FENPROPIMORPH (188)

EXPLANATION

Fenpropimorph is a fungicide whose major use is to control diseases in cereals. It was reviewed for the first time at the 1994 JMPR which considered toxicological aspects. An ADI of 0-0.003 mg/kg bw was allocated. Owing to the late receipt of data, the review of residue and analytical aspects was postponed until 1995.

IDENTITY

ISO common name: fenpropimorph

Chemical name

IUPAC:(±)-cis-4-[3-(4-tert-butylphenyl)-2-methylpropyl]-2,6-dimethylmorpholine

CA: *cis*-4-[3-[4-(1,1-dimethylethyl)phenyl]-2-methylpropyl]-2,6-

dimethylmorpholine

CAS No.: 67564-91-4

Synonyms: 108406; BAS 421 F (BASF); Ro 14-3169 (Maag); CA 101031 (Ciba)

Structural formula:

$$\begin{array}{c} CH_3 \\ CH_3 \\ CH_3 \end{array} \\ \begin{array}{c} CH_2 \\ CH_2 \\ CH_3 \end{array} \\ \begin{array}{c} CH_3 \\ CH_2 \\ CH_3 \end{array} \\ \begin{array}{c} CCH_3 \\ CH_3 \\ C$$

Physical and chemical properties

Pure active ingredient

Vapour pressure: 3.5 x 10⁻³ Pa at 20°C (BASF, 1988a)

Melting point: colourless liquid Boiling point: 120°C (at 0.067 mbar)

Octanol/water

partition coefficient: $\log P_{OW} = 4.1 (22^{\circ}C, pH 7)$

2.6 (22°C, pH 5) (Keller, 1986)

Solubility: 7.3 mg/l (20°C, pH 4.4) (BASF 1988b)

5.1 mg/l (unbuffered) (Rüdel 1988) 4.3 mg/l (20°C, pH 7) (BASF 1988c) 3.5 mg/l (20°C, pH 9-11) (BASF 1988c)

Toluene>1 kg/kg at 20°C Ethanol >1 kg/kg at 20°C Acetone>1 kg/kg at 20°C

Specific gravity: not stated (see density of technical material below)

Hydrolysis (half-life): >64 days 50°C (pH 5, 7, 9) (BASF 1983)

>64 days 70°C (pH 5) 15 days 70°C (pH 7, 9)

No hydrolysis half-life could be determined when fenpropimorph was incubated at pH 3, 5, 7 and 9 in the dark at 25°C for 32 days (Rüdel, 1988). Although it was concluded that fenpropimorph is hydrolytically stable, losses of 20-35% of radioactive material were observed, especially at pH 9 after 20 days incubation. The loss was attributed largely to either increased volatility or adsorption to the glass walls of the container or stopper owing to reduced solubility at higher pH.

Although no hydrolysis products were detected by TLC, fenpropimorph hydrochloride was detected at pH 3 and a compound with similar TLC characteristics to 4-[3-(4-*tert*-butylphenyl)-2-methyl-1-oxopropyl]-*cis*-2,6-dimethylmorpholine (BF 421-13: see Table 1) was found at pH 9.

Photolysis: Stable in artificial sunlight (≥290 nm) up to 30 days at pH 5 and

25°C (Herrchen, 1988a)

Technical material

Purity Minimum 93% fenpropimorph (reported to JMPR, undocumented).

Melting range: Liquid

Boiling point: >250°C at 1 atm (discoloration at 190-210°C) (Ciba, 1992)

Stability: No information provided. Density: 0.933 g/cm³ (Ciba, 1992)

Formulations

Fenpropimorph is a fungicide which has found its most important use for the control of powdery mildew and rust in cereals, and of barley leaf blotch. It is also reported to be active as a post-harvest dip against *Penicillium* fungi, *Alternaria citri*, *Diplodia spp.*, and *Phomopsis citri* in citrus, although it is not recommended for this use (Lafuente *et al.*, 1986). It is formulated into 49 fungicidal products, alone as an emulsifiable concentrate (750 g ai/l) or with one or two additional active ingredients as an EC, soluble concentrate (SC) or wettable powder (WP). Other fungicides in mixed formulations include propiconazole, fenpropidin, fenbuconazole, chlorothalonil, carbendazim, mancozeb, tridemorph, prochloraz, epoxiconazole and flusilazole. The concentrations of the active ingredients included in all 49 products were provided to the Meeting. The fenpropimorph concentration in mixed formulations is less than when fenpropimorph is formulated alone (118-563 g/l instead of 750 g/l). The total active ingredients in formulations never exceed 750 g/l. Registered uses are listed in Table 17.

METABOLISM AND ENVIRONMENTAL FATE

Table SUBJECT

- 1. Names and structures of fenpropimorph and related compounds
- 2. Goats material balance

- 3. Goats residue distribution
- 4. Poultry material balance
- 5. Poultry residue distribution (total residue)
- 6. Poultry residue distribution (metabolites)
- 7. Plant and animal metabolites (summary)
- 8. Barley metabolism
- 9. Rotational crops distribution of ¹⁴C
- 10. Spring wheat metabolism (morpholine label)
- 11. Spring wheat metabolism (morpholine and phenyl labels)
- 12. Soil half-lives
- 13. Degradation in water-sediment systems (laboratory)
- 14. Degradation in water-sediment systems (field)

Information was provided on the fate of residues in animals, plants, soil, water, and water-sediment systems. Structures, codes and chemical names of fenpropimorph and its degradation products are shown in Table 1. Oxidation is common to all the degradation routes. The metabolism of fenpropimorph in plants is similar to that in animals to the extent that oxidation is the first step and is followed by degradation of the morpholine ring. There are differences (see especially Table 11) in that fenpropimorph is generally the main residue in plants but is not found in animals except in hen kidneys, whereas BF 421-3, BF 421-4, BF 421-16 and BF 421-17 (and/or their conjugates) are metabolites in animals but have not been reported in plants. The plant metabolites BF 421-2-Me, BF 421-7, BF 421-10, BF 421-13 and BF 421-15 have not been reported in animals, although in plants most of these would be at levels below one quarter of the level of the parent compound. An exception may be BF 421-7 (the hydroxypropylamine) which has been reported at a similar level to the parent compound in wheat straw under some conditions.

Under neutral conditions fenpropimorph is largely stable in water. Under field conditions degradation in soil proceeds by oxidation and opening of the morpholine ring to give BF 421-2 (the acid), BF 421-7, BF 421-8 (the hydroxyethylamine) and BF 421-10 (dimethylmorpholine). In aerobic water/soil systems the degradation is similar except that the morpholine ring is not opened. In addition to these compounds, BF 421-13 (the alkyl ketone) and BF 421-15 (the morpholine-3-one derivative) can be formed by photolysis of fenpropimorph in soil.

The metabolites referred to by codes are identified below (Table 1).

Table 1. Fenpropimorph metabolites, degradation products and related compounds - structures, chemical names and codes.

$$H_{3}C = CH_{3}$$

$$HOH_{2}C = CH_{3}$$

$$HOCC = CH_{3}$$

$$H_{3}COOC = CH_{3}$$

$$H_{3}COOC = CH_{3}$$

$$HOOC = CH_{3}$$

$$H_{3}C = CH_$$

BF 421-1 4-{3-[4-(2-hydroxy-1,1-dimethyl)ethylphenyl]-2-methylpropyl}-cis-2,6-dimethylmorpholine

BF 421-2 2-methyl-2-{4-[2-methyl-3-(*cis*-2,6-dimethylmorpholin-4-yl)propyl]phenyl}propionic acid

BF 421-2-Me methyl 2-methyl-2-{4-[2-methyl-3-(*cis*-2,6-dimethylmorpholin-4-yl)propyl]phenyl}propionate

BF 421-3 2-methyl-2-{4-[2-methyl-3-(*cis-*2-hydroxymethyl-6-methylmorpholin-4-yl]propyl)phenyl}propionic acid

BF 421-4 2-methyl-2-{4-[2-methyl-3-(2-hydroxypropyl)aminopropyl]-phenyl}propionic acid

BF 421-6 [3-(4-*tert*-butylphenyl)-2-methylpropyl]bis(2-hydroxypropyl)amine

BF 421-7 [3-(4-tert-butylphenyl)-2-methylpropyl](2-hydroxypropyl)amine

BF 421-8 [3-(4-*tert*-butylphenyl)-2-methylpropyl](2-hydroxyethyl)amine

BF 421-9 2-methyl-3-(4-tert-butylphenyl)propylamine

BF 421-10 *cis*-2,6-dimethylmorpholine

BF 421-12 2-methyl-2-(4-carboxyphenyl)propionic acid

BF 421-13 4-[3-(4-*tert*-butylphenyl)-2-methyl-1-oxopropyl]-*cis*-2,6-dimethylmorpholine

BF 421-14 4-[3-(4-*tert*-butylphenyl)-2-methylpropyl]-*cis*-2,6-dimethylmorpholine *N*-oxide

BF 421-15 4-[3-(4-*tert*-butylphenyl)-2-methylpropyl]-*cis*-2,6-dimethylmorpholine-3-one

BF 421-16 2-methyl-2-[4-(carboxyphenyl)]propan-1-ol

BF 421-17 2-methyl-2-[4-(2-carboxypropyl)phenyl)]propionic acid

Animal metabolism

The fate of residues was studied in rats, goats and poultry. Metabolism is similar but not identical, consisting mainly in progressive oxidation of *tert*-butyl, methylpropyl and morpholine-methyl groups. Further metabolism of the morpholine ring is demonstrated by the expiration of significant amounts of ¹⁴CO₂ by rats. The expiration of ¹⁴CO₂ was not measured in goats or hens.

Rats. Because material balance and metabolism studies in rats (von der Mühll and Gätzi, 1979; van Dijk and Vogel, 1989; Pryde et al., 1979, 1980) with both morpholine- and phenyl-labelled fenpropimorph were described in the 1994 toxicological evaluation they are not considered in detail here. In summary, 64-72% of the ¹⁴C from the morpholine label was excreted within 96 hours after its administration (mostly in faeces and urine) and as much as 12% in expired air (showing breakdown of the morpholine ring). Tissue residues were highest in the liver and somewhat lower in fat. Elimination in the faeces and urine was even higher from the phenyl-labelled compound. No residues of fenpropimorph were reported. Qualitatively the following metabolites were reported (see especially van Dijk and Vogel, 1989).

Metabolite	*		Urine	Faeces	Bile	Liver	kidney		Plasma
BF 421-2		+	+	+	+	+		+	
BF 421-3		+	+	-	+	+		+	
BF 421-3	conj.	+	+	+	-	-		-	
BF 421-4*	*		+	-	-	-	-		-
BF 421-16	j		+	+	-	-	+		-
BF 421-17	7		+	-	-	-	+		-
*	See Table	1							
**	Described	as "U3-	-1, probabl	y identical	to BF	421-4". Ter	ntatively co	onfirmed b	y TLC

Goats. Three studies were available, the first on the distribution of radioactive residues in milk (and

other fluids) and tissues (Hawkins et al., 1980a), the second a supplementary study (on the same animals) of the excretion in faeces and urine (Hawkins et al., 1980b) and the third on the distribution, degradation and excretion of radioactive residues (Ritter, 1989a).

In the first study (Hawkins et al., 1980a) two lactating goats of approximately 56 kg were administered daily by gelatin capsule for ten consecutive days 1.6 mg of [14C]fenpropimorph hydrochloride labelled in the morpholine ring (1.5 mg free base = 0.03 mg/kg bw). Although the feed consumption was not recorded, the dosage would approximate 0.6 ppm in the feed if all of the daily offering of 1.5 kg hay and 1 kg concentrate was consumed. Treated goats and an untreated control were milked twice daily, immediately before dosing and 6 hours after dosing, then 24 hours after the last dose and just before slaughter. Blood samples were taken at several hourly intervals and 24 hours after the last dose, and samples of bile were retained. Twenty four hours after the last dose samples of liver, kidneys, heart, brain, muscle and fat were also taken. Analyses were by liquid scintillation counting. Approximately 1.5 to 2% of the daily dose was eliminated in the milk with mean residues (combined milk samples before dosing and 6 hours after dosing) reaching a plateau at approximately 7 to 8 ng/ml (ppb) (9 ng/ml maximum) after 4 to 6 days. A similar profile and concentration were observed in plasma and a similar profile but lower levels in whole blood. Maximum and average residues (fenpropimorph equivalents) in the fluids and tissues from the two goats 24 hours after the last dose were as shown below

<u>Tissue</u>	ng/g or n	g/ml
	Max. N	<u>Iean</u>
Liver	103	96
Kidneys	29.4	28.5
Brain	12.4	12
Heart	8.3	8.2
Fat (back)	7.6	7
Fat (omental)	12	9.6
Muscle (leg)	3.4	3.1
Muscle (loin)	4	3.9
Bile	1618	1264
Plasma	7	6.9
Milk	8.4	8.4
Blood	5.4	5.3

In the supplementary study with the same goats (Hawkins et al., 1980b) the authors reported daily urinary excretion of radioactivity increasing from about 13% of the administered dose after 24

hours to 32 and 39% after 6 or 7 days. Elimination in the faeces increased from 16 to 24% of the daily dose during the first 24 hours to 70-73% after 7 days. Over the 10-day dosing period the average cumulative elimination of administered radioactivity for the two goats was 26.7% in the urine and 57.5% in the faeces, giving a total of 84.2%. Residues in the urine remained constant after about 7 days, but continued to increase in the faeces through the tenth day.

In the third and most comprehensive study (Ritter, 1989a) performed according to GLP guidelines, two goats were dosed orally by stomach tube for 5 consecutive days with either [14 C]phenyl-labelled fenpropimorph (2335 ppm daily in the feed = 55.5 mg/kg bw/day) or with [14 C]morpholine-labelled fenpropimorph (labelled adjacent to the ring oxygen) (1421 ppm daily in feed = 54.6 mg/kg bw/day). The test material was stable over the period of the study.

Blood samples were taken daily one hour after dosing and after the last dose. The goats were milked twice daily, 1 and 8 hours after dosing (1 and 4 hours after the last dose) and urine and faeces samples were taken at regular intervals. The animals were slaughtered 5 hours after the last treatment and samples of heart, liver, kidneys, spleen, teat, brain, muscle, fat and bile were taken for analysis, mainly by scintillation counting and silica gel TLC (8 different solvent systems with 15 reference compounds). TLC of methylated metabolites and hydrolysed conjugates was also used in comparing metabolites and standards. The material balance of the radioactivity is shown in Table 2.

Table 2. Material balance of radioactivity from lactating goats administered [¹⁴C]phenyl- or [¹⁴C]morpholine-labelled fenpropimorph daily for 5 days (Ritter, 1989a).

Sample	% of administ	ered radioactivity
	Goat 1, phenyl label	Goat 2, morpholine label
Urine	14.49	21.34
Faeces	20.43	28.95
Cage wash	1.02	0.44
Milk	0.06	0.32
Tissues and organs ¹	2.65	3.13
Bile	1.80	1.08
Total ²	40.45	55.26

The radioactivity in the whole blood and plasma increased steadily from 6.8 and 9.2 mg/kg fenpropimorph equivalents for the phenyl label and 2.5 and 3.1 mg/kg for the morpholine label 1 hour after the first dose to 54 and 93 and 29 and 40 mg/kg respectively 5 hours after the last dose. BF 421-2 was the most prominent metabolite in protein-free plasma with both labels (52-58% of the recovered radioactivity) followed by BF 421-3 (0.6-3%) with other unidentified metabolites \leq 7%.

A significant level of radioactivity in the bile indicated biliary excretion. This was confirmed in a 5-day feeding study with the highest residues found in the faeces (20-29% of the total administered radioactivity) with slightly less in the urine (15-21%). The elimination of radioactivity in the faeces or urine was highest from the phenyl label 48 hours after the first administration (after the 2nd treatment), whereas it was at its highest from the morpholine label 96 hours after the first administration (after the 4th treatment).

A relatively low level of radioactivity was found in the milk (0.06% from the phenyl label, 0.3% from the morpholine label) and organs and tissues (about 3% from both labels), although only 40 to 55% of the administered radioactivity was accounted for in the urine, faeces, cage wash, milk, organs, tissues and bile. The rest was assumed to be in the gastrointestinal tract.

Residues in milk (ì g parent equivalents/ml) from the phenyl label increased from 0.7 and 2.9 ì g/ml 1 and 8 hours after the first administration to 6.9 and 9.7 ì g/ml and 1 and 8 h after the fourth, decreasing to 1.9 and 0.9 ì g/ml 1 and 4 hours after the fifth. For the morpholine label residues were similar at 0.7 and 3.9 ì g/ml 1 and 8 hours after the first dose and 11 and 19 ì g/ml after the fourth but continued to increase to 16 and 23 ì g/ml 1 and 4 hours after the fifth. From the phenyl label 76.2 and 23.8% of the radioactivity was in the whey and protein fractions of the milk respectively and from the morpholine label 60 and 4.7%. A further 6.6% of the radioactivity from the morpholine label was in a hexane fraction and 28.7% was unextracted.

The distribution of identified and unidentified metabolites in the urine, faeces, milk and tissues is shown in Table 3.

Table 3. Distribution of identified and unidentified metabolites in excreta, milk and tissues of lactating goats dosed daily for 5 days with phenyl- or morpholine-labelled [¹⁴C]fenpropimorph¹ (Ritter, 1989a).

Sample	% Extract- able ¹⁴ C	BF 421-	BF 421-1 ¹		BF 421-2 ¹		BF 421-3 ¹		Unidentified metabolites			
	able C	% of recov'd	ì g/g ²	% of recov'd	ì g/g ²	% of recov'd	ì g/g ²	No.	% of recov'd	ì g/g ²		
				Phenyl	label							
Urine	-	-	-	-	-	20.1	193	9	2.1-17	20-160		
Faeces ³	83-86	38	924	-	-	9.4	229	5	2.1-13	51-310		
Faeces ⁴	83-86	12	1.4	12	1.4	-	-	6	3.8-18	0.5-2.2		
Muscle ⁵	96	-	-	68	6	-	-	5	3.4-6.5	0.3-0.6		
Liver	92	-	-	33 6.7 ⁶ 31 ⁶	47 9.4 ⁶ 43 ⁶	-	-	2	9-12	13-17		
Kidneys	99	-	-	11	25	6.3	15	4	11-34	25-78		
Fat ⁵	96	25	1.1	38	1.6	-	-	5	1.9-17	0.1-0.8		

¹ Assumes fat and muscle are 12 and 40% respectively of body weight

² The author assumed that most of the remaining radioactivity was in the gastrointestinal tract.

Sample	% Extract- able ¹⁴ C	BF 421-	BF 421-1 ¹		BF 421-2 ¹		BF 421-3 ¹		Unidentified metabolites		
	doic C	% of recov'd	ì g/g ²	% of recov'd	ì g/g ²	% of recov'd	ì g/g ²	No.	% of recov'd	ì g/g ²	
Milk whey ⁷	-	-	-	-	-	-	-	6	5-22	0.2-0.6	
				Morpholi	ine label						
Urine	-	-	-	-	-	43	136	7	1.7-20	5.4-61	
Faeces 3	86-88	7.6	139	53	973	12	214	3	3.4-5.8	63-107	
Faeces ⁴	86-88	4.7	153	60	1949	9.7	314	2	4.6-8.4	147-273	
Muscle ⁵	86	-	-	64	4	-	-	3	4.2-12	0.3-0.8	
Liver	84	-	-	40 32 ⁶	50 40 ⁶	-	-	2	4.2-7.6	5.2-9.5	
Kidneys	90	-	-	27	14	11	5.8	5	2.2-28	1.2-15	
Fat ⁵	64	27	5	3	0.6	0.9	0.2	6	0.9-22	0.2-4	
Milk whey ⁷	-	-	-	17	6.5	7.2	2.8	3	6-24	2.3-9.2	

¹ See Table 1

A high percentage of the radioactivity (\geq 83%) was extractable from most substrates. No parent compound was identified in any sample with either label and all samples contained from 2-9 unidentified metabolites. BF 421-3 was the only identified metabolite in urine from either label. BF 421-1, BF 421-2 and BF 421-3 were identified in the faeces from both labels. BF 421-2 was the only identified metabolite in the muscle and liver. In the liver two conjugated metabolites of BF 421-2 were also observed. The identified metabolites (predominant compound underlined) were BF 421-2 and BF 421-3 in the kidneys; BF 421-1, BF 421-2 and BF 421-3 in the fat from the morpholine label, but only BF 421-1 and BF 421-2 from the phenyl label; BF 421-2 and BF 421-3 in milk whey from the morpholine label with none from the phenyl label, although 6 unidentified metabolites were observed.

<u>Poultry</u>. In a study in accordance with GLP, two groups of ten white leghorn hybrid laying hens were dosed once daily for five days by syringe intubation with about 6.8 mg/hen (3.9 mg/kg bw) of [¹⁴C]fenpropimorph labelled in the phenyl or morpholine ring. Based on average feed consumption the doses were equivalent to 51.5 ppm in the diet for phenyl label and 39.3 ppm for the morpholine label. These levels are respectively 206 and 157 times the dietary intake calculated to result from maximum residues of 0.5 mg/kg in grain consumed as 50% of the diet (Ritter, 1989b).

Eggs were collected twice daily (before and 8 hours after dosing) and before slaughter, and separated into yolks and whites which were pooled by group and sampling interval. The faeces were collected and pooled in a similar manner, although sampled only once daily. At slaughter (5 hours after the last dose for treated hens) blood samples were taken and analysed within 24 hours, and samples of kidneys, liver, muscle, gizzard, heart, brain, fat, skin (+ subcutaneous fat), ovaries and spleen were taken for analysis. Samples were analysed by scintillation counting for material balance and distribution, and characterized by TLC against reference standards before and after acidic hydrolysis. The material balance of administered radioactivity is shown in Table 4 and its distribution among tissues in Table 5.

Table 4. Material balance of radioactivity from laying hens administered [\frac{14}{C}]phenyl- or [\frac{14}{C}]morpholine-labelled fenpropimorph daily for 5 days (Ritter, 1989b).

² Fenpropimorph equivalents; faeces fresh weight

³ Sampled from 1st to 4th dose

⁴ Sampled 5 hours after 5th dose

⁵ Assumes fat and muscle are 12% and 40% respectively of body weight.

⁶ Conjugated. Confirmed by hydrolysis, methylation, and TLC co-chromatography.

⁷ Sampled 1 and 4 hours after last dose

Sample	% of administered radioactivity					
	Group 1, phenyl label	Group 2, morpholine label				
Excreta	83.11	79.1 ²				
Cage wash	3.6	2.7				
Eggs	0.2	0.4				
Tissues, organs, blood ³	3.1	3.6				
Total	90	85.8				

Table 4 illustrates the predominant and rapid elimination in the excreta, as in goats and rats, and the low proportion of the administered dose retained in the tissues.

Table 5. Distribution of residual radioactivity in tissues, eggs and blood of laying hens dosed daily for 5 days with phenyl- or morpholine-labelled [14C]fenpropimorph (Ritter, 1989b).

Sample		Phenyl label		N	Morpholine Label	
	% extractable ¹	% of total dose	mg/kg ²	% extractable ¹	% of total dose	mg/kg ²
Egg white ³		0.008	0.17^{4}		0.02	0.42^{5}
Egg yolk ³		0.02	0.83^{6}		0.09	34
Liver	87	0.38	2.8	76	0.55	3.9
Kidneys	87	0.11	2.8	70	0.10	2.4
Muscle (chest)		0.58	0.28		0.52	0.24
Muscle (Leg)	97	0.91	0.42	67	0.73	0.34
Gizzard	88	0.05	0.40	69	0.12	1.1
Heart		0.02	0.93		0.01	0.71
Brain		< 0.01	0.16		< 0.01	0.31
Fat (stomach)	99	0.95	1.4	97	0.70	1.1
Skin (+ subcut. fat)	96	0.01	0.97	92	0.01	0.68
Ovaries		0.29	1.3		0.94	4.2
Spleen		0.01	0.82		0.01	1.5
Blood		0.6	1.4		0.47	1.1
Plasma			1.9		-1	1.3

 $^{^1}$ 93% of recovered radioactivity extracted 2 88.1% of recovered radioactivity extracted 3 Assumes muscle, fat and blood are 40, 12 and 8% of body weight respectively.

Table 5 shows the high percentage of the radioactivity extractable from the tissues and blood. The highest residues from the phenyl label (about 3 mg/kg) were found in the liver and kidneys, with about 1-1.5 mg/kg in the fat, ovaries, skin, heart and blood and lower levels in muscle and other tissues. The highest residues from the morpholine label were found in the ovaries, liver, egg yolk and kidneys in that order with lower levels in the fat, gizzard, spleen, blood and heart and little in muscle. Residues in the whites of eggs from both labels reached a plateau approximately 48 hours after the first dose, whereas in the yolks they continued to increase throughout the collection period.

Although 3-10 unidentified metabolites were detected and measured as fenpropimorph equivalents in the organosoluble or water-soluble fractions of the excreta, eggs, muscle, fat, skin and gizzard, compounds were identified and determined only in the plasma, liver and kidneys, with the results shown in Table 6.

Table 6. Distribution of identified and unidentified compounds in plasma, liver and kidneys of laying hens dosed daily for 5 days with phenyl- or morpholine-labelled [14C]fenpropimorph (Ritter, 1989b).

Sample	fenpropin	orph	BF 42	1-1 ¹	BF 421	l-2 ¹	BF 421-3 ¹		Unidentified metabolites		
	% of recov'd	mg/ kg	% of recov'd ¹⁴ C	mg/k g ²	% of recov'd	mg/k g ²	% of recov'd	mg/k g ²	No.	% of recov'd	mg/kg ²
Phenyl label											
Liver (org. sol.)									10	2.2-13	0.06-0.36
Liver (H ₂ O sol.)									8	0.8-6.3	0.02-0.17
Plasma					13	0.24			5	5.5-30	0.1-0.56
Kidneys (org. sol)	11	0.31			3.5	0.1	1.9	0.05	6	1.5-4.7	0.05-0.13
Kidneys (H ₂ O sol)									5	1.2-21	0.03-0.6
				Morp	holine lab	el					
Liver (org. sol.)					3.9	0.15			5	3.8-21	0.15-0.83
Liver (H ₂ O sol.)									8	1.1-5.9	0.04-0.2
Plasma			2.3	0.03	12	0.16			5	3.2-25	0.04-0.33
Kidneys (H ₂ O sol)									3	9.9-15	0.23-0.36

¹ Extractable ¹⁴C as % of ¹⁴C in sample

² As fenpropimorph equivalents

³ 5 hours after last dose

⁴ 79.7% of radioactivity in protein-free fraction and 20.3% in protein

⁵ 21.6% of radioactivity in protein-free fraction and 78.45 in protein

⁶ 62% of radioactivity in protein-free fraction and 37.2% in protein

Table 6 shows the (phenyl-labelled) parent compound to be found only in the kidneys (analyses of the organosoluble fraction were not provided for the morphine label). BF 421-1 was found in the plasma (morpholine label) but not in the liver or kidneys. Metabolite BF 421-2 was found in the kidneys and liver (morpholine label) and BF 421-3 only in the kidneys.

The metabolites found in rats, goats, hens and wheat plants are listed in Table 11 at the end of the section describing metabolism in plants.

The studies show a similar metabolic fate in rats, goats and hens. Residues of the parent compound were detected only in hen kidneys. BF 421-1 was detected in goats and hens, but not in rats. No conjugates were detected in hens, but conjugates of BF 421-2 were found in goats and of BF 421-3 in rats. Metabolites BF 421-16 and -17 were detected only in rats. On the basis of reported studies the proposed metabolic fate of fenpropimorph in animals is as shown in Figure 1.

Plant metabolism

The metabolism of fenpropimorph in plants is similar to that in animals to the extent that oxidation is followed by degradation of the morpholine ring, but certain metabolites are found in animals and not in plants and vice versa.

The fate of fenpropimorph in plants was investigated in four studies: one on barley (Pryde and Etterli, 1979), one crop rotation study with spinach, sugar beet and wheat (Pryde and Etterli, 1980) and two metabolism studies on wheat (Huber, 1979a; Rüdel, 1990).

In the first study Pryde and Etterli (1979) grew summer barley under greenhouse conditions and treated leaves by syringe at the five-leaf tillering stage with [\$^{14}\$C]fenpropimorph (free base) labelled at the benzylic carbon between the rings. The specific activity was 152 i Ci/mg and application was at a rate equivalent to 0.9 kg free amine/ha (5000 l/ha). The goal was to determine the dissipation and translocation of total radioactivity and of fenpropimorph from topical applications. Treated and untreated leaves from the same plant as well as untreated leaves from controls were sampled at intervals. Surface residues were removed for analysis by stirring the leaves with a mixture of HC1 and methanol and the washed leaves were then macerated with the same mixture and filtered.

Radioactivity was measured and characterized by combustion, scintillation counting, TLC on alumina and silica, with radio scanning, and HPLC. Whole plants were examined by autoradiography. Results are shown in Table 7.

Table 7. Distribution of total radioactivity and fenpropimorph in or on barley leaves from plants treated with [¹⁴C]fenpropimorph (Pryde and Etterli, 1979).

Days after treatment		¹⁴ C residues									
		Treated leaves									
	Surfac										
	Extracted radioactivity, % of applied	fenpropimorph, % of extracted radioactivity ¹	Extracted radioactivity, % of applied	fenpropimorph, % of extracted radioactivity ²	Extracted radioactivity, % of applied						
0	62-65	93	3.1-4.6	87	0.02-0.04						

¹ See Table 1

² fenpropimorph equivalents

Days after treatment			¹⁴ C residues		
		Treate	ed leaves		Other green plant parts (untreated)
	Surfac	e wash	Washed		
	Extracted radioactivity, % of applied	fenpropimorph, % of extracted radioactivity ¹	Extracted radioactivity, % of applied	fenpropimorph, % of extracted radioactivity ²	Extracted radioactivity, % of applied
1	45-58	77	5.3-14	69	0.02-0.03
5	49	71	11-12	17	0.06-0.07
8	33-35	70	12	9	0.05-0.12
15	26	57	17-22	6	0.07-0.3
20	9.2-16	63	27-29	7	0.2-1.1

 $^{^{1}}$ Average of TLC and HPLC results 2 TLC

Figure 1. Metabolic pathways of fenpropimorph in animals.

detected in goats and hens, not in rats

Table 7 shows that up to 65% of the applied radioactivity is on the leaf surface on the day of application and 93% of the surface residue is unchanged fenpropimorph. Even after 20 days up to 16% of the residue is on the surface, of which 63% is fenpropimorph. The proportion of the applied radioactivity remaining in the leaf after washing increased from about 5% on the day of application to almost 30% after 20 days and in the same period the proportion of fenpropimorph decreased from 87% to 7% of the extracted ¹⁴C. Although always at low levels, the increase in the radioactivity in untreated plant parts during the test period indicates a limited translocation from the leaves to the roots and uptake into other plant parts. Autoradiography of the plants was reported to confirm that. The decrease in the mean overall recovery of the radioactivity from 67.2% on the day of application to 44.3% on day 20 was attributed to the loss of volatile material.

In the second study (Pryde and Etterli, 1980) spinach, sugar beets and wheat were grown in soil used in a previous wheat metabolism study in which a 1 m² plot of sandy loam soil with plants of Probus winter wheat had been spray-treated with [¹⁴C]fenpropimorph hydrochloride labelled at the same carbon as before at a rate equivalent to 0.72 kg ai free base/ha (2450 l/ha). The top 5-10 cm depth of soil was used to grow the rotational crops. The plants were grown under greenhouse conditions and both plants and soil were collected at various intervals for measurement of radioactivity by combustion analysis. No grain was produced under the greenhouse conditions, so only the mature wheat plants were analysed. The results are shown in Table 8.

Table 8. Distribution of radioactivity in soil and rotational crops after treatment of soil with [14C]fenpropimorph (Pryde, Etterli, 1980).

Days after treatment of plot	Age of crops (days)	Average to	otal residue, mg/k	g fenpropimorph ed	quivalents
-		Soil	Spinach ¹	Sugar beet ²	Wheat ³
7		0.42			
102	10		0.010 0.13		0.08
103	11	0.1			
105	13		0.014	0.15	0.11
109	17		0.013	0.11	0.09
112	20		0.011	0.11	0.12
116	24			0.11	0.10
119	27		0.010	0.11	0.09
123	31	-	0.011		-
126	34		0.011	< 0.002	0.09
130	38		0.009		
144	52		0.006		
151	59		0.005		
263	171	0.02 ⁴ 0.07 ⁵		0.01 green plant 0.004 mature beet	<0.02

¹ Fresh wt. basis, 90.6% water

² Fresh wt. green plant, 19.1% water, or mature beet (without leaves or roots), 20.6 water

³ Fresh wt., 19.6% water

⁴ Soil from sugar beet pot

⁵ Soil from wheat pot

In the third study (Huber, 1979a) Kolibri variety summer wheat was planted (30 seeds/pot) in 36 pots (18 × 18 × 18 cm, 15 cm sandy loam soil) and spray-treated 55 days after seeding with fenpropimorph hydrochloride labelled at the morpholine ring carbons adjacent to the oxygen (specific activity 9.2 mCi/mMol, 60083 dpm/i g) at a rate equivalent to 1.5 l/ha (1.3 kg ai/ha). Plants were grown in an open greenhouse. Green plants were sampled 21 and 43 days after treatment, and straw and seed at harvest 84 days after treatment. Samples were deep-frozen, then homogenized and stored at -25°C until analysis, for which they were extracted with methanol. Liquid/liquid extraction, liquid chromatography, derivatization, GLC, radio-GLC, radio-HPLC, scintillation counting, and GC-MS were used to characterize and identify the radioactive residues. The results are shown in Table 9.

Table 9. Radioactive residues in spring wheat treated with morpholine-labelled fenpropimorph (Huber, 1979a).

Compound or fraction		Green p	olant		Strav	v 84 days		Grain (84 d	lave)
		1 days		3 days		0/ .CTDD	Grain (84 days) "Bound" radioactivity, % of TRR		
	mg/kg ¹	% of TRR ²	mg/kg	% of TRR	mg/kg	% of TRR			
Fenpropimorph	3	47	1.7	38	2.6	22			
BF 421-1 (M=319)	0.5	7	0.3	7	0.9	8			
BF 421-7 (M=263) ³	0.7	12	0.5	10	3.1	26			
BF 421-10 (M=115)	0.2	3	0.1	3	0.3	3			
M=103 ³	0.1	2	0.08	2	0.1	1	Starch	Protein	Polysac- charides
Total of identified compounds	4.5	71	2.7	59	6.9	60	49	16	5
TRR in crop part	6.5	100	4.3	100	11.9	100	0.43 mg/kg ¹		
MeOH-extractable		87		79		62		9%	

¹ As fenpropimorph equivalents

Table 9 shows that both the levels and the extractability of the residues decrease with time. Only about 5-9% of the radioactivity in harvested seeds (0.43 mg/kg fenpropimorph equivalents) was extractable and the very low residues were not identified. Almost half of the unextracted residue was incorporated into the starch, 16% was in a protein precipitate and 5% in the polysaccharide fraction. Of the 40% unextractable radioactivity in wheat straw 21% was found in the lignin fraction.

Fenpropimorph was the predominant residue (38-47% of the TRR) and BF 421-7 the main metabolite (10-12% of the TRR) found in green plants. BF 421-7 was the main residue in straw (26% of the TRR), slightly higher than the parent compound at 22% of the TRR. Lower levels of BF 421-1 and 421-10 were also measured in all plant parts except the grain.

² Total radioactive residue

³ BF 421-10 further degraded

In the fourth study (Rüdel, 1990) spring wheat plants (Ralle variety, 10-12 plants/pot) were treated under field conditions at the tillering stage with both phenyl- and morpholine-labelled fenpropimorph hydrochloride at 0.75 kg ai/ha and harvested according to the following schedule.

	I	Days
	Phenyl label	Morpholine label
forage	0	7
	21	28
straw	56	57
grain	56	57
roots	56	57

Samples were extracted with methanol, transferred to water, acidified and extracted successively with hexane, chloroform, acidic ethyl acetate and basic ethyl acetate. The organic extracts were cleaned up by solid-phase extraction or preparative TLC and analysed by 2-dimensional TLC. Major fractions were analysed by GLC with ion-trap detection. The total radioactivity was measured by scintillation counting. Straw samples were also subjected to enzymatic and hydrolytic treatment to release any conjugated compounds. Results are shown in Table 10.

Table 10. Radioactive residues in spring wheat treated with morpholine- and phenyl-labelled [14C]fenpropimorph (Rüdel, 1990).

Compound or fraction	Fora	age	Stra	W	Grai	n	Forage	
	% of TRR	mg/kg ¹	% of TRR	mg/kg ¹	% of TRR	mg/kg ¹	% of TRR	mg/kg
Phenyl 1	Phenyl lal (0 days)							
Fenpropimorph	26.7	2.4	24	2.6				
BF 421-2	0.08	0.01	7	0.8				
BF 421-2-Me &/or BE 421-15	0.3	0.02	0.02	0.00				
BF 421-7 &/or BF 421-1	2.4	0.23	0.4	0.05				
BF 421-13	1.5	0.13	0.2	0.02				
Unidentified metabolites (no.)	0.03-6 (30)	0.00-0.5	0.04-5.6 (37)	0.00-0.6				
Total extractable	61	5.4	61	6.8	12.2	0.01	97	64
TRR	100	8.8	100	10.9	100	0.11	100	66
Morpholin	e label (28 d	lays forage	e, 57 days stra	aw/grain)			Morpholine la days)	abel (7
Fenpropimorph	16.4	2	20	4.8				
BF 421-1	0.01	0.00						
BF 421-1 &/or BF 421-7			0.3	0.07				
BF 421-2	0.6	0.08						
BF 421-2-Me &/or BF 421-15								
BF 421-7	0.65	0.08						·
BF 421-13	0.2	0.03						
Unidentified metabolites (no.)	0.02-4.4 (33)	0.00-0.6	0.01-12 (43)	0.00-3				
Total extractable	52	6.4	56	14	12.9	0.05	94	38
TRR	100	12.2	100	24	100	0.37	100	40

TRR = total radioactive residue

¹ Fenpropimorph equivalents

At the first sampling (0 or 7 days) almost all of the radioactivity (≥94%) was extractable. As in the Huber greenhouse study, the parent compound was the predominant residue in the forage at harvest. It was also the main residue in the straw, whereas in the Huber study BF 421-7 slightly exceeded fenpropimorph at harvest. Fenpropimorph was confirmed in forage by GC-MS, and metabolites by two-dimensional TLC. BF 421-2, BF 421-7 and/or 421-1, and BF 421-13 were prominent metabolites in the forage. Numerous unidentified metabolites were characterized, no one of which exceeded 0.6 mg/kg fenpropimorph equivalents.

In straw the pattern was generally similar but BF 421-2 was higher than in forage and other metabolites were lower, with fewer identified with the morpholine label. Again numerous unidentified metabolites were found, of which only one (morpholine-labelled) at 3 mg/kg parent equivalents exceeded 0.8 mg/kg. Most of the radioactivity in straw from the phenyl label was associated with the lignin fraction. Hydrolysis of straw before extraction yielded BF 421-1, identified by GC-MS.

Metabolite BF 421-2-Me was reported in forage and straw from the phenyl label, but not from the morpholine label. BF 421-13 was found in the straw from the phenyl but not the morpholine label.

The metabolites found in this study but not reported by Huber were BF 421-2, BF 421-2-Me (phenyl label only), BF 421-13 and BF 421-15 (phenyl label only). There was some difficulty in distinguishing quantitatively between BF 421-1 and BF 421-7 and between BF 421-2-Me and BF 421-15.

Again, only a low level of the radioactivity was found in the grain (\leq 0.4 mg/kg parent equivalents) and of that less than 13% was extractable. None of the residues in the grain were identified, but little of the radioactivity was found in the protein or polysaccharide fractions (\leq 8% and \leq 1.1% of the TRR respectively). The starch contained about 32% of the TRR, but <3% was found in osazone derivatives.

In combination the cereal plant metabolism studies show oxidation of the *tert*-butyl group to the alcohol and then to the acid, with some methylation of the acid. The morpholine ring is oxidized before its cleavage to form the hydroxypropylamine. A minor route involves oxidation of the *N*-carbon of the methylpropyl group before cleavage of the morpholine ring. The results of these studies are the basis for the proposed metabolic pathway in plants shown in Figure 2.

The components of the residues found in animals and plants are listed in Table 11.

TC 11 11	r · 1	metabolites found	1	1 1 1 ,
I anie I I	Hennronimornh	metabolites tolin	a in anima	ic and niante
тапистт.		metabonics roun	u iii aiiiiiia	is and mains.

Compound (see Table 1)	Sites in which compound found (F = forage; S = straw)								
	Rats	Goats	Hens	Wheat plants					
Fenpropimorph			kidneys	F, S					
BF 421-1		faeces, fat	blood	F, S					
BF 421-2	urine, faeces, bile, liver, kidneys, plasma	faeces, muscles, liver, kidneys, fat, milk	blood, kidneys, liver	F, S					
BF 421-2 conj.		liver							
BF 421-Me				F, S					
BF 421-3	urine, faeces, liver, kidneys, plasma	urine, faeces, kidneys, fat	kidneys						
BF 421-3 conj.	urine, faeces, bile								
BF 421-4	urine								
BF 421-7				F, S					

Compound (see Table 1)	Sites in which compound found (F = forage; S = straw)							
	Rats	Goats	Hens	Wheat plants				
BF 421-10				F, S				
BF 421-13				F, S				
BF 421-15				F, S				
BF 421-16	urine, faeces, kidneys							
BF 421-17	urine, kidneys							

Environmental fate in soil

Six studies were provided on the fate of fenpropimorph in soil: photolysis in soil (Herrchen, 1988b), behaviour in soil under field conditions (Hesse and Tilting, 1991, 1992), mineralization (Huber, 1980), aerobic degradation (Huber, 1979b), and dissipation, degradation and leaching under field conditions (von der Mühll *et al.*, 1980).

In the photolysis study (Herrchen, 1988b) a loamy sand (pH 5.8, 63 cm² surface area, 0.4 cm depth, 40% natural moisture) was treated with 10 mg [¹⁴C]phenyl-labelled fenpropimorph/kg soil and irradiated for 30 days with artificial sunlight (xenon lamp filtered to exclude UV below 290 nm) at 1.5 times the intensity of natural sunlight. Residues were determined and characterized by scintillation counting, TLC and HPLC, and the identity of the parent compound was confirmed by MS. After 30 days of irradiation the parent compound had decreased to 50% of the TRR (6.1 mg/kg).

The oxidation products BF 421-13 and BF 421-15 were identified at maximum levels of 9.3% of the TRR (1.1 mg/kg) and 5.3% of the TRR (1.4 mg/kg) respectively after 30 days. Two unidentified compounds were each <6% of the TRR. No volatile products were detected. The half-life of the parent compound was estimated to be about 30 days. The increase in unextractable radioactivity (from 1.6% to 11.9% over 30 days) was taken to be an indication of the sorption of photodegradation products by the soil.

In the field studies (Hesse and Tilting, 1991, 1992; von der Mühll, 1980) several fallow soils at various locations in Germany and one in Switzerland were treated with fenpropimorph at 0.75 or 1.1 kg ai/ha (in c. 400 l water/ha), periodically sampled over 14-15 months down to a 1 m depth, and analysed by GLC with a nitrogen FID for fenpropimorph and BF 421-2. Half-lives varied, according to soil type and location, from 10 to 90 days (Table 12).

Figure 2. Metabolic pathways of fenpropimorph in plants.

Table 12. Half-lives of fenpropimorph in different soils under field conditions.

Soil type (Location)	Half-life (days)	Application		Reference
		Rate g/ha	Month	
Loam (Niedersachsen, Germany)	40	750	May	Hesse and Tilting, 1991

Soil type (Location)	Half-life (days)	Application		Reference
		Rate g/ha	Month	
Loamy clay (Baden-Württemberg, Germany)	90	750	April	Hesse and Tilting, 1991
Sandy loam (Oberding, Germany)	10	1125	May	Hesse and Tilting, 1992
Sandy loam (Brockhausen, Germany)	29	1125	April	Hesse and Tilting, 1992
Sandy loam (Dielsdorf, Switzerland)	43	1130	June	von der Mühll et al., 1980
Loamy sand (Birkenheide, Germany)	approx. 15-30	1125	May	Hesse and Tilting, 1992

In the 1991 and 1992 trials by Hesse and Tilting the parent compound was detected in the top 25 cm of soil at one or more sites, decreasing from maximum levels in the top 10 cm up to 0.6 mg/kg at day 0 to \leq 0.1 mg/kg after approximately 363 days. BF 421-2 was detected mainly in the top 10 cm where it did not exceed 0.05 mg/kg (day 0). It did not exceed 0.02 mg/kg in the 10-25 cm layers. Neither compound was detected (<0.01 mg/kg) below 25 cm.

Huber (1979b) applied [¹⁴C]fenpropimorph labelled in the morpholine ring to sandy loam soil in beakers at 6 mg/kg. The soil was kept in the laboratory under aerobic conditions in the dark at 22°C and sampled at intervals for 12 months. After methanol extraction and liquid-liquid partition with chloroform, samples were analysed by scintillation counting, LC on alumina, TLC, HPLC, and radio-GLC and/or GLC-MS. The total radioactivity expressed as fenpropimorph decreased from 6.1 mg/kg initially to 3.5 mg/kg after 12 months.

The proportion extracted by methanol decreased from 87% of the TRR to 17.3% in the 12 months. 80% of the methanol extract could be partitioned into chloroform, and the radioactivity due to the parent compound was shown by TLC to decrease from 96% of that in the chloroform extract to 78% during the 12 months, while degradation products increased. The major compounds in the chloroform extract (months 2 and 3 combined), separated by LC and confirmed by MS, were 62.4% parent, 10% BF 421-8, and 6.7% BF 421-10. The half-life was estimated to be about 20 days.

Huber (1980) treated loamy sand and sandy loam soils with morpholine-labelled fenpropimorph at approximately 5.6 mg/kg soil and measured the $^{14}\text{CO}_2$ evolved. Within 45 days approximately 9 and 18% of the fenpropimorph was mineralized respectively. Only trace levels (0.2% of the applied radioactivity) of volatile compounds were detected.

In the study by von der Mühll $et\ al.$ (1980) PVC tubes 5.8 cm i.d. \times 15 cm long were driven into sandy loam soil in a wheat field and treated at the equivalent of 1.13 kg/ha with fenpropimorph (free amine) labelled at the benzylic carbon of the methylpropyl group. Dissipation, breakdown and leaching were studied during 12 weeks by Soxhlet-extracting soils from different depths with methanol and analysing them by scintillation counting, HPLC, and TLC with radioscanning. Identities were confirmed by MS.

Radioactivity in the top 5 cm decreased from about 77% of that applied after one week to about 30% after 12 weeks. The proportions in the 5-10 and 10-15 cm levels were 2.3 and 0.6% respectively after the first week and \leq 1.5% and 0.3% for the rest of the study. HPLC analysis of the top 5 cm showed the parent compound and BF 421-7 (confirmed by MS) to be the predominant residues (see below).

Time	% of a	pplied radioactiv	<u>ity</u>
(weeks)	fenpropimorph	BF 421-7 Ur	identified
1	65	7.8	3.7
2	63	7.5	2.3
4	45	9.8	5.1
8	25	8.1	2.5

12 22 6.1 2.2

In a separate experiment soil was treated at 5.5 mg/kg with [¹⁴C]fenpropimorph and incubated at 22°C for 10 weeks. ¹⁴CO₂ was determined for 32 weeks as a measure of the degradation of the parent compound. By the end of the test period a cumulative total of 50% of the applied radioactivity had been evolved as CO₂. No other volatile radioactivity was reported. This was judged to indicate rapid degradation of the parent compound.

The residues identified in soil (Huber, 1979b; von der Mühll *et al.*, 1980) thus include fenpropimorph, BF 421-2, BF 421-7, BF 421-8 and BF 421-10, suggesting the pathways shown in Figure 3.

Figure 3. Degradation pathways of fenpropimorph in soil.

Environmental fate in water/sediment systems

The hydrolysis of fenpropimorph by water (BASF 1982; Rüdel, 1988) is mentioned above in the section on physical and chemical properties. In those studies no hydrolysis products were observed at a neutral pH, although a compound with the TLC characteristics of BF 421-13 was detected at pH 3 and 9. Two laboratory studies on the fate of fenpropimorph in soil/water systems were provided, with the label on the morpholine ring in one (Hamm, 1982) and on the phenyl ring in the other (Ritter, 1990).

In the Hamm study 10-g quantities of a loamy sand at pH 6 (15.7% water = 40% capacity) were treated uniformly in separate conical flasks with 103 i g of [14 C]fenpropimorph labelled in the morpholine ring. The flasks were shaken in the dark at 20°C with 90 ml of a sterile nutrient solution at pH 7 and sampled for analysis at intervals up to 56 days.

After successive extractions with methanol and acid and basic organic solvents, clean-up on an XAD-4 resin column and hydrolysis of some fractions with esterases, samples were examined by scintillation counting, radio-TLC, radio-HPLC, derivatization, and GC-MS (after methylation or acetylation). A control without soil showed no significant degradation by hydrolysis. The production of $^{14}\text{CO}_2$ at a level of 11% of the applied radioactivity indicated considerable microbial degradation of the morpholine ring. The half-life was estimated to be about 36 days. The results are shown in Table 13.

Table 13. Degradation of	[¹⁴ C]fennro	nimornh in	a soil/water	suspension	(Hamm	1982)
Table 13. Degradation of	լ Շյլեոթյե	ւրուստւրո ու	a son/water	suspension	(11anını,	1704).

Days	¹⁴ C, % of applied								
	Fenpropimorph	npropimorph Conjugated BF 421-2 ¹ BF 421-10 ¹⁴ CO ₂		Unextracted	Total				
0	95.4	0.0	0.0	0.0	4.3	99.7			
14	72.7	11.9	1.4	1.2	4.3	91.5			
28	40.4	29.4	2.5	3.3	6.8	82.4			
56	39.8	28.7	4.2	10.4	6.9	90.0			

¹ Hydrolysis of conjugate in methanolic HCl produced methyl ester of BF 421-2. The conjugate was not identified but was probably an ester since its TLC Rf was changed by esterase but not by papainase

In the study by Ritter (following OECD/Swiss GLP) phenyl-labelled fenpropimorph was applied in the laboratory at 0.1 mg/l to natural Rhine river water and pond water (c. pH 7.3), each containing 10% of sediment, and incubated for 84 days at 22°C in the dark. Water and soil samples were taken periodically (0, 3, 7, 14, 21, 28, 42 and 84 days) for analysis and CO₂ as well as other volatiles were trapped. Water was extracted with neutral or acidic dichloromethane and soil with methanol for scintillation counting and TLC.

During the 84 days fenpropimorph decreased from 84.2% of the applied radioactivity to 18.2% in the Rhine water system and from 87.1 to 6.8% in the pond water system. Two products were identified and several unidentified compounds were characterized. Representative results in Table 14 show oxidation of the *tert*-butyl group to the alcohol BF 421-1 and acid BF 421-2.

Table 14. ¹⁴C residues in Rhine and pond water and sediments after incubation with [¹⁴C]fenpropimorph for 84 days (Ritter, 1990).

Compound or fraction			¹⁴ C, %	of applied		
	Rhine (day 3)	Pond (day 3)	Rhine (day 21)	Pond (day 21)	Rhine (day 84)	Pond (day 84)

	water	soil										
Fenpropimorph	9.6	66	2.8	77	0.8	31	1.0	20	1.2	17		5.8
BF 421-1	2.6					1.2		1.3		0.4		0.2
BF 421-2	5.5				14.2	3.1	7.7	6.9	13.4	1.7	12.3	0.8
Unknown 1					6.6		15.1	1.7				
Unknown 2					4.2		2.3					
Polar					4.5	0.6	4.0	0.7	19.1	1.3	3.6	0.7
Total	18	66	2.8	77	30	36	30.1	30	34	20.4	15.9	7.5

There was 29 and 40% mineralization after 84 days in the Rhine and pond water systems respectively, in which the half-lives of fenpropimorph were 13 and 5 days. Degradation was related to biomass.

The degradation pathways shown in Figure 4 were proposed on the basis of the results of the two studies.

Figure 4. Metabolic Pathways of fenpropimorph in water/soil systems.

$$\begin{array}{c} CH_3 \\ H_3C \\ CH_2 \\ CH_3 \\ CH_4 \\ CH_5 \\ CH$$

BF 421-2

METHODS OF RESIDUE ANALYSIS

Analytical methods

Several analytical methods are available for the determination of fenpropimorph in plant materials (mainly cereals and citrus), soil and water, with determination by GLC (usually) or HPLC. A method was also provided for the determination of BF 421-2 in soil. The limits of determination and recoveries for the substrates studied are shown in Table 15.

<u>Plants</u>. Seven methods have been described for the determination of fenpropimorph in plants, five by GLC and two by HPLC. Some are also applicable to soil and water. BASF method 137 (Beutel, 1979) is available for green plants, cereal grains and straws, soil and water. Green plants and straw are chopped with dry ice before extraction with methanol/water; grains are extracted directly. The extracts are concentrated under acidic conditions, partitioned with chloroform, and the concentrated chloroform extract eluted through a cation exchange column with methanol/HCl. The required eluate fraction is partitioned into chloroform, cleaned up on an alumina column, and concentrated. Determination is by GLC with an alkali flame detector. No chromatograms or details of the validations were provided to allow independent confirmation of the limit of determination. The "limit of detection" was reported as 0.05 mg/kg.

BASF method 241 (Beutel and Tilting, 1987) is applicable to cereals, rape, Brassicas, citrus and grapes. Chopped or cut high-moisture samples are added to distilled water and extracted with chloroform for two hours in a Bleidner apparatus. The concentrated extract is cleaned up on a silica gel column (eluted with 2% methanol in methylene chloride), and the eluate concentrated and determined on a packed or capillary column with nitrogen FID detection. Smaller samples are used for low-moisture samples (e.g. straw), and rape and grain samples are macerated in distilled water before the chloroform extraction. Sample chromatograms and other documentation support a limit of determination of 0.05 mg/kg. Detection may be possible at lower levels. Sample chromatograms showed controls to be generally substantially below 0.05 mg/kg.

BASF method 156 (Hänni and von der Mühll, 1981) is also applicable to cereals, soil and water. It is similar to method 241 except that all samples apart from water are macerated before extraction, the extraction is with methylene chloride rather than chloroform, and the alumina column clean-up step is omitted. Controls were <0.05 mg/kg for all samples. The "limit of detection" was reported as 0.05 mg/kg. Limits of determination of 0.05-0.1 mg/kg (0.01 mg/kg for water) appear to be feasible on the basis of the sample chromatograms provided. Detection may be possible at a lower level.

An HPLC method (with confirmation by GLC) has been applied to the determination of fenpropimorph in barley, oats, rye and wheat grains (Zoonen and Harten, 1988). Samples are blended with 5:75 v/v acetonitrile/light petroleum, and to the extract concentrated and partitioned with 1:1 acetonitrile/0.1 N HCl. The aqueous phase is adjusted to pH 9 and extracted with hexane for residue determination. Determination is by HPLC with an ODS stationary phase and a mobile phase of 85:15 acetonitrile/water with 0.25% ammonia. A UV detector at 220 nm is used. Confirmatory analyses are by GLC with alkali flame detection. The reported limits of determination are 0.01 mg/kg for HPLC and 0.005 mg/kg for GLC. The data did not allow confirmation of these levels.

Three methods have been published for determining fenpropimorph specifically in citrus, two by GLC and one by HPLC. In one GLC method (Lafuente *et al.*, 1986) citrus samples are chopped, ground and homogenized, and extracted with hexane. The extract is washed with a buffer solution and concentrated, and fenpropimorph is determined by GLC with an NPD. A "limit of detection" of 0.01 mg/kg is reported. On the basis of the limited number of chromatograms available, limits of determination of about 0.1 mg/kg for whole fruit and pulp and 0.2-0.5 mg/kg for peel seem possible.

The other (multi-residue) GLC method is also capable of determining imazalil, prochloraz, propiconazole and thiabendazole (Lafuente and Tadeo, 1987). Whole fruit or peel is homogenized and, after adjustment of the pH, extracted with 90:10 v/v hexane/ethyl acetate. The extract is filtered, concentrated and, without any clean-up, analysed (for fenpropimorph) by GLC with a nitrogen-phosphorus detector. The method is recommended for screening purposes. Although fortification levels were relatively high and few analyses were reported, the "limit of detection" is stated to be 0.1 mg/kg. A limit of determination of 0.2 to 0.5 mg/kg appears to be possible on the basis of the single chromatogram of a control and a sample fortified at 1.8 mg/kg.

The same extraction procedure, without clean-up, is used in the HPLC method (Tadeo and Lafuente, 1987). Determination is by HPLC on an RP-18 stationary phase, with a mobile phase of 87:13 methanol/water plus 0.25% ammonia and UV detection at 215 nm. The limit of "detection" was reported to be 0.03 mg/kg (twice the noise level). The few chromatograms suggest that 0.05 to 0.1 mg/kg may be a feasible limit of determination.

<u>Soil</u>. BASF methods 137 (Beutel, 1979) and 156 (Hänni and von der Mühll, 1981), described above, are applicable to soil. In method 137 dry soil samples are continuously extracted with methanol for 5 hours in a Thiele-Pape or equivalent extractor, and the extract is acidified and concentrated before partition with chloroform. The analysis is completed as described for plant materials. The reported limit of "detection" of 0.05 mg/kg could not be confirmed with the information provided. The limit of determination for method 156 could be estimated to be 0.05 to 0.1 mg/kg for soil. Controls were <0.05 mg/kg.

The simultaneous determination of fenpropimorph and its acid metabolite BF 421-2 in soil has been described by Dieckmann, *et al.* (1993). The method involves extraction with 2:1 acetone/water, liquid/liquid partition with dichlormethane, clean-up by gel permeation chromatography and determination by GLC with an NPD or GC-MS. The acid metabolite is methylated with diazomethane before analysis. Detection limits for GC-MS (electron-impact) were reported as 0.005 mg/kg for fenpropimorph and 0.01 mg/kg for the metabolite. These could not be confirmed nor could limits of determination be estimated from the information available. A single sample chromatogram obtained by GLC with an NPD from soil spiked with 0.4 mg/kg fenpropimorph and 0.04 mg/kg of the acid metabolite as its methyl ester suggests that limits of determination of 0.05-0.1 mg/kg for fenpropimorph and 0.01 to 0.02 mg/kg for the metabolite may be feasible with NP detection.

BASF method 298 (Tilting, 1989) is for BF 421-2 only. Soil samples are extracted with aqueous pH 9 buffer solution, partitioned with dichloromethane and methylated with diazomethane before determination by GLC with NP detection. The buffer does not extract fenpropimorph. The limit of determination is reported as 0.01 mg/kg and sample chromatograms suggest that this level might be achieved, as with the similar method of Dieckmann *et al*.

<u>Water</u>. The general BASF methods 137 and 156 are suitable. Water is acidified before extraction and the analysis is continued as described for plant materials. A limit of detection of 0.05 mg/kg was reported for method 137, but could not be confirmed with the information provided. The limit of determination for method 156 can be conservatively estimated to be 0.01 mg/kg. Controls were <0.01 mg/kg on the evidence of sample chromatograms.

BASF method 271 (Tilting 1987) is specifically for the determination of fenpropimorph in water. Dichloromethane extraction is followed by clean-up on a non-activated silica gel column, concentration and determination by GLC with an NPD, using an internal standard. The reported limit of determination is 0.05 mg/kg. Although sample chromatograms were provided, they were not sufficiently legible for satisfactory confirmation.

Table 15. Analytical methods for fenpropimorph and BF 421-2. All GLC unless shown as HPLC.

Made al maf	C1-	T ::t -f d-t:t:	Fautification	Mana
Method, ref.	Sample	Limit of determination, mg/kg	Fortification level, mg/kg	Mean recovery, %
BASF 137 (Maag 840-MD-01) Beutel, 1979	Green plants Straws Cereal grains Soil Water	0.051	0.05-5	101 90 86 88 93
BASF 241 Beutel and Tilting, 1987	Wheat forage spindles stalks straw grains Rape forage seeds Brussels sprouts Tangerines Oranges Grapes	0.05	0.05	97 84 86 83 82 76 89 104 94 98 98
Dieckmann <i>et al.</i> , 1993 (published)	Soil fenpropimorph BF 421-2	$0.05 - 0.1^2 \\ 0.01 - 0.02^2$	0.01-0.1 ³	99 ⁴ 97 ⁴
BASF 156 (Maag 840-MD-02) Hänni and von der Mühll, 1981	Green plants Cereal grains Straws Soil Water	0.05-0.1 0.05 0.05-0.1 0.05-0.1 0.01	0.1	88 83 94 74 101
Lafuente and Tadeo, 1987 (published)	Citrus whole fruit peel	0.2	0.6 0.9 0.9 2.3	96 94 100 89
Lafuente <i>et al.</i> , 1986 (published)	Citrus whole fruit peel pulp	0.1 0.2-0.5 0.1	1 2 0.05	74 85 74
Tadeo and Lafuente, 1987 (published) HPLC	Citrus whole fruit pulp peel	0.05-0.1	1 2 0.05	74 84 78
BASF 271 Tilting, 1987	Water	0.05 ì g/l	0.05 0.5	88 90
BASF 298 Tilting, 1989	Soil BF 421-2	0.01	0.01	88
Zoonen and Harten, 1988 (submitted for publication)	Cereal grains	0.01 (HPLC) ¹	0.5	95
HPLC with GLC confirmation		0.005 (GLC) ¹		90

Stability of pesticide residues in stored analytical samples

The stability of fenpropimorph in ground wheat grain, straw and green plants, and of both fenpropimorph and BF 421-2 in loamy sand, during frozen storage has been investigated by Tilting (1993). Samples spiked at 1 mg/kg with the unlabelled compound(s) were stored in polyethylene bottles in a walk-in freezer up to two years at -20°C. Samples were removed at various intervals for analysis by GLC with nitrogen-FID detection (0.05 mg/kg limit of determination for plant materials and 0.01 mg/kg for soil). Residues of the acid were derivatized with diazomethane before analysis. The proportions remaining were \geq 87% except in grain, in which they were 76% \pm 21% s.d. The results are shown in Table 16. Those for grain are corrected for the lower and less consistent analytical recoveries.

Table 16. Deep-freeze storage stability of fenpropimorph in wheat and soil and of BF 421-2 in soil after fortification at 1 mg/kg (Tilting, 1993).

Days storage	mg/kg (mean of replicate samples)									
			BF 421-2							
	Green plant	Green plant Straw Grain ¹ Soil								
0	0.94	1.1	0.96	0.96	0.91					
30-32	0.72	1.1	0.70	0.87	0.86					
60-62	0.83	0.95	1.1	0.87	0.70					
118-119	0.91	0.89	0.89	0.80	0.83					
180	1.0	0.93	0.96	0.72	0.90					
242-243	0.71	1.0	0.88	0.79	0.76					
362	0.84	1.0	1.0	0.89	0.78					
735-742	0.96	0.99	0.93	0.93	0.82					

¹ Corrected for analytical recoveries

Residue Definition

In plant metabolism studies fenpropimorph was shown to be by far the predominant residue shortly after application and even at harvest it is typically at least three times the level of any identified metabolite. A possible exception in some circumstances is the metabolite BF 421-7 (the hydroxypropyl amine) which was shown in one study, but not in another, to occur at about the same level as the parent compound. For enforcement purposes fenpropimorph *per se* is therefore the appropriate definition of the residue in plants. If worst-case assumptions are needed for risk assessment purposes, it might be argued from the metabolism studies that total residues of the parent plus identified metabolites in plants could be as much as three times those of fenpropimorph alone, but the preponderance of the evidence suggests that no more than half that ratio is likely.

Metabolism studies have not shown unchanged fenpropimorph in goats or hens except in hen kidneys. If the residue in animal products is to be defined, residue data would be needed for the metabolites BF 421-1, BF 421-2 and BF 421-3, the predominant residues identified in goats and hens.

¹ Reported. Could not be independently confirmed

² NPD detection

³ Only range reported, not individual analyses

⁴ MS detection

USE PATTERN

Fenpropimorph is registered in over 30 countries (mostly in Western Europe), mainly in cereals for the control of powdery mildew, rust and (in barley) leaf blotch. The EC is reported to be the most widely used formulation. Many of the products are mixed formulations with other active ingredients. GAP relevant to the residue data provided for cereals is shown in Table 17 and includes information from the manufacturer and governments. It refers only to fenpropimorph, although in some cases the formulation might be mixed. GAP for crops other than cereals on which residue trials were reported is shown in Table 18.

Table 17. GAP for field uses of fenpropimorph on cereals.

Crop, country		Applic	ation		PHI, days	Notes (Growth stage of application etc.)	
	Form.	Rate		No.			
		kg ai/ha	kg ai/hl				
Cereals							
Austria	EC	0.75	0.13-0.25	1-2	35		
Norway	EC	0.75	0.38	1*	28	stage 59 * country submission states 1-2 applic. to "corn"; stage 59	
Spain	EC	0.75	≤0.38	1-2	35	stage P	
Sweden	EC	0.19-0.75	0.1-0.38	1-3		stage P, stage 59	
Saudi Arabia	EC	0.75	0.19	1			
UK	EC	0.75		3	35		
Barley					-		
Austria	EC	0.75	0.15-0.38	1-2	35		
Belgium	EC/SC/SE	0.33-0.75	0.09-0.25	1-2	28-42	at last leaf beard or knot; 14 days not confirmed	
Belorussia	EC	0.2-0.3		1-2		stage 29-37	
Croatia	EC	0.75		1	42		
Czech Republic	EC	0.75		1	42		
France	EC/SC/SE	0.26-0.75	0.19-0.75	1-2		1-2 nodes	
Germany	EC	0.42-0.75	0.12-0.14	1-2	35	at infection; stage 29-61	
Greece	EC	0.75	0.15	2	30	at flowering	
Ireland	EC	0.38-0.56	0.12-0.28	2-3	35	stages GS 32, 59, 71	
Italy	EC	0.75	0.15	1-2	35	at infection; blooming	
Kasakstan	EC	0.2-0.3		1-2			
Luxembourg	EC/SC/SE	0.38-0.75	0.1-0.25	1-2	42	last leaf beard, first knot	
Netherlands	EC	0.75	0.19	1-2	42	DC 39, 59 stage	
New Zealand	EC	0.38-0.75	0.19-0.38* 0.38-1.5**	1-2	42	*ground **aerial at disease/ear emergence	
Norway	EC/SC	0.6-0.75		1			
Poland	EC	0.75		1			
Portugal	EC	0.75	0.08	1-2	35	stage 31-Z	
Rumania	EC	0.3-0.75	0.15-0.3	1-2		stage 21-55, 29-37, 51-59	
Russia	EC	0.2-0.3		1-2			
Serbia	EC	0.75		1	42		

Crop, country		Applic	ation		PHI, days	Notes (Growth stage of application etc.)	
	Form.	F	Rate	No.			
		kg ai/ha	kg ai/hl				
Slovakia	EC	0.75		1	42		
Slovenia	EC	0.3-0.75	0.08-0.15	1-2	42	stage 37-49	
Spain	EC	0.75			35	at infection	
Switzerland	EC/SC	0.38-0.75	0.06-0.12	1		stage 31-51	
UK	EC	0.3-0.75	0.08-0.38	1-3	35	at disease; ≥3 months autumn/spring to summer applic.	
Uruguay	EC	0.75	1	1-2	35		
Yugoslavia	EC	0.75		1	42		
Summer Barley							
Belgium	SE/SC	0.8		1-2	28		
Belorussia	EC	0.75		2		vegetative stage	
Denmark	EC	0.75		1-2	30		
Netherlands	EC	0.75	0.13-0.38	1	42	NL submission. Tillering till 1st ears	
Russia	EC	0.75		2		vegetative period	
UK	EC	0.5		1-2	35		
Ukraine	EC	0.75		2		vegetative period	
Spring Barley	1						
Denmark	EC	03-0.75	0.15-0.38	1-2	30	stage 39-49	
UK	EC	0.38-0.75		2	35	UK submission, early milk stage; including ear emergence; ≥3 months autumn/spring-summer applic.	
Winter Barley							
Belgium	SC	0.8		1	28		
Belorussia	EC	0.75		2		vegetative stage	
Denmark	EC	0.3-0.75	0.15-0.38	1-2	30	stage 59	
Germany	EC	0.75		2	35	at infection	
Netherlands	EC	0.75	0.13-0.28	1-2	42	NL submission, at infestation; tillering till 1st ear	
Russia	EC	0.75		2		vegetative period	
UK	EC	0.5-0.75		2-3	35	max. 2 applic. Jan. 1-harvest; before early milk stage, including ear emergence	
Ukraine	EC	0.75		2		vegetative period	
Oats				<u> </u>			
Austria	EC	0.75	0.15-0.38	1-2	35		
Belgium	EC/SC	0.38-0.80	0.09-0.25	1	28-42	at or before infection	
Ireland	EC	0.38-0.56	0.19-0.28	3	35	stage 71	
Italy	EC	0.75	0.15	1-2	35	at infection; start of blooming	
Luxembourg	EC	0.38	0.1-0.25	1	42	totally visible ear	
Netherlands	EC	0.75	0.13-0.38	1-2	42	NL submission; at infestation; DC 39, 59	
Portugal	EC	0.75	0.08	1-2	35	stage 31-Z	

		ation		PHI, days	Notes (Growth stage of application etc.)		
Form.	F	Rate	No.				
	kg ai/ha	kg ai/hl					
SC	0.75		1	35 52*	*before flowering; UK submission		
SC	0.56		2	35 56*	*before flowering; UK submission		
EC	0.38-0.5		3	35	UK submission. Lower rate ≥3 months between autumn/spring and summer applic.		
EC	0.75	0.38	1-2	35	UK submission		
EC	0.75		2	35	UK submission		
SE	0.8		2	42			
SE	0.8		2	42			
EC	0.75		3	35	UK submission. Max. 2 applic. Jan. 1-harvest		
			•				
EC	0.75	0.15-0.38	1.2	35			
SE	0.35		1-2	42			
EC	0.75		1	42			
EC	0.3-0.75	0.15-0.38	1-2	30	stage 39		
EC	0.50.75	0.11-0.14	1-2	35	stage 29-51		
EC	0.75	0.15	1-2	35	at infection; start of blooming		
EC	0.75		1				
EC	0.75		1	42			
EC	0.38		1		stage 39-61		
EC	0.75	0.38	1-2	35	at disease; including ear emergence, before early milk		
EC	0.38-0.5		3	35	≥3 months between autumn/spring-summer applic. UK submission		
SC	0.56		2	35	UK submission		
SC	0.75		1	35	UK submission		
EC	0.75		1-2	35			
					·		
EC	0.75		2				
EC	0.75		2		vegetative period		
EC	0.66		2	35			
EC	0.75		2		vegetative period		
			•				
EC	0.75		2		vegetative period		
EC	0.75		2		vegetative period		
EC	0.75		2		vegetative period		
	SC SC SC SC EC EC EC SE EC	kg ai/ha SC 0.75 SC 0.56 EC 0.38-0.5 EC 0.75 EC 0.75 SE 0.8 EC 0.75 EC 0.75 EC 0.35 EC 0.75 EC 0.75 EC 0.75 EC 0.75 EC 0.75 EC 0.38 EC 0.75 EC 0.38-0.5 SC 0.56 SC 0.75 EC 0.75	kg ai/ha kg ai/hl SC 0.75 SC 0.56 EC 0.38-0.5 EC 0.75 0.38 EC 0.75 0.38 EC 0.75 0.15-0.38 EC 0.75 0.15-0.38 EC 0.75 0.15-0.38 EC 0.75 0.11-0.14 EC 0.75 0.11-0.14 EC 0.75 0.15 EC 0.75 0.38 EC 0.75 0.38 EC 0.75 0.38 EC 0.75 0.38 EC 0.75 SC 0.75 EC 0.75	kg ai/ha kg ai/hl SC 0.75 1 SC 0.56 2 EC 0.38-0.5 3 EC 0.75 0.38 1-2 EC 0.75 0.38 1-2 EC 0.75 2 3 EC 0.75 0.15-0.38 1.2 SE 0.35 1-2 1 EC 0.75 0.15-0.38 1.2 EC 0.75 0.15-0.38 1-2 EC 0.75 0.15-0.38 1-2 EC 0.50-75 0.11-0.14 1-2 EC 0.75 0.15-0.38 1-2 EC 0.75	kg ai/ha kg ai/hl SC 0.75 1 35 52* SC 0.56 2 35 56* EC 0.38-0.5 3 35 EC 0.75 0.38 1-2 35 EC 0.75 0.38 1-2 35 SE 0.8 2 42 EC 0.75 0.15-0.38 1.2 35 EC 0.75 0.15-0.38 1.2 35 EC 0.75 0.15-0.38 1.2 35 EC 0.75 0.15-0.38 1-2 30 EC 0.75 0.15-0.38 1-2 30 EC 0.75 0.11-0.14 1-2 35 EC 0.75 0.11-0.14 1-2 35 EC 0.75 0.15 1-2 35 EC 0.75 0.38 1-2 35 EC 0.75 0.38 1-2 <		

Crop, country		Applic	ation		PHI, days	Notes (Growth stage of application etc.)
	Form.	I	No.			
		kg ai/ha	kg ai/hl			
Wheat						
Austria	EC	0.75	0.15-0.38	1-2	35	
Belgium	EC/SC	0.38-0.75	0.1-0. 25	1-2	14-42	stage 1/L/M; 14 days not confirmed (label states 4 wks. for cereals)
Belorussia	EC	0.2-0.3		1-2		stage 29-37, 51-59
Brazil	EC	0.5-0.93		1-2	35	at first disease
France	EC/SC/W P	0.35-0.75	0.12-0.75	1-2		1-2 node stage
Germany	EC	0.5-0.56	0.11-0.14	1-2	35	stage 29-61
Greece	EC	0.75	0.15	2	30	ear appearance
Ireland	EC	0.38-0.56	0.12-0.28	2-3	35	stages 32, 71
Italy	EC/SC/NP	0.75	0.15-0.5	1-2	35	start of blooming
Kasakstan	EC	0.2-0.3		1-2		
Luxembourg	EC	0.38-0.75	0.1-0.25	1-2	14-42	totally visible ear
Netherlands	EC	0.75	0.1-0.38	1	42	DC 39, 59; development of first ear till blossom
New Zealand	EC	0.38-0.75	0.19-0.38* 0.38-1.5**	1-2	42	*ground **aerial at disease/ear emergence
Poland	EC	0.75		1		
Portugal	EC	0.75	0.08	1-2	35	stage 31Z
Rumania	EC	0.3-0.75	0.15-0.3	1-2		stage 21-55, 29-37, 51-59
Russia	EC	0.24-0.3		1-2		stage 29-37, 51-59
Slovenia	EC	0.3	0.08-0.15	1-2		stage 37-49
Spain	EC	0.75				before flowering; Spanish submission
Switzerland	EC/SC	0.38-0.75	0.06-0.25	1-2		stages 30-32, 30-39, 30-61, 31-32, 29-39, 31-39. 37-61, 37-61, 51-61
UK	EC	0.3-0.75	0.08-0.38	1-3	35	at disease
Spring wheat	•					
UK	EC	0.38-0.56		2	35	including ear emergence; ≥3 months autumn/spring-summer applic.
Summer Wheat						
Belgium	SC/SE	0.38-0.8		1-2	28	
Belorussia	EC	0.75		2		vegetative period
Luxembourg	SE	0.6		2	42	
Netherlands	EC	0.5-0.75	0.08-0.25 for 0.5 kg/ha 0.1-0.38 for 0.75 kg/ha	1-2	42	up to blooming; till first ears visible
Russia	EC	0.75		2		vegetative period
Ukraine	EC	0.75		2		vegetative period
Winter Wheat						
Belgium	SC/EC/SE	0.38-0.8		1-2	28-42	42 days for SE or EC; till ear forming for EC
Belorussia	EC	0.75		2		vegetative period

Crop, country		Applic	ation		PHI, days	Notes (Growth stage of application etc.)
	Form.	F	No.			
		kg ai/ha	kg ai/hl			
Denmark	EC	0.38-0.75	0.19-0.38	1-2	30	stage 59
Hungary	EC	0.75	0.15-0.3	1-2	42	stage 21-55
Netherlands	EC	0.5-0.75	0.1-0.38 h. rate	1-2	42	first ears till blooming
Luxembourg	SC/SE	0.6-0.75		2		
Russia	EC	0.75		2		vegetative period
UK	EC/SC	0.5-0.75		1-3	35	at infection; including ear emergence, before milk; max. 2 applic. Jan. 1-harvest; ≥3 months autumn/spring-summer applic.
Ukraine	EC	0.75		2		vegetative period
Soft Wheat						
Belgium	SC	0.6		1	28-42	
Bulgaria	EC	0.75		1		
Croatia	EC	0.75		1	42	
Czech Republic	EC	0.75		1	42	
Denmark	EC	0.75		1-2	30	
France	EC/SC/SE	0.28-0.75				
Germany	EC	0.75		2	35	
Greece	EC	0.75		2	30	at flowering
Italy	EC	0.75		2	35	at infection
Luxembourg	SC	0.8		1		
Netherlands	EC/SC	0.6-0.75		3	42	1 day for SC
Norway	SC	0.6		1		
Poland	EC	0.75		1		
Serbia	EC	0.75		1	42	
Slovakia	EC	0.75		1	42	
Slovenia	EC	0.75		1	42	
Spain	EC	0.75			35	at infection
Taiwan	EC	0.30		2	14	
Uruguay	EC	0.75	-	1-2	35	
Yugoslavia	EC	0.75		1	42	

Table 18. GAP for the use of fenpropimorph on beans, carrots, leeks and sugar beet,

Crop, country	Application					Notes (Growth stage of application etc.)
	Form.	Ra	No.			
		kg ai/ha	kg ai/hl			
Beans						
UK (incl. field beans)	EC	0.75	0.19-0.38	1-2	35	at disease
Carrots			•			

Crop, country	Application					Notes (Growth stage of application etc.)
	Form.	Ra	Rate			
		kg ai/ha	kg ai/hl			
UK	EC	0.75		3	28	UK submission
Leeks				•		
Belgium	EC	0.75			21	at disease
Denmark	EC	0.56-0.75	0.28-0.38	1-2	28	max 1/yr.; leaves not to be used as fodder
Luxembourg	EC	0.75			21	at disease
Netherlands	EC	0.75	0.12-0.19	2-4	21	Autumn
UK	EC	0.75	0.19-0.38	1-6	21	at disease; 14 day interval
Sugar beet						
Belgium	EC SC ¹	0.56 0.56		1	 28	at disease
Denmark ("beet")	EC	0.56-0.75	0.28-0.38	1-2	28	before Sept.; max 1/yr., leaves not to be used as fodder
France ("beets")	EC	0.75				
Greece sugar beet "beet"	EC EC	0.5-1.1 0.75-1.1	0.15-0.25	1 2	7 7	at flowering 1-2 month pre-harvest
Luxembourg	EC	0.56				at disease
Switzerland	EC ² /EC ³	0.30-0.38	0.06-0.13	1		at symptoms

¹ 375 g fenpropimorph + 125 g carbendazim/l product

RESIDUES RESULTING FROM SUPERVISED TRIALS

In plants

Results of residue trials were available for cereals, beans, leeks, and sugar beet (Ciba, 1994). Summary reports of supervised trials were also received from Norway on barley, onions, wheat, oats and carrots (Race, 1994). In the absence of the detailed data these reports were not reviewed by the Meeting.

In Tables 19-22 underlined residues are from treatments according to or approximating GAP.

<u>Cereals</u>. About 300 reports of supervised trials were available for barley, oats, rye, and wheat, mainly in Western Europe. The results are shown in Tables 19 (barley, 102 reports), 20 (oats and rye, 17 reports), and 21 (wheat, about 180 reports).

Table 19. Supervised trials with fenpropimorph on barley.

Country, year, (variety)	Application*			PHI, days	Fen	Ciba, 1994 Report No.			
	Form. No. kg, kg,	Grain (E=ear)	Straw	Green plant or whole plant					
Barley									
Belgium 1980, 2 sites	EC	1	0.75		71 76 controls	<0.08 <0.08 <0.08			81/046

² 375 g fenpropimorph + 125 g propiconazole/l product

³ 187 g fenpropimorph + 250 g chlorothalonil + 80 g flusilazole/l product

Country, year, (variety)		Appli	cation*		PHI, days	Fen	propimorph	, mg/kg ¹	Ciba, 1994 Report No.
	Form.	No.	kg, ai/ha	kg, ai/hl		Grain (E=ear)	Straw	Green plant or whole plant	
France 1979	EC	2	0.75		48 controls	< <u>0.05</u> <0.05	<u>0.2</u> 0.1		79035
Germany 1989	EC	2	0.56 3/4 × max	0.14	0 21 35 controls	0.6 (E) 0.09 <0.05	 1.1 <0.05	2.4 <0.05	89085
Germany	EC	2	0.56	0.14	0 36 50 controls	 0.1 (E) <u>0.08</u> < <u>0.05</u> <0.05	 0.2 0.4 <0.05	4.4 <0.05	89086
New Zealand 1983	EC	2	0.75 1.5		53 53 controls	0.4 0.5 <0.05	0.6 1.1 <0.05		83033
New Zealand 1984 (Triumph)	EC	2	0.75 1.5		66 66 controls	< <u>0.05</u> <0.05 <0.05	 		84036
New Zealand 1984 (Gold-maker)	EC	2	0.75 1.5		61 61	<u>0.07</u> 0.15			84037
New Zealand 1984 (Triumph)	EC	2	0.75 1.5		62 62	< <u>0.05</u> <0.05	 		84038
Sweden 1981 (Mona)	EC	1	0.75	0.19	69	< <u>0.05</u>	0.3		82064/c
Sweden 1980 ² 2 varieties each rate	EC	1	0.75 2.3		36 36 controls	0.09 (2) 0.4, 0.2 <0.05	0.9, 1.2 2.9, 3.4 0.3		80058

Spring barley									
Denmark 1982	EC	1	0.38	0.19	36 75-88 controls	< <u>0.05</u> (2) <0.05	0.4 (2) 0.05	0.2(2) 0.05	82082, 82083, 82103-4
Denmark 1979-80 7 sites	EC	1	0.75		74-97	< <u>0.05</u> (7)	<0.05 (2) 0.06 (2) 0.07 0.12 <0.05		79069, 80094- 7
D 1 1070	EC	1	1.1		controls				70060
Denmark 1979 3 sites	EC	1	1.1		74-86 controls	<0.05 (3) <0.05	<0.05, 0.07, 0.09 <0.05		79069
Denmark 1980	EC	1	0.75	0.19	<u>30</u>	0.2	0.2		42100F- 80/24E
Denmark 1982	SC ^{3,4}	1	0.6	0.15	84	< <u>0.05</u>	0.2		43805F-82/1E
Denmark 1990	EC ⁵	2	0.38	0.19	0 20-21 34-35			5(2), 7 0.1(2) 0.05, 0.1, 0.06	2145/91 2148/91 2150/91
					55-65 controls	< <u>0.02(2),</u> <u>0.02</u> <0.02-0.04	0.04, 0.08(2) 0.02	0.02-0.1	
Denmark 1990	EC ⁵	1	0.38	0.19	25-26 39-40			0.08, 0.09, 0.1 0.04, 0.05, 0.09	2146/91 2149/91 2151/91
					61-70 controls	< <u>0.02(3)</u> <0.02-0.04	0.04, 0.06, 0.05 0.02-0.03	0.02	
Denmark 1990	EC ⁵	1	0.75	0.38	26 40			0.13 0.06	2147/91
					70 controls	< <u>0.02</u> <0.02	0.05 0.02	0.02	
Finland 1981	EC	1	0.56	0.26	52	< 0.05			3278/81
France 1979	EC	1	0.75		71 control	< <u>0.05</u> <0.05	0.05 <0.05		79028
France 1979	EC	2	0.75	0.15	53 controls	< <u>0.05</u> <0.05	0.5 <0.05		79041
France 1979	EC	2	0.75		43 controls	0.08 <0.05	0.14 <0.05		79048
France 1978	EC	1	1.1	0.19	79	< 0.05	0.31		42100F-78/10 E
France 1979 3 trials	EC	1	0.75	0.13	55-75	< <u>0.05</u> (3)	0.9, 0.2, 0.5		79/3E, 79/4E, 79/5E
Germany 1980	EC	1	0.75	0.13	0 14 21 28 35	< <u>0.05</u>	1.8	14 0.6 0.5 0.6	42100F- 80/6A
Germany 1980	EC	2	0.75	0.13	0 14 21 28 35	2.5 <u>0.1</u>	1 <u>0.3</u>	11 0.6 0.4 	42100F- 80/5A
Germany 1980	EC	2	0.75	0.13	0 21			20 0.4	-80/7A

			1	1	20	I	1	0.3	
					28 35 42	0.1 0.07	<u>0.7</u> <u>1.3</u>	0.3 	
Germany 1980	EC	2	0.75	0.13	0 14 21 28 35 44	0.2 <u>0.06</u> < <u>0.05</u>	0.5 0.8 0.7	8 0.7 0.7 	-80/8A
Germany 1980	EC	2	0.75	0.13	0 14 21 28 35	0.1 <u>0.1</u>	0.9 <u>1.3</u>	27 0.8 0.6 	-80/9A
Germany 1982	SC ^{3,4}	1	0.6	0.1	0 21 28 35 42	< <u>0.05</u> < <u>0.05</u>	0.3 0.4	13 <0.1 <0.1 	43805F-82/7A
Germany 1988	EC ⁶	2	0.56	0.14	0 21 28 35 42	0.3 (E) 0.3(E) 0.1 0.1	1.8 1.4	6.1 1.2 (stalk) 0.9 (stalk)	-88/5A
Netherlands	EC	1	0.75 1.1 1.5		70 70 70 controls	< <u>0.05</u> < <u>0.05</u> < <u>0.05</u> <0.05	< <u>0.05</u> <u>0.08</u> <u>0.06</u> <0.05		79082
Sweden 1982	SC ^{3,4}	1	0.6	0.13	55-79	< <u>0.05</u> (6)			43805F82 /1E, /2E, /3E, 4E, /5E, /6E
Sweden 1983	SE ^{3,4}	1	0.6 0.8X	0.25	50-74	0.1, 0.2(3)			43805F 83/1E-4E
Sweden 1983	SE ^{3,4}	2	0.6	0.25	36 51 58	0.2 0.1 0.2, 0.4			-83/5E-8E
UK 1979	EC	2	0.75 1.1 1.1 1.1	0.36 0.53	(GAP=30) 29 29 29 71 controls	0.2, 0.2 0.2, 0.3 0.2, 0.3 < <u>0.05</u> (2) <0.05	0.6, 0.8 1.2, 1.1 0.6, 0.9 0.5, 0.6 0.14		79052
UK 1979	EC	2	0.75 1.1 1.1 1.1	0.36 0.53 0.53	32 32 32 74 controls	0.08, 0.09 0.09, 0.1 0.1 (2) <0.05 (2) <0.05	0.3, 0.8 0.2, 0.7 0.14, 0.2 0.1, 0.2 0.3		79053
UK 1979	EC	1	1.1	0.45	65-82	<0.05 (5)	<0.05 (4) 0.4		42100F-78/1E , /2E, /3E, /8E, /9E
UK 1979	EC	1	0.75	0.3	0 7 14 21 75	< <u>0.05</u>	1.2	27 7.3 2.6 2	42100F-79/13 A
UK 1979	EC	1	1.1	0.56	29 32 63	0.13 0.08 <0.05	2.5 1.3 1.5		42100F- 79/29E, /32E, /33E

UK 1979	EC	2	0.75	0.3	28 77	< <u>0.05</u>	1.3	1.8	42100F- 79/11A
UK 1979	EC	2	0.75	0.3	28 43 48	< <u>0.05</u>	2.2	1.1	-79/12A
UK 1986	EC	1	1.5	0.63	62	0.09 0.06 malt			42100F-87/9E
Winter Barley			I						
Denmark 1980	EC	1	0.75	0.19	(GAP=30) 67 79	< <u>0.05</u> < <u>0.05</u>	<u>0.6</u> <u>0.1</u>		42100F- 80/19A
Denmark 1980	EC	1	0.56 0.75		67 53 67 control	< <u>0.05</u> < <u>0.05</u> < <u>0.05</u> <0.05	0.4 0.5 0.6		80098
Denmark 1983 2 trials	SC ^{3,4}	1	0.6	0.15	31-32 62-63 76-77	<0.05(2)(E) < <u>0.05(2)</u>	0.2 0.1, 0.3	<0.1, 0.2	4305F-83/1A, /2A
France 1979	EC	2	0.75 1.1	0.15 0.23	60 60 control	< <u>0.05</u> <0.05 <0.05	0.3 0.4 <0.05		79034
France 1979 2 trials	EC	2	0.75		64 ⁷ control	< <u>0.05</u> < <u>0.05</u> <0.05	0.06 0.07 <0.05		79036
France 1978	EC	1	1.1	0.19	75-79	<0.05(2)	<0.05, 0.08		42100F 78/16-17
France 1979 3 trials	EC	1	0.75	0.13	49 88 91	< <u>0.05</u> < <u>0.05</u> < <u>0.05</u>	1 0.1 0.09		-79/1E, /2E, /6E
Germany 1987	EC	2	0.4	0.2	0 15 28 35 42 controls	16 (E) 0.1 (E) 0.05 (E) 0.07 <0.05 <0.05 (grain & E)	0.2 0.3 <0.05		87070
Germany 1981	EC	3	0.75	0.13	0 21 28 35 42	0.8(E) 0.6(E) 0.3 ⁸ 0.3	9.8 5.9 <u>4.8</u> ⁸ <u>4.2</u> ⁸	31	42100F81/1A
Germany 1981	EC	3	0.75	0.13	0 21 28 35 42 56	0.2 (E) 0.1 (E) 0.09 (E) < <u>0.05</u> ⁸	$0.5 \\ \underline{1.5}^{8} \\ \underline{0.7}^{8} \\ \underline{0.6}^{8}$	9.6 1.7	-81/2A
Germany 1981	EC	3	0.75	0.13	0 21 28 35 42 controls	0.4 (E) 0.4 (E) 0.5 (E) 0.1 ⁸ <0.05	1.4 ⁸ <0.1	13 2.3 (stalk) 2.2 (stalk) 1.8 (stalk) <0.1	-81/5A
Germany 1981	EC	3	0.75	0.13	0 21 28	0.4(E) 0.3(E)	1.1 1.3	14	-81/6A

					35 38/42 controls	0.3(E) 0.1 ⁸ (2) <0.05	$\frac{1^8}{0.7^8}(2)$ <0.1	<0.1	
Germany 1981	EC	3	0.75	0.13	8 21 28 31 35 42 controls	1.2(E) 0.7(E) 0.2 0.2 0.2 0.1 ⁸ <0.05	1.6 1.6 2.6 2.1 1.8 ⁸ 1.8 ⁸ <0.1		-81/7A
Germany 1982	SC ^{3,4}	1	0.6	0.1	0 21 28 35 42 50 controls	0.2(E) 0.06 <0.05 <0.05	0.8 0.9 <0.1	7 0.6 0.3	43805-82/5A
Germany 1982	SC ^{3,4}	1	0.6	0.15	0 21 28 35 42 49 controls	0.05 0.07 <0.05	1.3 <0.1 <0.1	6 0.3 0.4 0.2	-82/6A
Germany 1988	EC ⁶	2	0.56	0.19	0 21 28 35	0.3(E) <u>0.2</u>	<u>0.6</u>	11 0.5 0.8 (stalk)	46400F- 88/1A
Netherlands 1979	EC	1 2	0.56 <u>0.75</u>		61 40 controls	< <u>0.05</u> < <u>0.05</u> <0.05	0.08 0.1 <0.05		79081
Netherlands 1979	EC	2	0.56 0.75		46 46 controls	0.05 0.06 <0.05	0.1 0.1 <0.05		80079
Switzerland 1979	EC	2 (GA P=1)	0.75		0 14 28 42 49 control	<0.05 <0.05	0.06 <0.05	8.2 0.2 0.09 0.07 0.06 <0.05	79014
UK 1979	EC	1	<u>0.75</u> 1.1		55 55 controls	0.05, 0.12 0.07, 0.14 <0.05	0.06, 0.16 0.2, 0.12 <0.05		79042
UK 1979	EC	1	0.75 1.1		35 35 controls	0.1 (2) 0.2 (2) <0.05	0.6, 1.1 0.9 (2) <0.05		79044
UK 1979 2 trials	EC	1	1.1	0.5	37 48	0.07 <0.05	1.9 1.6		42100F- 79/17A
UK 1979 2 trials	EC	1	1.1	0.56	35 57	0.09 <0.05	1.1 1		-79/35E, 36E

^{*} kg ai/ha or /hl for mixed formulations refers only to fenpropimorph

1 Numbers in parentheses = no. of samples with that residue

2 Only 4m² plot. Plots in most other trials were ≥40m²

3 200 g fenpropimorph, 330 g chlorothalonil/1 product

4 GAP is for EC formulations

5 375 g fenpropimorph, 225 g prochloraz/1 product

6 563 g fenpropimorph, 187 g tridemorph/1 product

7 Harvest to analysis 3 years. No data on storage conditions or analytical method

Table 20. Supervised trials with fenpropimorph on oats and rye.

Country, year, (variety)	Application Form. No. kg, ai/ha kg, ai/h				PHI, days	Fenpr	opimorph,	, mg/kg ¹	Ciba, 1994 Report No.
	Form.	No.	kg, ai/ha	kg, ai/hl		Grain (E=ear)	Straw	Green plant or whole plant	
Oats									
UK 1980	EC	2	0.75	0.38	38 49 83	<0.05	1.2	1.1 0.5	42100F-80/12A
UK 1980	EC	2	0.75	0.38	35	0.4	0.8		-80/2E
UK 1980	EC	2	0.75	0.38	81	< 0.05			-80/3E
Rye									
Germany 1989	EC ²	2	0.56	0.14	0 21 <u>35</u> 42 controls	0.4(E) < <u>0.05</u> < <u>0.05</u> < <u>0.05</u>	0.3 0.3 <0.05	2.7	89083
Germany 1989	EC ²	2	0.56	0.14	0 22 42 49 controls	0.3(E) 0.1 0.09 <0.05	0.4 0.2 <0.05	3.8	89084
Sweden 1980	EC	1	0.75			< 0.05			80069
Sweden 1984 4 trials	SC ³	1	0.75	0.19	95-111	< <u>0.05</u> (4)			43102F-84/5E, 6E, 7E, 8E
Sweden 1983 2 trials	SC ³	2	0.75	0.19	82	0.3, 0.4			43102F-83/5E, - 83/6E
UK 1983 aerial	EC	2	0.75	1.7	45	< <u>0.05</u>	<u>1</u>		42100F-83/1E
UK 1983	EC	2	0.75	0.34	49	< <u>0.05</u>	1.3		-83/2E
Winter rye									
Sweden 1980	EC	1	0.75		 controls	< <u>0.05</u> <0.05	0.09 <0.05		80067
Sweden 1981	EC	1	0.75	0.19	107 controls	< <u>0.05</u> <0.05	0.05 <0.05		82064/a
Sweden 1981	EC	1	0.75	0.19	80 controls	< <u>0.05</u> <0.05	<u>0.6</u> <0.05		82064/b

 $^{^{8}}$ Referred to UK GAP which allows up to 3 applications. German allows 2 $\,$

Table 21. Supervised trials with fenpropimorph on wheat.

Country, year, (variety)		Appli	cation		PHI, days	Fen	propimorph, r	mg/kg ¹	Ciba, 1994 Report No.
	Form.	No.	kg, ai/ha	kg, ai/hl		Grain (E=ear)	Straw	Whole plant	
Wheat	•	•							
Belgium 1980	EC	1	0.75		66	< <u>0.08</u> < <u>0.08</u>			81/047
France 1979	EC	1	0.75		71 controls	<0 <u>.05</u> (2)	0.05, 0.06		79037
France 1984 5 trials	WP ²	2	0.66	0.13	58-90 controls	< <u>0.05(5)</u> <0.05			84112, -13, -14, -15, -16
Greece 1986 (Vergina & Egis)	EC	2	0.75	0.19	56 controls	< <u>0.05(2)</u>			86208, 86209
Italy 1982 (Creso &	EC	1	0.75		81-88	< <u>0.05</u> (4)	<u>0.1(2)</u> ,		82084, 82085
Irnerio) 2 trials		1	0.75		38-39	< <u>0.05</u> (4)	0.2(2) 1.6, 1.9 1.1, 2.2		
		2	0.75		38-39	< <u>0.05</u> (4)	1.9, 2.3, 1.9, 1.8		
					controls	< 0.05	0.07		
New Zealand 1983 3 trials	EC	2	0.75 1.5		48-52 controls	< <u>0.05(3)</u> < <u>0.05(3)</u> <0.05	2.4, 2.1 4.5, 4.3 0.2		83032/a, /b, /c
Switzerland 1980	EC	2	0.75		52 56	< <u>0.05</u>	0.7	0.9	80031
Spring wheat		1	I				Į.	l	1
France 1978	EC	1	1.1	0.19	48	< 0.05	0.3		42100F- 78/11E
France 1978	EC	1	1.1	0.19	76	< 0.05	< 0.05		-78/12E
France 1978	EC	1	0.75	0.13	32	< <u>0.05</u>	0.9		-79/7E
France 1979	EC	1	0.75	0.13	70	< <u>0.05</u>	<u>0.5</u>		-79/11E
Germany 1977	EC	1	0.75	0.13	0 28 35 42 49 56 63	< <u>0.05</u> < <u>0.05</u>	0.1 0.08	10 1.1 0.4 0.3 0.1	42100F- 77/1A
Germany 1977	EC	1	0.75	0.13	0 28 35 42 49 56 63	< <u>0.05</u> < <u>0.05</u> < <u>0.05</u>	0.2 <0.05 <0.05	14 0.1 0.1 0.05	-77/2A
Germany 1977	EC	1	0.75	0.13	0 28 35 42			17 0.8 0.3 0.1	-77/3A

¹ Numbers in parentheses = no. of samples with that residue ² 281 g fenpropimorph, 200 g prochloraz/1 product ³ 375 g fenpropimorph, 125 g carbendazim/1 product

Country, year, (variety)		Appli	cation		PHI, days	Fen	propimorph,	mg/kg ¹	Ciba, 1994 Report No.
	Form.	No.	kg, ai/ha	kg, ai/hl		Grain (E=ear)	Straw	Whole plant	
					49 56 63 70	< <u>0.05</u> < <u>0.05</u> < <u>0.05</u>	0.08 0.07 0.07	<0.05	
Germany 1977	EC	1	0.75	0.13	0 28 35 42 49 56 63	< <u>0.05</u>	0.09 0.07	8.6 0.2 0.2 0.08 0.1	-77/4A
Germany 1977	EC	1	0.75	0.13	0 21 28 35 42 49 56	< <u>0.05</u> < <u>0.05</u> < <u>0.05</u>	0.2 0.1 0.1	7.8 0.8 0.4 0.4	-77/8A
Germany 1977	EC	2	0.75	0.13	0 21 28 35 42 49 56	< <u>0.05</u> < <u>0.05</u> < <u>0.05</u>	0.3 0.2 0.2	7.3 0.5 0.4 0.3	-77/5A
Germany 1977	EC	2	0.75	0.13	0 21 28 35 42 49 56 63	< <u>0.05</u> < <u>0.05</u> 	0.1 0.1 0.1	17 1.4 0.4 0.3 0.2	-77/6A
Germany 1977	EC	2	0.75	0.13	0 21 28 35 42 49 56	0.07 <0.05 <0.05 <0.05	0.2 0.2 0.06 0.06	11 0.3 0.3	-77/7A
Germany 1978	EC	2	0.75	0.13	0 21 28 35 42 49 56 63	< <u>0.05</u> < <u>0.05</u> < <u>0.05</u>	0.2 0.2 0.2	15 1.1 0.6 0.4 0.3	-78/1A
Germany 1978	EC	2	0.75	0.13	0 21 28 35 42 49	< <u>0.05</u>	0.08	15 0.2 0.08 0.08 <0.05	-78/2A

Country, year, (variety)					PHI, days	Fenj	propimorph,	mg/kg ¹	Ciba, 1994 Report No.
	Form.	No.	kg, ai/ha	kg, ai/hl		Grain (E=ear)	Straw	Whole plant	
					56	< <u>0.05</u>	<u>0.1</u>		
Germany 1978	EC	2	0.75	0.13	0 21 28 35 42 49 56 63	< <u>0.05</u> < <u>0.05</u>	0.05 < <u>0.05</u>	8.1 0.4 0.3 0.2 0.07 0.06	-78/3A
Germany 1978	EC	2	0.75	0.13	0 21 28 35 42 49 56	< <u>0.05</u> < <u>0.05</u> < <u>0.05</u>	0.4 0.3	17 1 0.6 0.5	-78/4A
Germany 1978	EC	1	1.1	0.19	0 21 28 35 42 49 56	<0.05 <0.05 <0.05	0.2 0.08 0.07	17 0.5 0.3 0.3	-78/5A
Germany 1978	EC	1	1.1	0.19	0 21 28 35 42 49 56 63	<0.05 <0.05 <0.05	0.1 0.1 0.2	23 0.5 0.2 0.2 0.1	-78/6A
Germany 1979	EC	2	0.75	0.13	0 14 21 28 35	0.08 0.08	3.6 3.6	14 2.2 1.6	42100F-79/1A
Germany 1979	EC	2	0.75	0.13	0 14 21 28	0.12	<u>1.6</u>	6.9 2.5 1.8	-79/2A
Germany 1979	EC	2	0.75	0.13	0 14 21 28 35 42	< <u>0.05</u> < <u>0.05</u> < <u>0.05</u>	1.9 1.6 1.6	13 1.7 1	-79/3A
Germany 1979	EC	2	0.75	0.13	0 14 21 28 35	0.08 0.06	5.9 4.8 3.6	17 2.8	-79/4A
Germany 1979	EC	2	0.75	0.13	0 15 22 29	0.06 0.06	3.3 2.6	9.1 1.8	-79/5A

Country, year, (variety)	riety)				PHI, days	Fenj	propimorph,	mg/kg ¹	Ciba, 1994 Report No.
	Form.	No.	kg, ai/ha	kg, ai/hl		Grain (E=ear)	Straw	Whole plant	
					36	<u>0.06</u>	<u>2.9</u>		
Germany 1979	EC	2	0.75	0.13	0 18 25 32 39	< <u>0.05</u> < <u>0.05</u>	2.8 2.7	5.1 1.9 1.3	-79/6A
Germany 1979	EC	2	0.75	0.13	0 21 28 35	0.08	<u>0.9</u>	14 1.1 1.1	-79/7A
Germany 1979	EC	2	0.75	0.13	0 21 28 35 42	< <u>0.05</u> <u>0.05</u> < <u>0.05</u>	1.2 2.1 1.8	5.2 1.2	-79/8A
Germany 1979	EC	2	0.75	0.13	0 21 28 35 42	0.07 0.09 0.07	4.4 4.2 4.3	14 3.9	-79/9A
Germany 1979	EC	2	0.75	0.13	0 21 28 35 42 49	< <u>0.05</u> < <u>0.05</u> < <u>0.05</u>	1.1 0.8 0.8	13 0.8 0.5	-79/10A
Germany 1979	EC	2	0.75	0.13	0 28 35 42 49	< <u>0.05</u> < <u>0.05</u>	3.4 2.7	22 1.2 0.9	-79/18A
Germany 1979	EC	2	0.75	0.13	0 28 35 42	< <u>0.05</u>	<u>1.2</u>	20 0.6 0.6	-79/19A
Germany 1979	EC	2	0.75	0.13	0 28 35 42 49	< <u>0.05</u> < <u>0.05</u>	<u>1.2</u>	26 0.6 0.5	-79/20A
Germany 1982	SC ³	1	0.6	0.12	0 21 28 35 42 57 controls	0.2(E) < <u>0.05</u> < <u>0.05</u> < <u>0.05</u> <0.05	1.1 1.2 1.1 1.2 <0.1	7.9 0.6	43805F-82/4A
Germany 1983	SC ³	1	0.6	0.15	0 21 36	0.7(E) <u>0.2</u>	<u>2.3</u>	32	-83/7A ⁴
Germany 1983	SC ³	1	0.6	0.15	0 21 35 42	0.2 0.2	1.9 2.4	39 3.8	-83/8A ⁴

Country, year, (variety)		Appli	cation		PHI, days	Fen	propimorph, r	mg/kg ¹	Ciba, 1994 Report No.
	Form.	No.	kg, ai/ha	kg, ai/hl		Grain (E=ear)	Straw	Whole plant	
					49	0.3	2.2		
Germany 1983	SC ³	1	0.6	0.15	0 21 35 42 49	1.5(E) 1(E) 0.2(E) <u>0.2</u>	2.2(stlk) 2.3(stlk) 3(stlk) 2.3	38	-83/9A ⁴
Germany 1983	EC ⁵	2	0.56	0.14	0 21 28 35 42	0.14(E) 0.1 < <u>0.05</u> < <u>0.05</u>	0.5(stlk) 1 <u>0.7</u> <u>0.6</u>	6.2	46400F-88/6A ⁶
Netherlands 1981	SC ³	1	0.75	0.18- 0.25	61-62	< <u>0.05</u> (8)	$\leq 0.1(3),$ 0.2, 0.4, 0.5, 0.6(2)		43803F-81/1E, /2E, /3E, /4E, /5E, /6E, /7E, /8E
Sweden 1980 2 trials	EC	1	0.56/ 0.75	0.02/ 0.03	95	< <u>0.05(2)</u>			42100F-80/22E, /23E
Sweden 1981 2 trials	EC	1	0.75	0.19	88, 90 controls	< <u>0.05</u> 	0.06, 0.08 <0.05		82064/g, /h
Sweden 1980	EC	1	0.75			< <u>0.05</u>			80071
Winter wheat									
Denmark 1979	EC	1	0.75 1.1 0.75 1.1		99 99 91 91 controls	< <u>0.05</u> <0.05 < <u>0.05</u> <0.05 <0.05	0.05 <0.05 0.1 0.2 <0.05		79069/c 79069/d
Denmark 1980	EC	1	0.56 0.75 0.75		83 83 69		0.2 0.4 0.6		80092
Denmark 1980	EC	1	0.75		99	< <u>0.05</u>	0.2		80093
Denmark 1980	EC	1 2	0.56 0.75 0.75		85 85 43 controls	< <u>0.05</u> < <u>0.05</u> 0 <u>.05</u> <0.05	0.4 0.5 1.6		80099
Denmark 1982	EC	1 2 2	0.38 0.38 0.75		55 36 36 controls			0.1 0.2 0.3 <0.05	82080
Denmark 1982	EC	2	0.38 0.75 0.75		55 36 36 controls			0.2 0.3 0.6 <0.05	82081
Denmark 1982	EC	1 2 2	0.38 0.38 0.75		104 85 85 controls	< <u>0.05</u> < <u>0.05</u> < <u>0.05</u> <0.05	0.2 0.7 0.8 <0.05		82101
Denmark 1982	EC	1 2 2	0.38 0.38 0.75		96 77 77 controls	< <u>0.05</u> < <u>0.05</u> < <u>0.05</u> <0.05	0.3 1 2.3 0.08		82102
Denmark 1982	SC ³	1	0.6	0.15	66	< <u>0.05</u>	0.4		43805F-82/13E
Denmark 1983	SC ⁷	1	0.75	0.19	32 61	<0.05(E)	0.4(stlk)	0.4	43102F-83/1A

Country, year, (variety)		Appli	cation		PHI, days	Fen	propimorph, 1	mg/kg ¹	Ciba, 1994 Report No.
	Form.	No.	kg, ai/ha	kg, ai/hl		Grain (E=ear)	Straw	Whole plant	
					95	< <u>0.05</u>	0.5		
Denmark 1983	SC ⁷	1	0.75	0.19	31 60 96	<0.05(E) < <u>0.05</u>	0.2(stlk) 0.2	0.2	-83/2A
Denmark 1984	EC	2 2	0.56 0.56		70 73 controls	< <u>0.05</u> < <u>0.05</u> <0.05	1.6 1 0.3		85053 85054
Denmark 1984 2 trials	EC ⁸	2	0.56	0.28	70, 73 controls	< <u>0.05(2)</u> <0.05	1.7, 0.09		85075, 85076
Denmark 1990	EC ⁸	2	0.38	0.19	0 18 32 43 57 controls	< <u>0.02</u> <0.02	0.04 <0.02	1.8 0.1 0.05 0.03 <0.02-0.03	2142/91 ⁴
Denmark 1990	EC ⁸	2	0.75	0.38	0 18 32 43 57 controls	< <u>0.02</u> <0.02	0.05 <0.02	7 0.3 0.1 0.08	2143/91 ⁴
Denmark 1990	EC ⁸	2	0.38	0.19	0 20 31 42 63 controls	< <u>0.02</u> <0.02	0.08 <0.02	3.3 0.3 0.2 0.3 <0.02-0.07	2144/91 ⁴
France 1978 3 trials	EC	1	1.1	0.19	45 54 79	<0.05 <0.05 <0.05	0.2 0.1 0.1		42100F-78/13E, /14E, /15E
France 1979 3 trials	EC	1	0.75	0.13	42 48 49	< <u>0.05</u> < <u>0.05</u> < <u>0.05</u>	1.9 0.6 1.5		-79/8E, /9E, /10E
France 1979	EC	2	0.75	0.15	49 controls	< <u>0.05</u> <0.05	0.8 <0.05		79033
France 1979	EC	1	0.75 1.1		59 59 controls	< <u>0.05</u> <0.05 <0.05	0.1 0.1 <0.05		79040
France 1979	EC	1 2	0.75 0.75		70 71	< <u>0.05</u> < <u>0.05</u>	0.06 0.1		79047 79046
France 1979	EC	2	0.75 1.1		30 30 controls	< <u>0.05</u> 0.06 <0.05	0.6 0.8 0.06		79056
Germany 1981	EC	3	0.75	0.13	0 21 28 35 42 52	0.5(E) 0.5(E) < <u>0.05</u> < <u>0.05</u>	2.3 2.9 2.3 2.3	18 1.2	42100F- 81/3A
Germany 1981	EC	3	0.75	0.13	0 21 28			21 2.1 2	-81/4A

Country, year, (variety)		Appli	cation		PHI, days	Fenj	propimorph, 1	ng/kg ¹	Ciba, 1994 Report No.
	Form.	No.	kg, ai/ha	kg, ai/hl		Grain (E=ear)	Straw	Whole plant	
					35 42 62	1.2(E) 0.08 0.07	4.5 3.3 1.4		
Germany 1981	EC	3	0.75	0.13	0 21 28 35 42 controls	0.4(E) < <u>0.05</u> < <u>0.05</u> <0.05	2.5 <u>2.7</u> <u>2</u> <0.1	8.1 1.6	-81/9A
Germany 1981	EC	3	0.75	0.13	0 21 28 35 42 62 controls	0.5(E) < <u>0.05</u> < <u>0.05</u> <0.05	2 2.3 0.9 <0.1	14 1.2 1.2	-81/8A
Germany 1987	EC ⁹	2	0.56	0.14	0 16 28 37 43 59 controls	3.4(E) 0.3(E) 0.3(E) 0.4 0.4 <0.05 <0.05	0.8 0.5 0.7 <0.05		87072
Germany 1988	EC ⁵	2	0.56	0.14	0 21 28 35 42	0.1(E) 0.1(E) <0.05 <0.05	0.4 0.2 < <u>0.05</u> <u>0.8</u> yes	1.4	46400F-88/3A
Germany 1988	EC ⁵	2	0.56	0.19	0 21 28 35 42	0.1(E) <0.05 < <u>0.05</u> < <u>0.05</u>	0.2(stlk) 0.7 <u>0.3</u> <u>0.2</u>	7	-88/4A
Germany 1982	SC ³	1	0.6	0.1	0 21 28 35 42 controls	0.5(E) 0.7(E) < <u>0.05</u> < <u>0.05</u> <0.05	1.8 1.6 <u>1.8</u> <u>1.5</u> <0.1	<0.1	43805F-82/1A
Germany 1982	SC ³	1	0.6	0.15	0 21 28 35 41 54 controls	0.4(E) 0.07(E) 0.06 <0.05	1.7 2 <u>1.4</u> <0.1	6.4 1.1 1.1 <0.1	-82/2A
Germany 1982	SC ³	1	0.6	0.15	0 21 28 35 43 50 controls	<0.05(E) < <u>0.05</u> <0.05	1.1(stlk) <u>0.6</u> <0.1	8 0.7 0.4 0.6	-82/3A
Germany 1983	SC ³	1	0.6	0.15	21 35 42	0.9(E) <u>0.2</u>	1.6(stlk) 2.4	6.2	-83/3A

Country, year, (variety)		Appli	cation		PHI, days	Fen	propimorph, n	ng/kg ¹	Ciba, 1994 Report No.
	Form.	No.	kg, ai/ha	kg, ai/hl		Grain (E=ear)	Straw	Whole plant	
					49 controls	0.1 <0.05	2.1 <0.1	<0.1	
Germany 1983	SC ³	1	0.6	0.15	0 21 35 42 49	0.4(E) 0.4 0.3 0.2	0.6(stlk) 0.7 0.7 2.3 yes	17	-83/4A ⁴
Germany 1983	SC ³	1	0.6	0.15	0 21 35 42 49	1.2(E) 0.4 0.4	1.2(stlk) 1.2 1.1	15 6.5	-83/5A ⁴
Germany 1983	SC ³	1	0.6	0.15	0 21 36 43	0.4(E) 0.3	0.6(stlk) 0.8	31 3.9	-83/6A ⁴
Netherlands 1979 3 trials	EC	1	0.75	0.25	65	< <u>0.05</u> (3)	0.4, 0.4, 0.6		42100F-79/22E, 23E, 24E
Netherlands 1979 4 trials	EC	1	0.75	0.2	72	< <u>0.05</u> (4)	2.4, 2.3, 2.5, 2		-79/25E, 26E, 27E, 28E
Netherlands 1979 4 trials	EC	2	0.75	0.25	71	< <u>0.05</u> (4)	0.3, 0.4(3)		-79/13E, 16E, 18E, 19E
Netherlands 1979 4 trials	EC	1	1.1	0.38	71	< <u>0.05</u> (4)	<u>0.2, 0.3(2),</u> <u>0.4</u>		-79/14E, 15E, 17E, 20E
Netherlands 1979	EC	1	0.75- 1.5		101	< <u>0.05</u> (3)	<u>0.08</u> , <u>0.1</u> (2)		79078
		1 2	0.75, 1.1 0.75		87 87 controls	< <u>0.05(2)</u> < <u>0.05</u> <0.05	0.09, 0.1 0.2 <0.05		
Netherlands 1979	EC	1 2	0.75- 1.5 0.75		92 71 controls	< <u>0.05(3)</u> < <u>0.05</u> <0.05	<0.05, 0.08, 0.1 0.3 <0.05		79079
Netherlands 1979	EC	1	0.56, 0.75		76 controls	< <u>0.05(2)</u> <0.05	0.2(2)		79080
Netherlands 1979	EC	1	0.56 0.75		56 56 controls	< <u>0.05</u> < <u>0.05</u> <0.05	0.2 0.1 <0.05		79083
Netherlands 1979	EC	1 2	0.56 0.75		77 59 controls	< <u>0.05</u> < <u>0.05</u> <0.05	< <u>0.05</u> <u>0.07</u> <0.05		79084
Netherlands 1980	EC	2	0.75 0.94		67 67 controls	< <u>0.05</u> < <u>0.05</u> <0.05	0.8 1.2 <0.05		80056
Netherlands	EC	2	0.75		63 controls	< <u>0.05</u> <0.05	0.8 <0.05		80075
Netherlands 1980 7 trials	EC	1	0.75		10 20 31 38 45	0.07 0.1 <0.05 <0.05 < <u>0.05</u>	1.3 2 0.4 1.2 1.2		80076

Country, year, (variety)		Appli	cation		PHI, days		propimorph, n	ng/kg ¹	Ciba, 1994 Report No.
	Form.	No.	kg, ai/ha	kg, ai/hl		Grain (E=ear)	Straw	Whole plant	
					53 61 controls	< <u>0.05</u> < <u>0.05</u> <0.05	0.9 0.5 <0.05		
Netherlands 1980 6 trials	EC	1-2	0.75		51	< <u>0.05(3)</u>	0.6, 0.7, 2.7(1 appl.)		80077
		1-2	0.75 0.75		74 251 controls	< <u>0.05(2)</u> < <u>0.05</u> <0.05	0.2, 0.3 0.06 <0.05		
Netherlands 1980 2 trials	EC	1	0.75		62 76 controls	< <u>0.05</u> < <u>0.05</u> <0.05	0.4 0.3 <0.05		80080
Sweden 1980 3 trials	EC	1	0.75			< <u>0.05</u> (3)			80067, -68, -70,
Sweden 1981 3 trials	EC	1	0.75	0.19	96-101 controls	< <u>0.05(3)</u>	0.09, 0.2(2) <0.05		82064/d, /e, /f
Sweden 1981 2 trials	EC	1	0.75	0.19	88, 90 controls	< <u>0.05</u>	0.08, 0.06		82064/g, /h
Sweden 1984	EC ⁸	2	0.56	0.28	73 controls	< <u>0.05(2)</u> <0.05	0.9		85076
Sweden 1982 5 trials	SC ³	1	0.6	0.13	57-80	< <u>0.05(5)</u>			43805F- 82/7E, /8E, /9E, /10E, /11E
Sweden 1983 3 trials	SC ³	1	0.6		58-67	0.3, 0.3, 0.4			-83/9E, /10E, /11E
Sweden 1981 2 trials	SC ³	1	0.56		70	< <u>0.05</u> (2)			43800F-81/1E, /2E
Sweden 1984 4 trials	SC ⁷	1	0.75	0.19	98-126	< <u>0.05</u> (4)			43102F-84/1E, /2E, /3E, /4E
Switzerland 1979	EC	2	0.75	0.15	0 14 27 41 55 controls	< <u>0.05</u> <0.05	< <u>0.05</u> <0.05	6.2 0.5 0.2 0.2 0.2 0.2	79015
Switzerland 1981	EC	1	0.56		1 9 21 42 56 controls	< <u>0.05</u> <0.05	0.5 <0.05	1.9 0.7 0.4 0.2	81044
Switzerland 1981	EC	2	0.75		56 controls	< <u>0.05</u> <0.05	1 <0.05		81074
Switzerland 1981	EC	2 1	0.75 0.56		59 59 controls	< <u>0.05</u> < <u>0.05</u> <0.05	0.5 0.2 <0.05		81078 81079
UK 1978 4 trials	EC	1	1.1	0.5	69	<0.05(2)	<0.05, 0.08		42100F- 78/4E,
					78	0.05(2)	0.08, 0.2		/5E,/6E, 7E
UK 1979	EC	1	0.75	0.3	0 7			6.7 1.3	42100F- 79/14A

Country, year, (variety)		Appli	cation		PHI, days	Fenj	propimorph, 1	mg/kg ¹	Ciba, 1994 Report No.
_	Form.	No.	kg, ai/ha	kg, ai/hl		Grain (E=ear)	Straw	Whole plant	
			(1.5X)		14 21			1.1 0.6	
UK 1979	EC	1	1.5	0.75	28 77	0.07 <0.05	6 7.7		-79/15A
UK 1979	EC	1	1.1	0.5	27 62	1.1(E) <0.05	0.3 1.4		-79/16A
UK 1979	EC	1	1.1	0.5	93	< 0.05	1.8		-79/12E
UK 1979 2 trials	EC	1	0.75	0.38	34 84	< <u>0.05</u> < <u>0.05</u>	3.2 1.7		-79/31E -79/44E
UK 1979 2 trials	EC	2	0.75	0.38	34-36 41	< <u>0.05</u> < <u>0.05</u>	3.8 3.3		-79/46E -79/47E
UK 1979 2 trials	EC	1	1.1	0.56	34 91	<0.05 <0.05	3.3 1.8		-79/30E -79/48E
UK 1979 2 trials	EC	2	1.1	0.56	34 41	<0.05 <0.05	4.1 4.9		-79/45E -79/49E
UK 1979	EC	1 2 2	1.1 1.1 1.1 0.75	0.53	84 34 34 34 controls	<0.05(2) <0.05(2) <0.05,0.05 < <u>0.05(2)</u> <0.05	0.3, 0.2 0.4, 0.5 0.5, 0.7 <u>0.5, 0.4</u> 0.1		79065
UK 1979	EC	1 1 2 2	1.1 1.1 1.1 0.75	0.53	88 41 88/41 88/41	0.05(2) <0.05(2) <0.05(2) < <u>0.05</u>	0.05,0.08 0.2, 0.1 0.2(2) <u>0.2(2)</u>		79066
Summer wheat									
Germany 1987	EC ⁹	2	0.56	0.14	0 14 28 36 controls	3.1(E) 0.3(E) 0.2(E) < <u>0.05</u> 0.2(E) <0.05	0.5 <0.05		87069
Netherlands 1980 2 trials	EC	1	0.56 0.75		70 70 controls	< <u>0.05</u> < <u>0.05</u> <0.05	0.2 0.3 <0.05		80078
Sweden 1980	EC	1	0.75 2.3		36 36 controls	< <u>0.05</u> <0.05 <0.05	$\frac{1}{4}$ 4^{10}		80058

Beans. Data were available on fresh ripe bean seeds from 6 trials and on whole bean plants (including seeds and pods) from 3 trials in the UK. The results are shown in Table 22.

Table 22. Residues of fenpropimorph in beans resulting from supervised trials in the UK in 1983. All applications 0.75 kg ai/ha. All EC. All according to GAP.

Commodity (Cultivar)	Application		PHI, days	Fenpropimorph,	Ciba, 1884, Report No.	
	No.	kg ai/hl		Whole plant (incl. pods and seeds)	Seed (fresh ripe)	
Beans (Throws MS)	1 1 2	0.3	131 145 131	0.06 0.09 0.06		42100F- 83/10E- 83/8E-83/9E
Beans (Throws MS)	1	0.3	49		< <u>0.05</u>	-83/22E
Field beans	1		38		< <u>0.1</u>	-83/6E
Beans (Throws MS)	2	0.3	35		0.07	-83/23E
Field beans	2	0.22	29		< <u>0.1</u>	-83/3E
Field beans	2	0.22	26		< <u>0.1</u>	-83/4E
Field beans	2	0.3	72		< <u>0.1</u>	-83/5E

¹ Numbers in parentheses = no. of samples with that residue

² 300 g fenpropimorph + 500 g captafol/kg product ³ 200 g fenpropimorph + 330 g chlorothalonil/1 product

⁴ Although storage was at -20°C, sampling to analysis interval was excessive (3-4 yr.)

⁵ 563 g fenpropimorph + 187 g tridemorph/1 product

⁶ Summary data. Original reports were not in submission

³⁷⁵ g fenpropimorph + 175 g carbendazim/1 product

⁸ 375 g fenpropimorph + 200 g prochloraz/1 product ⁹ 281 g fenpropimorph, + 200 g prochloraz/1 product ¹⁰ Control reported as 4 mg/kg. Mislabelling suspected

<u>Leeks</u>. Eight trials were conducted in the UK in 1982-3. Residues in whole plants are shown in Table 23. All trials were according to GAP.

Table 23. Residues of fenpropimorph in leeks (whole plants) resulting from supervised field trials in the UK. All applications were EC, 0.75 kg ai/ha, and according to GAP.

Year	Арр	olication	PHI, days	Residues, mg/kg	Report no.
	No.	kg ai/hl			
1983	3	0.15	23 35	0.2 0.2	42100F- 83/1A
1983	5	0.17	3 26	<u>0.1</u> <u>0.1</u>	-83/2A
1982	4	0.21	21 38	0.3 0.2	-83/3A
1983	4	0.22	21	0.2	-83/11E
1983	4	0.22	21	0.4	-83/12E
1982	5	0.3	20	<u>0.3</u>	-82/7E
1983	5	0.19	29	< <u>0.05</u>	-83/7E
1983	3	0.13	124	< <u>0.1</u>	-82/8E

<u>Sugar beet</u>. Data from supervised trials in six countries were available in 29 reports. Application rates were within the range of reported Western European GAP, for which the shortest PHI is 7 days. The results are shown in Table 24.

Table 24. Residues of fenpropimorph in sugar beet resulting from supervised field trials.

Country, year		Application (kg ai/ha or /hl for mixed formulations refers only to fenpropimorph				•	pimorph, g/kg	Ciba, 1994 Report no.
	Form.	No.	kg ai/ha	kg ai/hl		Roots	Leaves	
Belgium 1985	EC	1	0.75	0.13	72 46 62 control	<0.02 <0.02 <0.02 <0.02	0.06 0.07 0.03 <0.02	86/10160 (= 86/200) 86/10161 86/10162
France 1983	EC	1 2 1	0.75	0.16	72 68 82	<0.05 <0.05 <0.05	0.2 0.3 0.09	42100F-83/13E- 83/14E-83/15E
France 1983	SC ¹	1 1 1 2	0.75	0.06	77 72 82 68	<0.05 <0.05 <0.05 <0.05	0.2 0.1 0.1 0.14	43102F-83/1E -83/2E -83/4E -83/3E
France 1982 1983 1982 1983 1983 1982 1982 1982	WP ²	1 1 2 2 2 2 2 2 2 2	0.38	0.08	49 70 25 34 42 49 75 76 controls	<0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	0.06 <0.05 0.1 0.07 0.08 <0.05 0.09 0.07 <0.05	82112 83105 82113 83106 83107 82114 82117 82118
Greece 1984	EC	3	1.1		0		2.6	84/10156

Country, year			ni/ha or /hl for only to fenpro		PHI, days	•	pimorph, g/kg	Ciba, 1994 Report no.
	Form.	No.	kg ai/ha	kg ai/hl		Roots	Leaves	
					3		1	
					6		0.6	
					9 12		0.4 0.3	
					15		0.3	
					18		0.2	
Italy 1989	EC	2	0.56	0.11	3	< 0.05	0.8	49100F-89/3E
					5	< 0.05	1.6^{3}	-89/4E
					16	< 0.05	0.2	-89/1A
					31	<0.05	0.06	-89/1A
					controls	< 0.05	< 0.05	
Italy 1989	EC	3	0.56	0.14	1	< 0.05	0.2	452100F89/2A
					7	< 0.05	0.9	
					13	< 0.05	0.3	
					32	< 0.05	0.14	
Italy 1988	EC	3	0.56	0.11	2	< 0.05	0.5	42100F88/8A
					8	< 0.05	0.8	
					15	< 0.05	0.3	
					30	< 0.05	1	
Italy 1987	EC	3	0.75	0.19	20	< 0.05	< 0.05	42100F87/12E
Sweden 1985	EC	3	0.75	0.3	65	< 0.05	0.2	42100F85/7E
Switzerland 1988	EC ⁴	2	0.56	0.07	96	< 0.05	0.3	88150
					113		< 0.05	
Switzerland 1988	EC ⁴	2	0.56	0.07	76	< 0.05	0.3	88151
					94		0.1	
Switzerland 1988	EC ⁴	1	0.75	0.19	51	< 0.05	1	88152

¹ 375 g fenpropimorph + 125 g carbendazim/1 product

In animals

Of the crops for which maximum residue levels have been estimated by the Meeting, those which may be fed to animals include the grains, straws, forage and fodders of cereals, and sugar beet tops. No information was available on residues in sugar beet molasses or pulp.

Many assumptions might be made for estimating residue levels in animal products. A theoretical, if perhaps unlikely, worse-case beef cattle diet might be 65% grain, 25% cereal forage and 10% dry cereal straw and fodder. That of a dairy cow might be 40% grain, 50% cereal forage and 10% dry cereal straw and fodder. Poultry might be fed cereal grain up to 70% of the diet. Assuming that fenpropimorph residues might occur up to 0.5 mg/kg in cereal grain, up to 2 mg/kg in fresh cereal forage, up to 5 mg/kg in dry cereal straw and fodder, and up to 1 mg/kg in sugar beet leaves, the maximum theoretical dietary intakes of fenpropimorph would be 1.3 ppm for beef cattle, 1.7 ppm for dairy cattle and 0.35 ppm for poultry if sugar beet leaves, which contribute least to the theoretical intake, are excluded.

No conventional feeding trials were reported to the Meeting. The goat metabolism studies described earlier were at 0.6 ppm (Hawkins *et al.*, 1980a, morpholine label), 1421 ppm (morpholine label) and 2335 ppm (phenyl label) (Ritter, 1989a) in the diet. The feeding levels for hens were 51.5

² 188 g fenpropimorph + 50 g carbendazim + 400 g mancozeb/kg product

³ An anomalous 1.6 mg/kg is the reported value. No explanation. Ripe leaf

⁴ 375 g fenpropimorph + 225 g prochloraz/1 product

ppm (phenyl label) and 39.3 ppm (morpholine label) (Ritter, 1989b). Only the 0.6 ppm feeding level in goats and the feeding levels in the hens are even remotely close to expected dietary intakes. The total residues, expressed as fenpropimorph, that might occur in tissues, milk and eggs under worse-case assumptions of 1.7 ppm in the diet of cattle and 0.35 ppm in that of poultry are presented in Table 25. Calculations based on the higher-level goat metabolism studies yield somewhat lower estimates than those shown in Table 25.

Table 25. Theoretical estimates of total residues, expressed as fenpropimorph, in cattle and poultry calculated from metabolism studies on goas and hens.

Sample		Resi	idues, mg/kg fenprop	imorph equivale	nts	
	Found in goats receiving 0.6 ppm	Calculated in cattle ¹	Found in hens receiving 41 ppm	Calculated in poultry ²	Found in hens receiving 55 ppm	Calculated in poultry ³
Muscle	0.004	0.01	0.4	0.003	0.34	0.002
Fat	0.012	0.03	1.4	0.01	1.1	0.007
Liver	0.103	0.3	2.8	0.02	3.9	0.025
Kidneys	0.029	0.08	2.8	0.02	2.4	0.05
Milk	0.008	0.02				
Eggs			0.5	0.004	1.7	0.01

¹ Assuming 1.7 ppm intake. Factor = 1.7/0.6 = 2.8

The relevance of these calculations to the estimation of maximum residue levels for animal products is discussed in the appraisal.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

Citrus fruit were dipped in a 1500 mg/l aqueous solution of fenpropimorph and analysed for residues after 2 days at room temperature and 30 days at 5°C (Lafuente *et al.*, 1986). The results are shown in Table 26.

Table 25. Fenpropimorph residues (mg/kg) in citrus stored for 2 days and 30 days after dip treatment at 1500 mg/l (Lafuente *et al.*, 1986).

Sample	Washington i	navel oranges	Hernandina clementines		
	2 days ¹	30 days ²	2 days ¹	30 days ²	
Whole fruit	1.9	0.73	1.0	0.79	
Peel	5.2	2.8	4.6	4.1	
Albedo	0.46	0.41	1.2	1.1	
Pulp	0.04	0.05	0.05	0.07	

¹ Room temperature

² Assuming 0.35 ppm intake. Factor = 0.0085

 $^{^{3}}$ Assuming 0.35 ppm intake. Factor = 0.006

 $^{^2\,5^{\}circ}\mathrm{C}$

In processing

Four summary reports were available on the processing and baking of wheat. The results are shown in Table 27.

Table 27. Residues in processed fractions of wheat field-treated with fenpropimorph at 0.75 kg ai/ha.

Reference	Sample	Resi	due, mg/kg, after into	erval (days)
		35	46	169-189
BASF 1979a	grain rolls	0.08		<0.05
BASF 1979b	grain rolls	0.05		<0.05
BASF 1979c	grain rolls	0.06		<0.05
BASF 1985	grain grain after cleaning wholemeal wholemeal bread bran semolina bran "Nachmehl" white flour white bread		<0.051	0.07 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05

¹ 59% recovery

Residues in the edible portion of food commodities

Cereal grains are the only edible food commodities in which the estimated maximum residue levels are above the LOD. The data indicate that median residues in grain are likely to be <0.05 mg/kg and maximum residues unlikely to exceed 0.5 mg/kg. As noted above, summary reports suggest that residues in processed grain fractions are likely to be lower than in grain, and residues would probably not be detectable (<0.05 mg/kg) in bread on the basis of the median residues expected in grain. These conclusions need to be confirmed.

Fenpropimorph residues in citrus pulp from post-harvest dip treatments are likely to be less than 10% of those in the whole fruit (Table 26).

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No information was provided.

NATIONAL MAXIMUM RESIDUE LIMITS

Information on national MRLs was provided for 11 countries in which fenpropimorph is registered.

National maximum residue limits for fenpropimorph.¹

Commodity	Country	MRL (mg/kg)
Commodity	Country	WIKE (IIIg/Rg)

Commodity	Country	MRL (mg/kg)
Cereal grains	Austria Belgium France Germany Hungary Italy (barley, oats, rye, wheat) Luxembourg Netherlands New Zealand Spain Switzerland	0.5 0.2 0.1 0.5 0.05 0.5 0.2 0.05 0.5 0.2 0.1
	Taiwan	0.1
Cereal straw	Hungary Spain	0.5 1
Leeks	Belgium Netherlands (December 1993)	0.5 0.5
<u>Milk</u>	Netherlands (December 1993)	0.05
Meat	Netherlands (December 1993)	0.05
Other food and feedstuffs of p	<u>plant origin</u> Austria Belgium Germany Netherlands Spain	0.1 0.05 0.1 0.05 0.05

¹ All MRLs are for fenpropimorph *per se*, including those for milk and meat in The Netherlands

APPRAISAL

Fenpropimorph is a fungicidal pesticide whose major use is for the control of diseases in cereals. It is formulated into more than 49 products, mostly in mixtures with other fungicides, although the EC formulation is reported to be the most commonly used. Typically 1-3 field applications are made at rates of 0.3 to 0.75 kg ai/ha. Fenpropimorph was reviewed for the first time at the 1994 JMPR which considered toxicological aspects. Owing to the late receipt of residue data the FAO Panel review was postponed until 1995.

<u>The fate of residues</u> has been studied in animals, plants, soil, water and soil/water systems. The metabolism in plants is similar to that in animals to the extent that oxidation is the first stage of metabolism, followed by degradation of the morpholine ring. There are differences, especially in that generally fenpropimorph is the main residue in plants but is not found in animals (fenpropimorph reported in hen kidneys is an exception).

The metabolites referred to by codes are identified below.

BF 421-1	4-{3-[4-(2-hydroxy-1,1-dimethyl)ethylphenyl]-2-methylpropyl}-cis-2,6-		
	dimethylmorpho	oline	
BF 421-2	2-methyl-2-{4-[2-methyl-3-(cis-2,6-dimethylmorpholin-4-	
	yl)propyl]phenyl}propionic acid		
BF 421-2-Me	methyl	2-methyl-2-{4-[2-methyl-3-(cis-2,6-dimethylmorpholin-4-	
	yl)propyl]pheny	1}propionate	
BF 421-2 conjugate	2-methyl-2-{4-[2-methyl-3-(cis-2,6-dimethylmorpholin-4-yl)-		
	propyl]phenyl}r	propionic acid conjugate	

BF 421-3	2-methyl-2-{4-[2-methyl-3-(cis-2-hydroxymethyl-6-methylmorpholin-4-
	yl]propyl)phenyl}propionic acid
BF 421-4	2-methyl-2-{4-[2-methyl-3-(2-hydroxypropyl)aminopropyl]-
	phenyl}propionic acid
BF 421-7	[3-(4- <i>tert</i> -butylphenyl)-2-methylpropyl](2-hydroxypropyl)amine
BF 421-10	cis-2,6-dimethylmorpholine
BF 421-13	4-[3-(4- <i>tert</i> -butylphenyl)-2-methyl-1-oxopropyl]- <i>cis</i> -2,6-dimethylmorpholine
BF 421-15	4-[3-(4- <i>tert</i> -butylphenyl)-2-methylpropyl]- <i>cis</i> -2,6-dimethylmorpholine-3-one
BF 421-16	2-methyl-2-[4-(carboxyphenyl)]propan-1-ol
BF 421-17	2-methyl-2-[4-(2-carboxypropyl)phenyl)]propionic acid

The Meeting noted that the animal metabolites (and/or their conjugates) BF 421-3, BF 421-4, BF 421-16 and BF 421-17 had not been reported in plants. The plant metabolites BF 421-2-Me, BF 421-7, BF 421-10, BF 421-13 and BF 421-15 have not been reported in animals. Residues of most of these would be expected to be less than 1/4 of those of the parent compound, although BF 421-7 has been reported in one study of wheat metabolism to be at a similar level to the parent compound in wheat straw under some conditions. In any future animal metabolism studies it may be prudent to analyse for these plant metabolites.

Studies on rats, goats and poultry show rapid absorption from the gastrointestinal tract and rapid elimination of residues in the faeces, urine or excreta, slightly less in the urine than the faeces. High bile residues were consistent with a high rate of faecal elimination.

The three test animals showed similar but not identical metabolism, consisting mainly in progressive oxidation of methyl groups of both the *tert*-butyl group and the morpholine ring. Further metabolism of the morpholine ring was demonstrated by the expiration of significant amounts of $^{14}\text{CO}_2$ by the rat. The expiration of $^{14}\text{CO}_2$ was not measured in goats or hens.

In animals, residues of the parent compound were detected only in hen kidneys. The highest residues of identified metabolites were in the liver and kidneys. The main metabolites were BF 421-1, detected in goat fat and faeces and hen plasma, BF 421-2 detected in several goat tissues and milk and hen kidneys and liver, and BF 421-3 detected in goat and hen kidneys and goat fat.

No conjugates were detected in hens, whereas conjugates of BF 421-2 were found in goat liver and a conjugate of BF 421-3 was found only in rat urine, faeces and bile. Metabolites BF 421-16 and BF 421-17 were detected only in rat urine, kidneys and faeces.

Although the faeces and urine were by far the predominant routes of elimination of residues, approximately 50% of the material administered to goats in one study was not accounted for by analyses of the urine, faeces, milk, bile, cage washes, and tissues or organs, and was assumed to remain in the gastrointestinal tract. This was not documented nor was radioactivity measured in expired CO₂, which had been shown to be a significant route of elimination in rats. Elimination in the faeces and urine combined was reported to be about 84% in a separate study in which animals were killed 24 hours after the last dose as compared with 5 hours in the first study.

In studies of <u>cereal metabolism</u> fenpropimorph with the benzylic carbon labelled was applied to leaf surfaces. Over 60% of the residue on the day of application was on the leaf surface and was mainly unchanged fenpropimorph. After three weeks about 30% of the applied radioactivity was absorbed into the leaf and only about 7% of that was unchanged fenpropimorph. Low levels of radioactivity were translocated to untreated plant parts. With the ring-labelled compound (either ring) most of the radioactivity was extractable and fenpropimorph was generally by far the main residue, although BF 421-7 was sometimes of the same order in straw, depending on the conditions. Other metabolites identified in cereal plants included BF 421-1, BF 421-2, <u>BF 421-2-Me</u>, <u>BF 421-10</u>, <u>BF</u>

421-13 and BF 421-15. The underlined compounds were not reported in studies of animal metabolism.

The radioactivity in cereal grains was too low for identification and was mainly associated with the starch fractions, although it did not appear to be incorporated into the glucose units. Less was associated with protein and polysaccharide fractions.

Rotational crop plantings in soil containing 0.42 mg/kg [¹⁴C]fenpropimorph resulted in residues in the mature crop of ≤0.01 mg/kg fenpropimorph equivalents in spinach and sugar beet tops, <0.02 mg/kg in green wheat plants and 0.004 mg/kg in mature sugar beets. Since metabolism studies showed relatively rapid degradation of fenpropimorph, the rotational crop data suggest that, at least in sandy loam soil, only trace residues would be expected in rotational crops after the previous crops had been sprayed with fenpropimorph.

Under neutral conditions fenpropimorph is largely stable in water. Degradation in soil proceeds by oxidation and opening of the morpholine ring to give BF 421-2, BF 421-7, BF 421-8 and BF 421-10. In addition, BF 421-13 and BF 421-15 can result from the photolysis of soil residues. The half-life of fenpropimorph in soil varies according to the conditions, ranging from 10 to 90 days. In aerobic water/sediment systems degradation is similar, except that the morpholine ring is not opened.

Analytical methods are available for the determination of fenpropimorph in plant materials, soil and water. For plants the emphasis in method development has been on cereals (grain, plant, forage and straw) and citrus. Most methods rely on extraction with methanol, chloroform, methylene chloride or other solvents and concentration, with or without clean-up by cation exchange, alumina chromatography or liquid/liquid partition. Determination is generally by GLC with an NPD or occasionally HPLC with UV detection or GC-MS, depending on the method and the matrix being analysed. A limit of determination of 0.05-0.1 mg/kg should be achievable in most cases and perhaps lower, especially for water, soil and grain. However, for some methods sufficient information was not available for an independent estimate of the limit of determination.

Methods for fenpropimorph acid (BF 421-2) in soil depend on methylation with diazomethane before determination by GLC with an NPD or by GC-MS.

The stability of fenpropimorph in stored analytical samples of wheat grain, green plants and straw and of fenpropimorph and its acid in soil was investigated over a 2-year period. The maximum losses of the parent compound or its acid were about 25%, but generally less than 10%.

Because residues in plants are generally mainly the parent compound the Meeting concluded that for regulatory purposes the residue in plants should be defined as fenpropimorph. For risk assessment purposes, the data suggest that the total residues of fenpropimorph plus its major plant metabolites will almost certainly be no more than 3 times the level of fenpropimorph alone, but more likely less than twice that level. Consideration of a definition of the residue in animal products must await further information. The Meeting was informed by the manufacturer that national definitions of the residue in foods and feeds of plant origin include only fenpropimorph. This is also the definition for residues in meat and milk in The Netherlands.

Supervised residue trials gave the following results.

Beans. Residues in fresh ripe bean seeds in 6 trials in 1983 in the UK at GAP rates were <0.05, <0.1 or 0.07 mg/kg after 26-72 days compared to the 28-day UK PHI. Residues in 3 UK trials, also according to GAP, were 0.06(2) and 0.09 mg/kg in whole plants. The results suggest that residues are unlikely to exceed 0.1 mg/kg in fresh shelled beans, but because the data were relatively old and limited and the residue reports did not include information on method(s) of analysis, sample chromatograms, control values, or analytical recoveries, the Meeting concluded that the information

was insufficient to support a limit.

<u>Carrots</u>. The Meeting did not use summary data submitted from Norway in the absence of detailed reports, acknowledged to be unavailable.

<u>Cereals</u>. Data from many supervised trials with a wide geographical distribution (mainly Western Europe) were available. A significant number of the reports were not sufficiently documented for full confidence in the validity of the data, but because over half of the reports were considered reasonably well documented and because of the similarity and mutual support of the results among the cereals, the Meeting concluded that limits could reasonably be recommended, even when discounting the less well documented studies. In most cases grain residues were less than 0.05 mg/kg.

Most of the trials were on barley and wheat. Although the results were minimal for oats and somewhat scanty for rye, the Meeting considered the data on cereal grains to be mutually supportive.

Barley. Over 100 supervised trials were conducted in 9 Western European countries and New Zealand, covering the range of reported GAP including 1-3 applications, usually of EC formulations, at rates ranging from 0.38 to 0.75 kg ai/ha and PHIs ≥30 days. Data were also available for exaggerated rates and shorter PHIs. Residues in <u>barley grain</u> judged by the Meeting to be from treatments according to GAP, expressed as mg/kg with the number of results in parentheses were/ ≤0.05 (62), <0.08 (2), 0.06 (3), 0.07 (4), 0.08 (3), 0.09 (4), 0.1 (12), 0.2 (8), 0.3 (2), 0.4 (2) and 0.5 (1), with a median of <0.05 mg/kg. The residues of 0.3 mg/kg (from the same trial), 0.5 mg/kg and one of 0.4 mg/kg were from German trials according to German GAP, but with 3 applications instead of the two allowed. GAP in the UK allows three applications if the last two are after January as they were in these trials. The distribution in <u>barley straw</u> was ≤0.1 (29), 0.2-0.5 (23), 0.6-1 (19), 1.1-1.5 (13), 1.8 (4), 1.9 (1), 2.2 (1) and 4.2-4.8 (2). The last two residues were from the same German trial as the residues of 0.3 mg/kg in the grain. In <u>whole barley plants</u> residues from applications according to GAP at the days PHI in parentheses were 2.4-31 mg/kg (0), 7.3 mg/kg (7-9), 0.2-2.6 (14-18), 0.08-2 (20-25), 0.09-1.8 (26-30), 0.05-0.2 (30-40) and 0.04-0.07 mg/kg (40-50).

Although there was an adequate number of results, there were deficiencies in the detail provided for a significant number of the trials. For example, the sample handling and storage conditions and analytical recovery values were not provided, nor were analytical methods identified for about a third of the studies. No sample chromatograms were provided for any of the field studies, although representative chromatograms from grain and straw analyses were provided in separate validations of one of the methods used (Method 840-MD-02).

By putting greater weight on the better documented studies and noting that data among the cereals were similar and mutually supportive, the Meeting estimated a maximum residue level of 0.5 mg/kg for barley grain. No data on moisture content were available to estimate a maximum level in fodder on a dry weight basis, but with maximum expected residues of 2 mg/kg in fresh barley forage from GAP treatments, and assuming 30% dry matter, a theoretical level of 7 mg/kg could be estimated for dry fodder. Observing that median residues in fresh forage after the shortest GAP PHI would be <0.5 mg/kg, and noting maximum residues in barley straw of 4.8 mg/kg, the Meeting estimated a maximum residue level of 5 mg/kg for dry barley straw and fodder.

Wheat. Over 150 supervised trials were conducted in 10 Western European countries and in New Zealand. Some of the German trials were with higher application rates than reported German GAP, but as they were according to the GAP of neighbouring countries the Meeting considered that they should not be disregarded. The distribution of residues, with the number of results within reported GAP in parentheses, were for grain <0.05 (203), 0.05-0.09 (18) and 0.1-0.4 (20), with a median of <0.05 mg/kg, and for straw <0.05 (9), 0.05-0.09 (31), 0.1-0.4 (59), 0.5-0.9 (39), 1-2 (32), 2.1-2.9 (26), 3-4 (9), 4.1-5 (7) and 5.9 mg/kg, with a median of 0.7 mg/kg. Residues in whole plants were 0.08-39

mg/kg at day 0, decreasing to about 0.05-1 mg/kg at 30-60 days.

As in the case of barley some of the studies were not well documented, especially with respect to sample handling and storage conditions and the identification or provision of the analytical methods used. In approximately 6 cases the interval from sampling to analysis was ≥ 2 years and in four trials the plot size was only 5 m². No representative chromatograms were provided with the supervised trials data although, as with barley, they were provided with some method validations. The Meeting considered the documentation for approximately 7% of the 150 trials to be unacceptable. Approximately 50% were reasonably well documented and another 40% fairly well documented.

As with barley, the Meeting gave greater weight to the better documented studies, took into account the similarities among the different cereals, and estimated a maximum residue level of 0.5 mg/kg for wheat grain. Noting that only one residue (of 5.9 mg/kg) in over 250 barley and wheat straw samples exceeded 5 mg/kg, the Meeting concluded that it was unlikely that residues in straw would exceed 5 mg/kg.

Again, no moisture contents were available to estimate fodder residues on a dry weight basis. Assuming 25% moisture in the whole plants, and estimating a maximum residue of 2 mg/kg in fresh wheat fodder (whole plants), a theoretical maximum level of 8 mg/kg could be estimated for the fodder on a dry weight basis. Observing that, as in the case of barley,the median residue in fresh fodder after the minimum 30-day GAP PHI is likely to be less than 0.5 mg/kg compared with the estimated maximum of 2 mg/kg, the Meeting estimated a maximum residue level of 5 mg/kg for wheat straw and fodder, dry.

Oats. Data were available from three supervised trials in the UK in 1980, all within reported UK GAP for the EC formulation (2 x 0.75 kg ai/ha; 30-day PHI). Residues in grain were <0.05 (2) and 0.4 mg/kg after 35 days and <0.05 mg/kg after 81-83 days, in straw 1.2 and 0.8 mg/kg after 83 and 35 days respectively, and in green plants up to 1.1 and 0.5 mg/kg after 38 and 49 days respectively. The studies were reasonably well documented, except that the analytical method used was not identified. Taking into account the mutual support of the barley and wheat data, the Meeting estimated maximum residue levels of a 0.5 mg/kg for oat grain and 5 mg/kg for oat straw and fodder, dry.

Rye. Data were available for about 12 supervised trials conducted in 1980-84 in Germany, the UK and Sweden. Swedish trials with EC formulations were according to GAP reported for the UK and Germany, which allows up to two 0.75 kg ai/ha applications of the EC with a 35-day PHI.

Four of the Swedish trials with an SC formulation were within reported UK GAP for SC but two trials giving residues of 0.3 and 0.4 mg/kg after 82 days were from two applications at 0.75 kg ai/ha whereas UK GAP allows only one at that rate (2 are permitted at 0.56 kg ai/ha). At PHIs at or longer than the 35-day German and UK GAP and relating Swedish results to GAP in those countries, the residues which reflected GAP were ≤0.05 (11 results), 0.09 and 0.1 mg/kg in grain and 0.05, 0.09, 0.2, 0.3, 0.4, 0.6, 1 and 1.3 mg/kg in straw. Residues in whole plants were 2.7 and 3.8 mg/kg on the day of application. Most of the studies were reasonably well documented, although as with oats in several cases information was lacking on the analytical methods used or on analytical recoveries and representative chromatograms were not provided with the trials. Taking these results into account and with the support of those for barley and wheat, the Meeting estimated maximum residue levels of 0.5 mg/kg for rye grain and 5 mg/kg for rye straw and fodder, dry.

<u>Leeks</u>. Residues in whole leeks in eight supervised trials in the UK in 1982-3 (6 locations) were 0.1 to 0.4 mg/kg 20 to 38 days after 3-5 GAP applications of 0.75 ai/ha. The GAP PHI is 21 days. There appears to be little correlation between the number of applications or PHI (3-28 days) and residue levels. With one exception the interval from sampling to analysis was ≤6 months and the samples were reported to have been deep frozen. In the absence of more detailed information on sample

handling and methods of analysis (the data suggest that there were more than one), sample chromatograms, analytical recoveries and control values, the Meeting could not estimate a maximum residue level. If adequate supporting information can be provided to a future Meeting, the data may support an estimate of 0.5 mg/kg for leeks.

Onions. The Meeting did not use summary data submitted from Norway in the absence of detailed reports, which were acknowledged to be unavailable.

Sugar beets. Over 25 supervised trials (1982-89) were conducted in 6 Western European countries at GAP rates (0.38-1.1 kg ai/ha), with samples of sugar beet and sugar beet leaves taken at PHIs ranging from 0 to 113 days. GAP for sugar beets was reported only for Belgium, Denmark, Greece, Luxembourg and Switzerland but the trials, which were in Belgium, France, Greece, Italy, Sweden and Switzerland, cover the full range of reported GAP for Western Europe, in which the minimum PHI is 7 days (Greece). In all cases 6 or more days after the last application residues were reported as ≤0.05 mg/kg in the roots and ≤1 mg/kg (0.09 mg/kg median) in the leaves, with leaf residues approaching 1 mg/kg in several trials. Where they were provided, control values for both roots and leaves were <0.05 mg/kg. No data were available for sugar beet molasses or pulp.

The Meeting was not satisfied with the level of information provided in approximately half the trials on one or more of the following items/ sample handling and storage conditions, recovery data, control values and plot sizes. More often than not this was because only the analytical reports (without the corresponding field reports) were submitted.

In most cases the analytical method was identified as being one of those for which information was supplied to the Meeting, although no sample chromatograms were provided for sugar beets. They were provided for other crops in separate method validations. Analyses were generally conducted within 14 months of sampling, usually less. No storage stability studies were provided for sugar beet, although fenpropimorph has been shown to be stable for over a year in soil and wheat grain, straw and plants under frozen storage.

Although the Meeting found the overall submission to be only marginally acceptable, because several of the trials in different geographical areas and in different years were relatively well documented it concluded that maximum residue levels could be estimated. The Meeting considered the submission of validation of methods specifically for sugar beet roots and leaves, and of storage stability studies for a root crop to be highly desirable for a future JMPR evaluation in order to confirm the estimates. The Meeting estimated that fenpropimorph residues are unlikely to exceed 0.05 mg/kg (limit of determination) in sugar beet roots or 1 mg/kg in sugar beet leaves when GAP is followed.

<u>Animal products</u>. Of the crops for which maximum residue levels were estimated by the Meeting, those which may be fed to animals include the grain (0.5 mg/kg), straws and fodders of cereals (5 mg/kg), cereal grain forage (2 mg/kg) and sugar beet tops (1 mg/kg). No information was available on sugar beet molasses or pulp.

Assuming worst-case feeding situations for the above crops and maximum residue levels in them, maximum theoretical fenpropimorph levels in the feed would be 1.3 ppm for beef cattle, 1.7 ppm for dairy cattle and 0.35 ppm for poultry.

No conventional feeding trials data were provided to the Meeting. It is possible to make a crude estimate of total residues as fenpropimorph equivalents in cattle and poultry using the above estimates in conjunction with residue levels found in the metabolism studies on goats and hens. With these assumptions the total residues (mg/kg) in cattle/poultry would be muscle 0.01/0.003, fat 0.03/0.01, liver 0.3/0.03, kidneys 0.08/0.02, milk 0.02 and eggs 0.01.

There are several obstacles to recommending reliable MRLs for fenpropimorph in animal products. One is the lack of data on sugar beet pulp and molasses, although the studies provided suggest that residues in the roots, if present, may not exceed 0.01 mg/kg. A more serious obstacle is the lack of conventional feeding studies.

The guidance on livestock transfer studies in the 1993 JMPR report states that such studies are required (1) when detectable residues are found in feed items and metabolism studies indicate that significant residues (>0.01 mg/kg) may occur in edible tissues, and generally (2) where significant residues (generally >0.1 mg/kg) occur in crops or commodities fed to animals. Both situations occur with fenpropimorph. The guidance allows the possibility of using metabolism studies to serve as transfer studies when only low residue levels (<0.1 mg/kg) are found in feed items, but this does not apply to fenpropimorph. The situation does not lend itself to waiving the need for transfer studies.

Another complicating factor is the indication in metabolism studies that fenpropimorph *per se* is unlikely to occur in animal products with the possible exception of hen kidneys. The studies indicate that low residues of metabolites may occur, especially in liver and kidneys. None of the analytical methods provided to the Meeting is suitable for animal products and none determines metabolites (except BF 421-2 in soil). The Meeting was advised that an analytical method for the determination of fenpropimorph *per se* in animal tissue is available but it was not provided. There would be little point in recommending limits for the parent compound in animal products when its residues would not normally be expected.

No method has been provided for enforcing limits based on a definition of the residue which includes the most likely animal metabolites BF 421-1 (hydroxy-fenpropimorph) BF 421-2 (fenpropimorph acid) and BF 421-3 (hydroxymethyl-fenpropimorph acid). The Meeting was informed that a method for the determination of BF 421-2 is expected to be completed by the end of 1995, but it has not yet been decided whether feeding trials employing this method will be conducted.

The Meeting concluded that the data were insufficient to estimate maximum residue levels for animal products. For that purpose conventional transfer studies are needed for cattle and poultry, following Codex guidance and with analyses for the parent compound and likely metabolites by validated methods.

Fenpropimorph residues were reduced by 20 and 60% in Washington navel oranges and Hernandina clementines respectively <u>held in storage</u> for 30 days at 5°C. In the processed fractions residues increased slightly in the pulp over the same period, remained the same in the albedo and decreased by 10 and 46% respectively in the peel.

A summary report of a <u>wheat processing</u> study suggests fenpropimorph losses of >30% when processing wheat grain containing 0.07 mg/kg into bran, wholemeal or white flour. In this and three other summary reports bread baked from grain with residues of 0.05 to 0.08 mg/kg was reported to contain residues of <0.05 mg/kg. The Meeting could not confirm the results reported for the processed products in the absence of the detailed processing procedures. Processing studies on wheat grain containing residues near the estimated maximum level would be desirable for a clear picture of the extent of residue reduction.

With regard to the <u>edible portions of food commodities</u>, cereal grains are the main items for which MRLS are recommended. Trials showed that median residues in grain are likely to be <0.05 mg/kg and maximum residues unlikely to exceed 0.5 mg/kg. As noted above, summary reports suggest that residues in processed grain fractions and bread are likely to be less than in grain and probably not detectable (<0.05 mg/kg) in bread baked from grain containing expected median residues. This needs to be confirmed.

Although no MRLs are recommended for beans, leeks or citrus fruits, the data suggest that residues may not exceed 0.5 mg/kg from approved uses in leeks or 0.1 mg/kg in fresh beans. Although data were not provided for estimating a maximum residue level for citrus, the available information indicates that residues in citrus pulp are likely to be less than 10% of the residue in the whole fruit.

No information was provided on residues in commodities in commerce or at consumption.

RECOMMENDATIONS

The Meeting estimated the maximum residue levels listed below, which are recommended for use as MRLs.

Definition of the residue: fenpropimorph

Commodity		Recommended MRL, mg/kg	PHI on which based (days)
CCN	Name		
GC 0640	Barley	0.5	30
GC 0647	Oats	0.5	30
GC 0650	Rye	0.5	30
GC 0654	Wheat	0.5	30
AS 0640	Barley straw and fodder dry	5	30
AS 0647	Oats straw and fodder dry	5	30
AS 0650	Rye straw and fodder dry	5	30
AS 0654	Wheat straw and fodder dry	5	30
VR 0596	Sugar beet	0.05*	7
AV 1051	Fodder beet leaves or tops (sugar beet leaves or tops)	1	7

^{*}At or about the limit of determination

FURTHER WORK OR INFORMATION

Desirable

- 1. Details (preferably in English) of the procedures used in the BASF wheat processing studies described in reports Nos. 79/10261, 79/10262, 79/10263 and 86/10411 for review at a future Meeting.
- 2. Conventional livestock and poultry feeding (transfer) studies with determination of fenpropimorph and the major metabolites identified in metabolism studies (e.g. BF 421-1, BF 421-2 and BF 421-3).
- 3. Validated analytical regulatory methods (including representative chromatograms) for the determination of fenpropimorph and its major metabolites in animal products.

- 4. Information on fenpropimorph residues in commodities in commerce or at consumption.
- 5. A study of the frozen storage stability of analytical samples of a root crop.
- 6. Validation of analytical methods used in the sugar beet trials.

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