-76: Lee and Behmke,1990
RIR-24-258-76: Lee and Behmke,1990
RIR-24-258-79: Lee and Behmke,1990
RIR-24-261-79: Lee and Behmke,1990
RIR-24-275-79: Lee and Behmke,1990
RIR-24-280-79: Naidu, 1990.
RIR-24-300-79: Lee and Behmke,1990
RIR-24-320-79: Lee and Behmke,1990
RIR-24-324-81: Lee and Behmke,1990
RIR-24-333-79: Lee and Behmke,1990
RIR-24-383-79: Lee and Behmke,1990
RIR-24-384-78: Lee and Behmke,1990
RIR-24-389-79: Lee and Behmke,1990
RIR-24-391-79: Lee and Behmke,1990
RIR-24-617-80-B: Barber *et al*. 1981
SBL/8/78/I/AC 505: Gilham and Woodbridge, 1978
SMO 295: Edmunds *et al*., 1988
SOI87001: Nambu and Yoshimura, 1988
SOI9300A: Itoh *et al*., 1995
SUM-0005V: Weeren and Pelz, 2000
SUM-9801V: Weeren and Pelz, 1998c
SUM-9802V: Weeren and Pelz, 1998a
SUM-9803V: Weeren and Pelz, 1998b
SUM-9805: Grolleau, 1999d
SUM-9806: Grolleau, 1999a
SUM-9807: Grolleau, 1999c
SUM-9913: Grolleau, 2000d
SUM-9914: Grolleau, 2000a
SUM-9915: Grolleau, 2000c
TIR-22-004-80: Fan and Lee, 1980
TIR-22-104-79: Ehmann, 1979b
TIR-22-106-79: Lee, 1979a
TIR-22-111-79: Ehmann, 1979d
TIR-22-112-79: Ehmann, 1979c
TIR-22-116-79: Ehmann,1979a
TIR-22-122-79: Lee, 1979b
TIR-24-135-79: Schneiders, 1989
TIR-24-140-79: Schneiders, 1989
TIR-24-413-78: Barber and Jelatis, 1979
WK 3/I/AC 304: Jackson and Roberts, 1976
WKGR.0130.76: Jackson and Roberts, 1976