FENPROPIMORPH (188)

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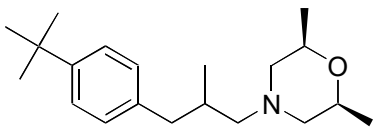
EXPLANATION

Fenpropimorph is a systemic morpholine fungicide for the control of various diseases, primarily in cereals but also finds use in controlling Sigatoka diseases in bananas. It acts by inhibiting the sterol pathway of fungus. It was first evaluated by JMPR in 1994 (T), 1995 （R）. The ADI for fenpropimorph was re-established as 0–0.004 mg/kg bw in 2016 and ARfDs of 0.1 mg/kg bw for woman of child-bearing age and 0.4 mg/kg bw for the general population established. Fenpropimorph was scheduled at the 48th session of the CCPR for the periodic evaluation of residue data by the 2017 JMPR.

The Meeting received information on the metabolism of fenpropimorph in lactating goats and laying hens, banana, sugar beet, barley and wheat, follow crops, methods of residue analysis, freezer storage stability, GAP information, supervised residue trials on bananas, sugar beet, barley, oats, rye and wheat as well as a livestock feeding study (lactating cow).

IDENTITY

Common name	fenpropimorph
Chemical name	
	IUPAC: (±)-*cis*-4-[3-(4-*tert*-butylphenyl)-2-methylpropyl]-2,6-dimethylmorpholine
	CAS: *cis*-4-[3-[4-(1,1-dimethylethyl)phenyl-2-methylpropyl]-2,6-dimethylmorpholine
Manufacturer's code numbers:	BAS 421 F, LAB 108 406, Reg.No. 108 406, ACR-3320, CGA 101031, Ro 12-3049
CAS number:	67564-91-4
CIPAC Code:	427
Structural formula:	



Molecular formula:	$C_{20}H_{33}NO$
Molecular mass:	305.48 g/mol

Fenpropimorph is a racemate, that is it consists of equal amounts of both enantiomers. The cis-dimethylmorpholine moiety has a plane of symmetry which renders it achiral, but the 1,3-disubstituted 2-methylpropane moiety between the carbocycle and the heterocycle is chiral.

Specifications

Specifications for fenpropimorph have not been developed by the FAO.

PHYSICAL AND CHEMICAL PROPERTIES (pure fenpropimorph 99.6% unless stated

otherwise)

Property	Results (method)	Reference
Appearance	Colourless liquids with faint aromatic odour	Daum 1999 11214
Melting point	-47 to – 41 °C (EEC A1. 1.4.4.2, DSC OECD 102)	Daum 1999 11214; Daum 1999 1008316
Boiling point	There is no endothermic effect which is unrelated to the melting range. Therefore, boiling of the test substance can be excluded. Decomposition is observed at *ca*. 310 °C. (EEC A2. 1.4.7, DSC OECD 103)	Daum 1999 11214; Daum 1999 1008316
Relative density	0.93g/cm³ at 20 °C (EEC A3. 1.4.4, Oscillation densitometer OECD 109) [99.2%]	Kästel 1994b 10395
Dissociation constant	The pKb value for fenpropimorph at 20 °C is 7.02 (pKa = 6.98 for fenpropimorph hydrochloride) (OECD 112) [99.4%]	Redeker 1988b 11671
Vapour pressure	0.0039 Pa at 20 °C (EEC A.4) [98.9%]	Kästel 2004 1016297
Volatility	Calculated Henry's law constant at 20 °C: 2.74×10^{-4} kPa m^3/mol	Ohnsorge 2004 1031205
Solubility in water (at 20 °C)	neutral range (purified water) is 4.3 mg/L, in the alkaline range (at pH 9-11) 3.5 mg/L and in the acid range (pH 4.4) 7.3 g/L (EEC A.6.1.4.1, column elution method OECD 105) [99.2%]	Redeker 1988a 11302, Redeker 1988c 11913
Solubility in organic solvents (20 °C)	dichloromethane 774.2 g/L methanol 789.2 g/L *n*-heptane 725.4 g/L ethyl acetate 778.0 g/L acetone 760.4 g/L toluene 764.6 g/L	Redeker 1992 11596
Partition coefficient *n*-octanol/water	The log P_{ow} values of the test substance at different pH values were calculated to be: 2.10 at pH 1 2.22 at pH 4 4.50 at pH 7 5.18 at pH 9 (EEC A.8.1 calculation for surface active substances according computer program ACD/LogP)	Ohnsorge 2004 1016476
Hydrolysis under sterile conditions	Stable at tested pHs 4, 7, 9 at 25 °C for 32 days (EPA guideline Subdiv. N, 161-1)	Rüdel 1988 0443
UV spectra	Molecular extinction coefficient (ε, L/mol/cm) at: 203 nm 1.1×10^4 219 nm 1.1×10^4 242 nm 2.1×10^2 264 nm 4.2×10^2 270 nm 3.2×10^2 272 nm 4.2×10^2	Türk 1996 10288
Photolysis	Based on UV spectrum, expected stable to aqueous photolysis	Sarafin 1991b 10344
Estimated photochemical oxidative degradation (calculation)	t½ 2.9 h (BBA guideline part IV, 6-1)	Sarafin 1991a 10301

Technical material (93.0%)

Property	**Results (method)**	**Reference**
Appearance	Colourless liquids with faint aromatic odour	Kästel 1994 10392
Surface tension	The surface tension of fenpropimorph technical is 49.0 mN/m at 0.5% (w/w) and 48.9 mN/m at 2.0% (w/w). (EEC A.5)	Kästel 1994 10392

Fenpropimorph is applied formulated alone or in combination with other active substances. It is formulated as emulsifiable concentrate (EC), suspo-emulsion (SE) and oil miscible liquid (OL) products.

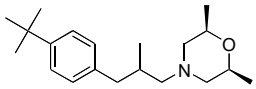
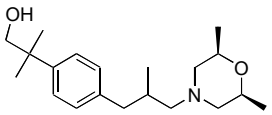
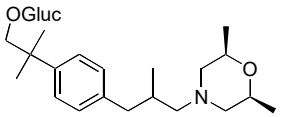
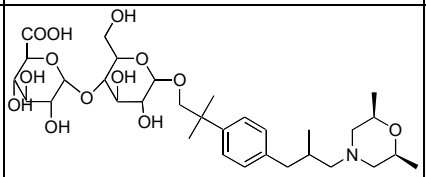
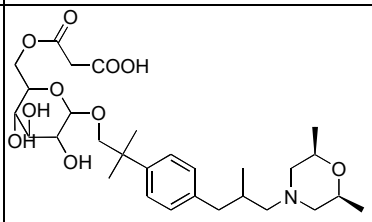
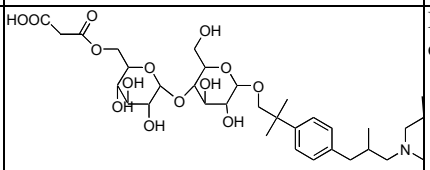
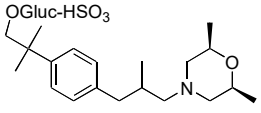
Formulation type	EC	OL	SE	SE	SE	SE	EC	SE	SE
Fenpropimorph	750	880	250	300	150	317	375	214.3	200
Epoxiconazole			84		125	83		42.9	62.5
Kresoxim-methyl				150	125	83			

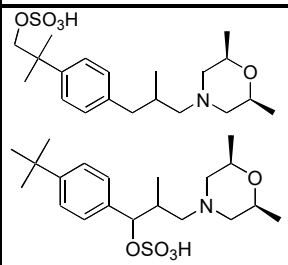
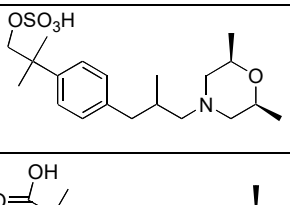
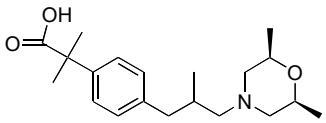
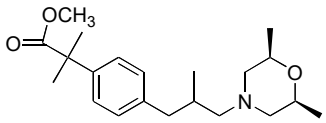
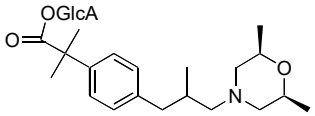
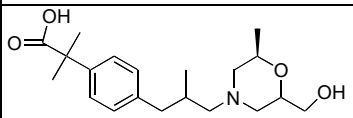
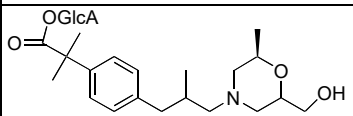
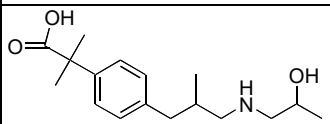
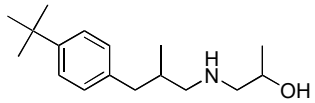
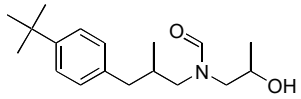
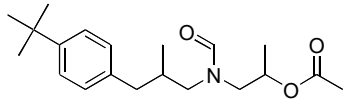
Formulation type	EC	OL	SE	SE	SE	SE	EC	SE	SE
Pyraclostrobin							100	114.3	
Metrafenone									75

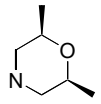
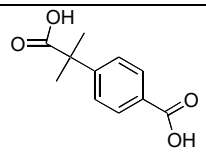
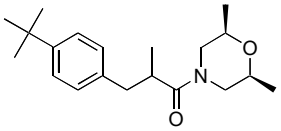
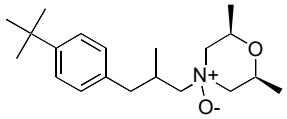
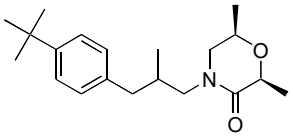
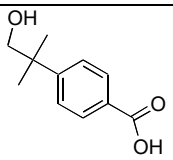
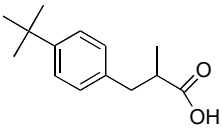
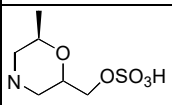
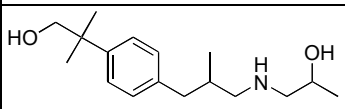
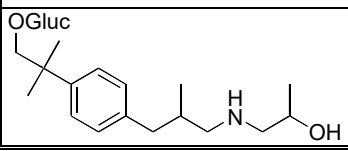
METABOLISM AND ENVIRONMENTAL FATE

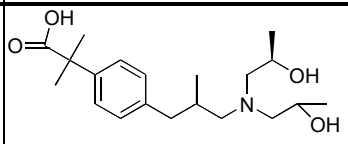
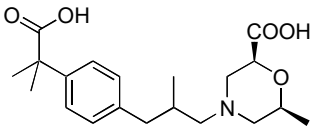
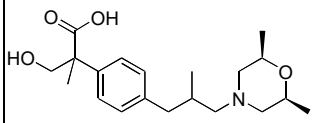
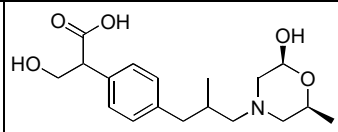
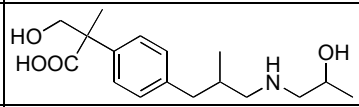
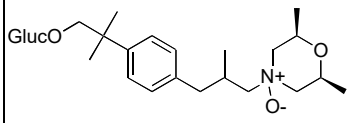
The metabolite summary table provides a reference for the numbering scheme used in the current evaluation (Table 1).

Table 1 Degradation compounds from metabolism of fenpropimorph in plants, animals and the environment

Code Numbers		Description		
Substance/ Metabolite Code	Reg. No.	Chemical Name CAS-No.	Structure	Compound found in
Fenpropimorph BAS 421 F	108406	(±)-cis-4-[3-(4-tert-butylphenyl)-2-methylpropyl]-2,6-dimethylmorpholine 67564-91-4		Hen Goat sugar beet banana wheat rotational crop rat
BF421-1	246719	2-(4-{(2*RS*)-3-[(2*R*,6*S*)-2,6-dimethylmorpholin-4-yl]-2-methylpropyl}phenyl)-2- methylpropan-1-ol		Hen Goat sugar beet wheat rotational crop rat
BF421-1 glucoside				Sugar beet Wheat Rotational crop
BF421-1 diglucoside				Sugar beet wheat
BF421-1 malonylglucoside				Sugar beet Wheat Rotational crop
BF421-1 malonyldiglucoside				Rotational crops
BF421-1 glucoside sulphate				Sugar beet

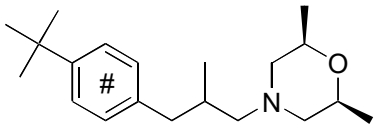
Code Numbers		Description		
Substance/ Metabolite Code	Reg. No.	Chemical Name CAS-No.	Structure	Compound found in
BF421-1-sulfate				Rat Goat
				goat
BF421-2	231346	2-(4-{(2*RS*)-3-[(2*R*,6*S*)-2,6-dimethylmorpholin-4-yl]-2-methylpropyl}phenyl)-2-methylpropanoic acid 121098-45-1		Hen Goat Wheat rat
BF421-2 methyl	168176	2-methyl-2-[4-[2-methyl-3-(cis-2,6-dimethylmorpholine-4-yl)propyl]phenyl)propionic acid methylester		Wheat (tentative, not distinguishable from BF421-15 in chromatography systems used)
BF421-2 glucuronide				Goat
BF421-3	5682525	2-(4-{(2*RS*)-3-[(2*R*,6*R*)-2-(hydroxymethyl)-6-methylmorpholin- 4-yl]-2-methylpropyl}phenyl)-2-methylpropanoic acid		Hen Goat Rat
BF421-3 glucuronide (or isomer)				Goat
BF421-4	235366	2-methyl-2-{4-[2-methyl-3-(2-hydroxypropyl)aminopropyl]phenyl}propionic acid		Rat
BF421-7	4994030	1-{[3-(4-tert-butylphenyl)-2-methylpropyl]amino}propan-2-ol		sugar beet wheat rat
BF421-7 formic acid adduct		Possibly a non-biotic artifact formed after extraction		wheat
BF421-7 formylated and acetylated derivative		Possibly a non-biotic artifact formed after extraction		Sugar beet Wheat

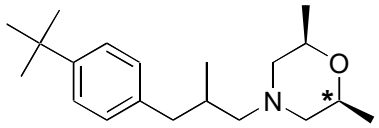
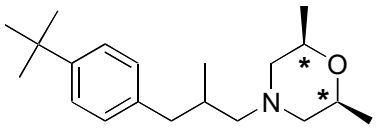
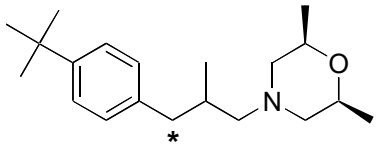
Code Numbers		Description		
Substance/ Metabolite Code	Reg. No.	Chemical Name CAS-No.	Structure	Compound found in
BF421-10	224399	(2R,6S)-2,6-dimethylmorpholine 141-91-3		Hen Goat sugar beet wheat rotational crop rat
BF421-12		4-(2-carboxypropan-2-yl)benzoic acid		hen
BF421-13	131051	4-[3-(4-tert-butylphenyl)-2-methyl-1-oxopropyl]-2,6-dimethylmorpholine 162821-88-7		wheat
BF421-14	234616	4-[3-(4-tert-butylphenyl)-2-methylpropyl]2,6-dimethylmorpholine N-oxide		sugar beet wheat
BF421-15	264957	4-[3-(4-tert-butylphenyl)-2-methylpropyl]-2,6-dimethylmorpholine-3-one		Wheat (tentative, not distinguishable from BF421-2-methyl in chromatography systems used)
BF421-16		4-(1-hydroxy-2-methylpropan-2-yl)benzoic acid		Hen rat
BF421-18				hen
BF421-19				goat
BF421-20 / BF421-55 [1]		2-{4-[(2RS)-3-{[(2RS)-2-hydroxypropyl]amino}-2-methylpropyl]phenyl}-2-methylpropan-1-ol		sugar beet wheat rat
BF421-20 glucoside				Sugar beet wheat

Code Numbers		Description		
Substance/ Metabolite Code	Reg. No.	Chemical Name CAS-No.	Structure	Compound found in
BF421-21				goat
BF421-22				Goat
BF421-24				Goat rat
BF421-26				Goat
BF421-30				Goat
BF421-36 glucoside		2-(4-{(2*RS*)-3-[(2*RS*,6*RS*)-2,6- dimethyl-4-oxidomorpholin-4-yl]-2-methylpropyl}phenyl)-2- methylpropyl D-glucopyranoside		sugar beet wheat

The Meeting received studies on the metabolism of fenpropimorph in plants (banana, sugar beet, barley and wheat), laboratory animals (rats) as well as lactating goats and laying hens. The metabolism of fenpropimorph in plants and animals was investigated using [phenyl-U-^{14}C]-fenpropimorph, [morpholine-2,(6)-^{14}C]-fenpropimorph, [morpholine-2,6-^{14}C]-fenpropimorph and [benzylic-^{14}C]-fenpropimorph. The structural formula and the positions of the ^{14}C label are shown below. The studies on rats were evaluated by the WHO Core Assessment Group.

Table 2 Location of labels in compounds used in metabolism and environmental studies

Study	Label positions
Sugar beet (Rabe and Gläßgen 2005) Goat (Ritter 1989) Goat (Leibold and Hoffmann 2000) Hen (Ritter 1989) Hen Goat (Leibold and Hoffmann 2000) Rotational crop (Rabe 2003)	 [phenyl-U-^{14}C]-fenpropimorph

Study	Label positions
Sugar beet (Hamm 2000) Sugar beet (Rabe and Gläßgen 2005) Goat (Leibold and Hoffmann 2000) Hen (Leibold and Hoffmann 2000) Rotational crop (Rabe 2003)	 [morpholine-2,(6)-^{14}C]-fenpropimorph
Wheat (Huber 1979) Banana (Hamm 1995) Goat (Ritter 1989) Hen (Ritter 1989)	 [morpholine-2,6-^{14}C]-fenpropimorph
Barley (Pryde and Etterli 1979)	 [benzylic-^{14}C]-fenpropimorph

The identification of residue components in the animal and plant metabolism studies was achieved using, where available, authentic standards of the compounds involved and in some cases by ^{13}C-NMR and MS-spectroscopy. Additional techniques, such as hydrolysis, derivatization and enzymatic degradation, were used in many cases to aid in characterizing metabolites. There was little change in metabolite profiles analysed shortly after samples were collected and at the end of the analytical phase suggesting the results of the studies reviewed are representative of the metabolites present.

Plant metabolism

The Meeting received plant metabolism studies with fenpropimorph on three different crop groups using foliar application treatments. The crops studied are representative of tropical fruit (banana), cereals (wheat, barley), and root and tuber vegetables (sugar beet).

Some scientific papers were also located in the literature that are relevant to plant metabolism.

As noted earlier, fenpropimorph consists of one pair of enantiomers. The stereoisomers of chiral pesticides may show selective metabolism in plants which can result in residues with a stereoisomer composition different from that in the applied product. Different stereoisomers can also exhibit different toxicological properties. It has been reported that the (-)-enantiomer of fenpropimorph exhibits a higher fungicidal activity than the (+)-enantiomer (Himmele and Pommer 1980, Pommer 1984, Baloch and Mercer 1987). The (-)-enantiomer was also reported to be more active against mildew and brown rust of wheat than the (+)-enantiomer (Himmele and Pommer 1980, Pommer 1984).

Buerge *et al.* 2016 studied the stereoisomer composition of fenpropimorph residues following application to sugar beet and wheat crops.

Sugar beet crops (mixed variety Amalia and Ribera) were treated at BBCH 45 with a single spray of fenpropimorph at 0.3 kg ai/ha. The residues of fenpropimorph in leaves decreased from 1.6 mg/kg one day after application to 0.11 mg/kg at harvest, 77 days after application (Figure 1a). Metabolism of fenpropimorph in leaves was enantioselective in sugar beet. The enantiomer

composition changed rapidly within the first days (Figure 1b), the enantiomer fraction (EF) differed slightly from racemic at 20 h after application (EF 0.45), decreasing to an EF of 0.25 on day 11 where after the enantiomer composition in leaves remained more or less constant (77 days, EF ≈0.3).

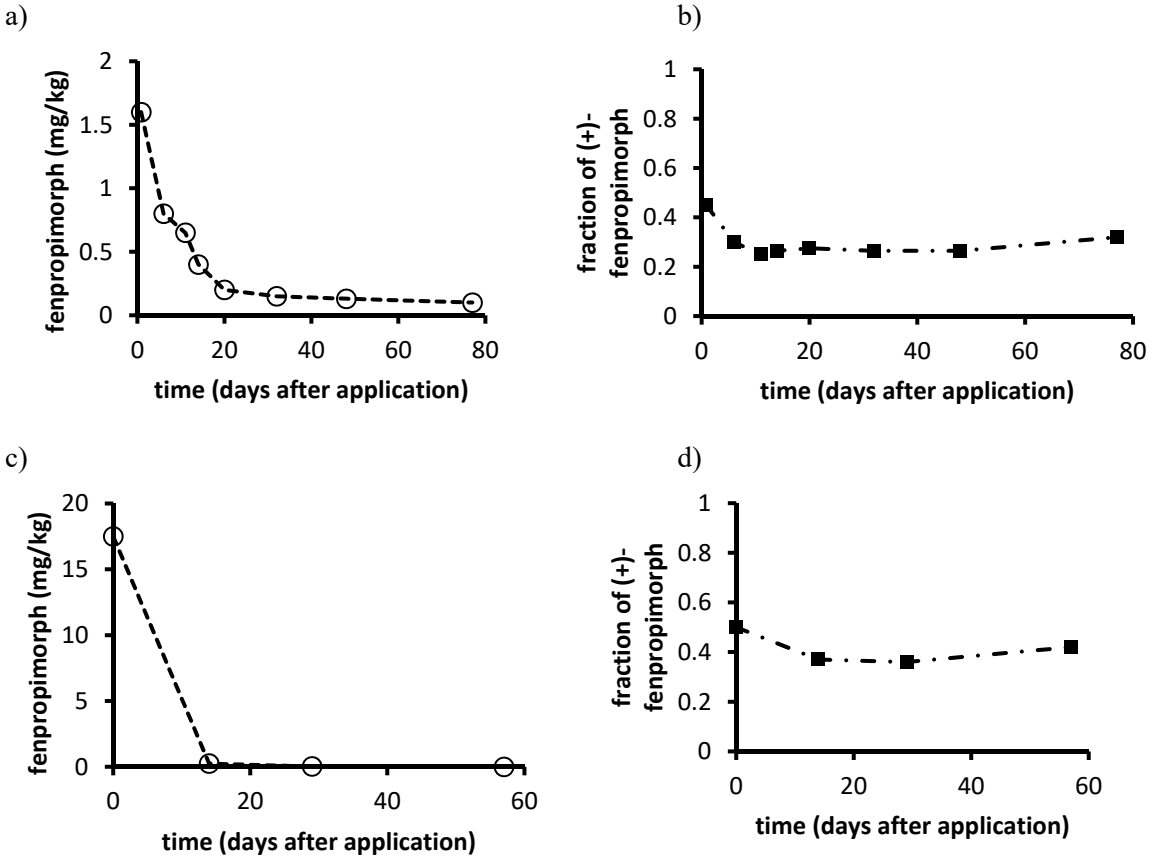


Figure 1 Fenpropimorph residues following foliar treatment of a) sugar beet residues, b) sugar beet enantiomer fraction, c) wheat residues and d) wheat enantiomer fraction

The change in enantiomer composition could have been the result of an interconversion of enantiomers rather than preferential metabolism, however in experiments with pure enantiomers applied to sugar beet leaves, no formation of the opposite enantiomer was observed. This suggests that the enantioselectivity observed was due to different rates of metabolism and not the result of interconversion of enantiomers.

In a separate experiment fenpropimorph was applied to spring wheat (variety Fiorina) as a single spray at 0.4 kg ai/ha and at BBCH 31, the earliest growth stage of wheat for the intended use against powdery mildew. The plants were about 30 cm high and then grew to 60 and 90 cm, 14 and 57 days after application, respectively, the rapid growth contributing to the decline in residues (Figure 1c). In the case of wheat leaves, weak enantioselectivity was observed (Figure 1d). The metabolism in forage was slightly enantioselective at day 14, however, in the following days, the enantiomer composition did not change much (57 days, EF≈0.4).

In both sugar beet and wheat, the (+)-isomer is more rapidly metabolised than the fungicidally more active (-)-isomer.

The rate of penetration of fenpropimorph into plant leaves has been investigated by Smelt *et al.* 1997 who studied the volatilization rates of fenpropimorph following application to sugar beet using micrometeorological methods. The highest rates (1.3–3.0% per hour) were measured in the first

hours after application. The rate gradually declined to less than 0.01% per hour by the sixth day after application (Figure 2a).

The residues on sugar beet leaves declined gradually from 68% calculated dosage one hour after application to 12.8% at six days after application (Figure 2b). The rinsability of the residues on the leaves was tested by sequential rinsing's of whole leaves with water, methanol and chloroform.

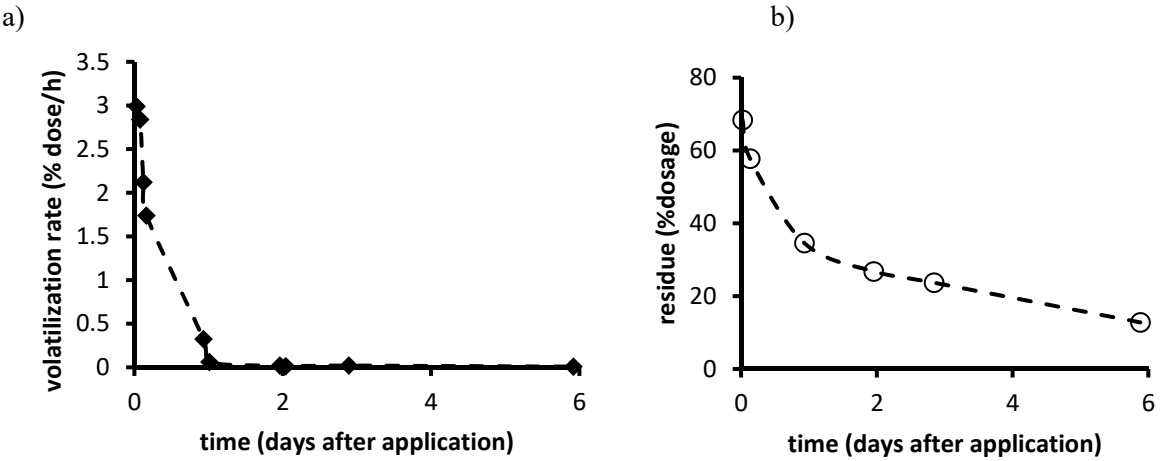


Figure 2 Sugar beet a) volatilisation of fenpropimorph following foliar application and b) residues

On the day of application, about 4 h after spraying, the greatest fraction of the residue of fenpropimorph was removed with ethanol rinsing (Table 3). This indicates that the pesticide had not much penetrated into the wax layer of the leaves. Six days later, the residue was mainly rinsed with chloroform, indicating a high penetration of fenpropimorph into the leaf surfaces.

Table 3 Rinsability of residues from leaf surfaces by solvents of decreasing polarity

Days after application		Surface wash			
		Water	Methanol	Chloroform	Total
0.16 d	Mass fenpropimorph (mg)	13.3	32.3	7.2	
	%surface wash	21.5	61.2	13.7	100
6.1 d	Mass fenpropimorph (mg)	0.24	0.91	8.3	
	%surface wash	2.5	9.6	87.9	100

By at least six days after application, most of the fenpropimorph had penetrated into the leaf wax layers. The decrease in volatilisation rate with time suggests penetration of the majority of fenpropimorph into the wax layer may have occurred by one day after application.

Foliar applications

Banana

Hamm (1995a 10709, 1995b 10710) conducted a metabolism study with [^{14}C]-fenpropimorph on banana plants (variety Gran Naine) maintained outdoors on a banana plantation in Costa Rica. ^{14}C-Fenpropimorph-[morpholine-2,6-^{14}C] or –[phenyl-U-^{14}C] was applied four times as an EC-formulation together with spray oil at an application rate of 0.9 kg ai/ha. For one plant, the first application was made before the bunch was protected by a plastic bag while for the next three applications the bunch was covered with a plastic bag (bagged). With a second plant, the bunch was unprotected (unbagged) for all four applications. The interval between the first and last application was 77 days with the interval between sprays 14, 51 and 12 days. The spray solution was applied to the top of the plants about 4.8 m above the ground in a spray volume equivalent to 25 L/ha. Samples

(fruits and leaves) were taken one day after the last application. Banana fruits from the upper, middle and lower parts of the bunches were taken, furthermore, the 1st, 3rd, 5th and 7th leaf of both plants and the 2nd leaf of second plant (unbagged). A sub-sample of the unripe bananas was ripened by acetylene treatment at 9–15 °C before freezing. Residues in fruit were extracted by Bleidner distillation to identify the ^{14}C in peel and pulp by radio HPLC and LC-MS.

Additionally, ^{14}C-fenpropimorph in unripe and acetylene-ripened fruits was extracted by use of a Bleidner distillation.

Samples of fruit from the above study were further characterised by Hamm 1997a (10536). Fruit was extracted with methanol. The extracted radioactivity was characterized and quantified by radio-HPLC. In addition, liquid/liquid partitioning experiments using dichloromethane were carried out. For further characterization of ^{14}C remaining in solids after methanol extraction, samples were further extracted with water and afterwards subjected to an enzymatic cleavage, e.g. with amylase, pectinase and cellulase. A liquid/liquid partition was carried out with the water phase and dichloromethane. For further characterization of the polar compound in the water phase, a fermentation with fresh baker's yeast was conducted to convert sugars present to ethanol. The ^{14}C-residue (^{14}C-starch) was treated with an amyloglucosidase to isolate ^{14}C-glucose. The enzymatic work was only carried out with the fruit from the M-label experiment, as ^{14}C levels for the P-label experiment were too low. Samples were analysed within 565 days of collection (harvest 2/09/1994, last extract analysed 20/03/1996).

The TRR in fruit from the M-label experiment (0.317–0.669 mg eq/kg) was always higher than that for the P-label (0.025–0.105 mg eq/kg). A comparison between ^{14}C extracted from bagged (protected) and unbagged (unprotected) fruits showed that the ratio of Bleidner extracted residue is almost identical between peel and pulp of both bagged and unbagged treatment groups (Table 5).

Table 4 TRRs (mg eq/kg) in leaves 1 day after 4 treatments of a banana plants with ^{14}C-fenpropimorph

Sample	M-label		P-label	
	Bunch protected (bagged) (mg eq/kg)	Bunch unprotected (mg eq/kg)	Bunch protected (bagged) (mg eq/kg)	Bunch unprotected (mg eq/kg)
leaf No. 1	48.8	85.7	101	101
leaf No. 2	165	-	-	-
leaf No. 3	17.4	168	100	124
leaf No. 5	129	97	70.1	110
leaf No. 7	150	150	142	75.7

Table 5 TRRs (mg eq/kg) in fruit 1 day after 4 treatments of a banana plants with ^{14}C-fenpropimorph

Sample	M-label				P-label	
	Bagged (mg eq/kg)	Extracted Bleidner (%TRR)	Unbagged (mg eq/kg)	Extracted Bleidner (%TRR)	Bagged (mg eq/kg)	Unbagged (mg eq/kg)
pulp, unripe	0.502	1.8	0.944	2.1	0.020	0.037
peel, unripe	0.197	49.4	0.404	48.1	0.043	0.147
pulp, ripe	0.407	2.5	0.734	4.8	0.020	0.036
peel, ripe	0.309	29.9	0.471	22.6	0.046	0.260

Unripe bananas were ripened by acetylene treatment at 9-15 °C

In fruits, the solvent extractability depended on the label and the degree of fruit maturity. The extraction behaviour is summarized below (Table 6).

Table 6 Characterization of TRR in banana fruit 1 DALA (Hamm 1997a 10536)

	M-label TRR	extracted MeOH (%TRR) Total	Organo-soluble	Water-soluble	unextracted (%TRR)	P-label TRR	extracted MeOH (%TRR)	unextracted (%TRR)
Unbagged								
peel, unripe	0.416	59.8	*38.0*	*19.4*	32.8	0.218	82.4	16.2
pulp, unripe	0.791	11.4	*2.5*	*8.2*	85.3	0.042	65.8	35.6
whole fruit, unripe	0.669	27.3			68.2	0.105	71.8	28.7
peel, ripe	0.412	82.7	*32.2*	*53.8*	19.9	0.192	82.0	14.9
pulp, ripe	0.734	82.8	*3.5*	*85.1*	14.4	0.025	92.7	13.9
whole fruit, ripe	0.610	82.7			16.5	0.094	88.3	14.4
Bagged								
peel, unripe	0.183	54.5	*29.7*	*23.8*	43.0	0.038	74.2	19.3
pulp, unripe	0.432	12.4	*3.3*	*9.7*	83.5	0.018	35.3	73.5
whole fruit, unripe	0.345	27.1			69.4	0.025	48.6	54.9
peel, ripe	0.221	80.8	*18.8*	*59.5*	23.5	0.040	77.8	17.7
pulp, ripe	0.376	67.7	*3.2*	*62.7*	34.9	0.017	88.8	15.7
whole fruit, ripe	0.317	72.7			30.6	0.026	84.4	16.5

In whole fruit, methanol extracted ^{14}C ranged from 27 and 83% TRR for the M-label, whereas the corresponding values varied between 49 to 88% TRR for the P-label.

Methanol extracts from experiments with the P-label showed only one prominent fraction on HPLC analysis identified as fenpropimorph whereas extracts from M-label experiments showed two major fractions, fenpropimorph and a polar one identified by acetylation and HPLC as the natural assimilation products glucose, fructose and saccharose and shown by fermentation with baker's yeast to produce alcohol. Identified components are summarised in Tables 7 and 8.

Solids remaining after methanol extraction of pulp of unripe fruit from the morpholine experiment contained about 83% TRR and were subjected to enzymatic cleavage using aminoglucosidase, which released ^{14}C-glucose (about 65% TRR) from insoluble starches.

Table 7 Summary of the identified components in banana samples (1 DALA) after treatment with ^{14}C-fenpropimorph M-label (all expressed on a whole fruit basis)

	Unbagged		Bagged	
	Unripe	Ripe	Unripe	Ripe
Peel TRR (mg eq/kg)	0.081	0.131	0.035	0.068
	%TRR			
Extracted (CH_3OH)	19.5	31.8	19.0	30.9
fenpropimorph	14.2	2.0	9.4	3.1
sugars [A]	4.4	26.4	7.2	22.9
unknowns	0.9 (n=2, 0.4-0.5)	3.4 (n=2, 1.0-2.4)	2.4 (n=4, 0.3-0.9)	4.8 (n=4, 0.2-2.8)
PulpTRR (mg eq/kg)	0.061	0.373	0.035	0.157
	%TRR			
Extracted (CH_3OH)	7.7	50.8	6.8	41.9
fenpropimorph	1.3	1.2	1.3	1.3
sugars [A]	5.2	49.3	6.8	40.5
unknowns	1.2 (n=5, 0.1-0.4)	0.4 (n=1)		
Whole fruit (mg eq/kg)	0.142	0.504	0.070	0.226
	%TRR			
Extracted (CH_3OH)	27.3	82.7	27.1	72.6
fenpropimorph	15.5	3.2	10.7	4.4

	Unbagged		Bagged	
	Unripe	Ripe	Unripe	Ripe
Peel TRR (mg eq/kg)	0.081	0.131	0.035	0.068
	%TRR			
sugars [A]	9.7	75.7	14.0	63.4
unknowns	2.1 (n=7, 0.1-0.5)	3.8 (n=3, 0.4-2.4)	2.4 (n=4, 0.3-0.9)	4.8 (n=4, 0.2-2.8)

[A] tentatively assigned to glucose based on acetylation and HPLC of fraction in pulp and a comparison of the retention time of fractions in peel compared to pulp.

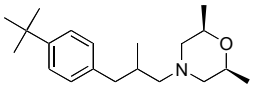
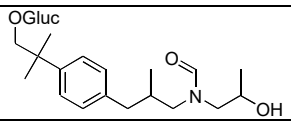
Table 8 Summary of the identified components in methanol extracts of banana samples (1 DALA) after treatment with ^{14}C-fenpropimorph P-label (all expressed on a whole fruit basis)

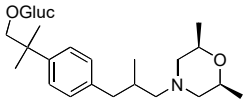
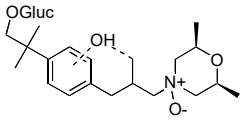
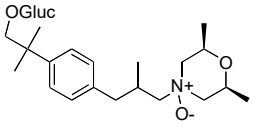
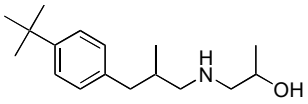
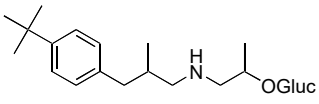
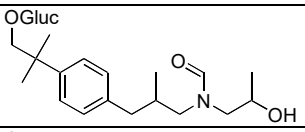
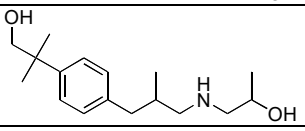
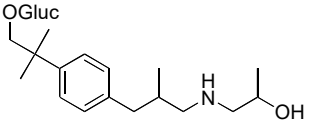
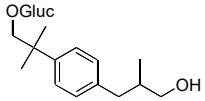
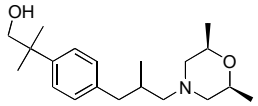
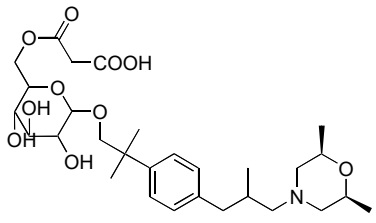
	Unbagged		Bagged	
	Unripe	Ripe	Unripe	Ripe
Peel TRR (mg eq/kg)	0.064	0.066	0.009	0.013
	%TRR in whole fruit			
Extracted (CH_3OH)	29.5	33.9	25.4	31.5
fenpropimorph	22.2	16.8	11.5	9.0
unknowns	7.3 (n=4, 0.8-3.2)	17.1 (n=6, 0.9-5.9)	13.9 (n=7, 0.7-4.1)	22.5 (n=7, 0.6-5.8)
Pulp TRR (mg eq/kg)	0.018	0.014	0.004	0.010
	%TRR in whole fruit			
Extracted (CH_3OH)	42.5	54.4	23.2	53.2
fenpropimorph	38.3	18.1	23.2	4.7
Unknowns	4.2 (n=2, 1.9-2.3)	36.3 (n=2, 11.0-25.3)		48.5 (n=3, 3.9-40.2)
Whole fruit (mg eq/kg)	0.142	0.08	0.013	0.023
	%TRR in whole fruit			
Extracted (CH_3OH)	72.0	88.2	48.6	84.7
fenpropimorph	60.5	34.9	34.7	13.7
unknowns	11.5 (n=4, 0.8-5.1)	53.3 (n=7, 2.2-25.3)	13.9 (n=5, 0.7-4.1)	71.0 (n=9, 0.6-40.2)

The morpholine ring can be opened during metabolism resulting in ^{14}C-fragments that can be utilised for biosynthesis of sugars in leaves. The sugars are subsequently transported to fruit for storage as starch. Water-insoluble ^{14}C-starch in fruit accounted for about 65% TRR in unripe unbagged bananas. During the acetylene ripening process, the ^{14}C-starch is enzymatically hydrolysed to mono- and di-saccharides accounting for up to 76% TRR in ripened fruit. The hydrolysis of starch to simple sugars is considered to be the reason for the significantly higher proportion of ^{14}C extracted using methanol and on partitioning of the extract with dichloromethane, the higher water-soluble portion in ripened fruits compared to unripe fruits.

Banana leaves contained much higher residue levels compared to fruit. Extracted ^{14}C from banana leaves were also characterised with a range of metabolites identified, but as the residues are not considered relevant for risk assessment, they were not quantified. Table 9 lists the metabolites identified in banana leaves for the experiments with the two labels.

Table 9 Summary of identified components in banana leaves after treatment with ^{14}C-fenpropimorph; morpholine and phenyl label

Metabolite code (Reg. No.)	Metabolite identity	M-label	P-label
Fenpropimorph		ident.	ident
Metabolite 1 (the metabolite is most likely an adduct of Met. 7 and formic acid from the HPLC solvent)		ident.	
Dimethylmorpholine = BF421-10		ident.	

Metabolite code (Reg. No.)	Metabolite identity	M-label	P-label
BF421-1-glucoside		ident.	ident
			ident.
Metabolite 3			ident.
Metabolite 4			ident.
BF421-7 glucoside			ident.
Metabolite 6 (the metabolite is most likely an adduct of Met. 7 and formic acid from the HPLC solvent)			ident.
Metabolite 7			ident.
Metabolite 8			ident.
Metabolite 9			ident.
Metabolite 10			ident.
BF421-1 malonylglucoside			ident.

In summary, metabolism of ^{14}C-fenpropimorph in banana proceeds with hydroxylation of the *t*-butyl at the phenyl ring, followed by glucosidation. The morpholine ring can be degraded with its C_1 fragments utilised during assimilation processes in the plant for the biosynthesis of carbohydrates. A summary is shown in Figure 3.

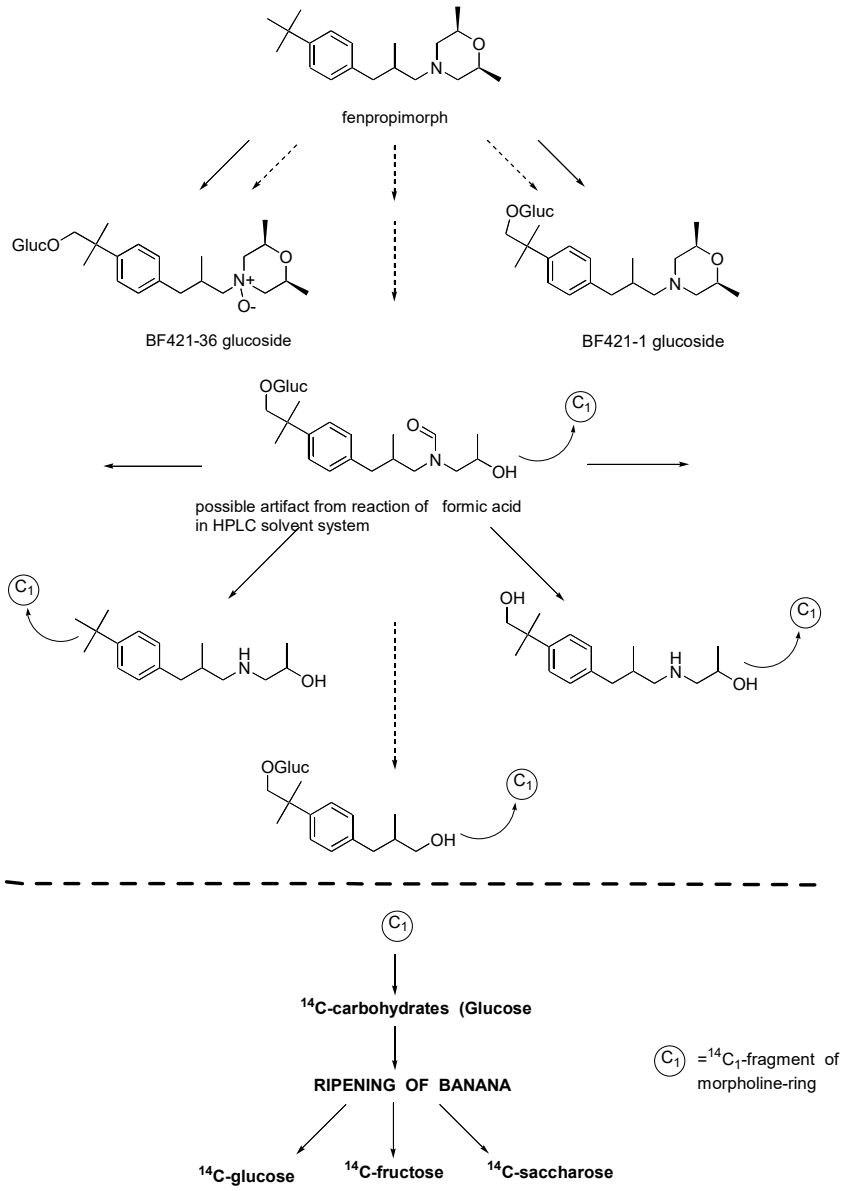


Figure 3 Metabolic pathway of fenpropimorph in bananas

Sugar beet

Hamm 2000a (1018643) studied the uptake of [morpholine-2,(6)-^{14}C]–fenpropimorph applied in an EC formulation to sugar beet plants (variety: Victoria) as two post-emergence applications, each at an application rate of 0.15 kg ai/ha in a spray volume of 291 L/ha. The plants were maintained in a vegetation hall with a glass roof. The first application was performed about 6 weeks after sowing (growth stage 39) and the second 32 days before harvest. Samples of leaves and roots were collected just before, after the second application, and at harvest.

The samples were extracted with methanol (3×) and in the case of leaves, additionally with aqueous ammonium solution. Liquid-liquid partitions were carried out with dichloromethane/water and ethyl acetate/water in order to classify the extracted radioactivity into organo- and water-soluble. For the determination of the nature of the radioactivity in the samples, aliquots of the methanol extracts and, if possible, from the organic and aqueous phases after partition were analysed by radio-HPLC. Metabolite identification was performed by comparison with reference substances or by LC-MS. The polar phase was further characterised by incubation with β-glucosidase at 37 °C for several

hours to release glucose from conjugates. Aliquots of post-extraction solids (PES) from leaf samples were incubated with β-glucosidase or extracted with NaOH or HCl. Additionally, an aliquot of the PES was refluxed with 10% NaOH for 3 h and centrifuged and the remaining residue (cellulose) washed while the water and NaOH supernatants were combined and acidified with concentrated HCl to precipitate the lignin.

TRRs were lowest in roots (0.026 to 0.034 mg eq/kg) and the highest in leaves (0.596 to 1.885 mg eq/kg) demonstrating limited translocation from treated leaves into the roots.

Methanol extracted > 70% of the ^{14}C in roots (70–79% TRR) and >79% from leaves (79–92% TRR) (Table 10).

Table 10 Extractability of ^{14}C in sugar beet roots and leaves using methanol (Hamm 2000a 1018643)

Sample	roots			leaves		
	TRR (mg eq/kg)	Extracted CH_3OH (%TRR)	Unextracted (%TRR)	TRR (mg eq/kg)	Extracted CH_3OH (%TRR)	Unextracted (%TRR)
65 DALA 1st	0.026	69.6	30.4	0.596	78.8	21.2
0 DALA 2nd	0.029	79.1	20.9	1.885	92.4	7.6
32 DALA 2nd	0.034	75.4	24.6	1.448	84.9	15.0

HPLC analysis of the extracts showed that in roots the major fraction of ^{14}C structurally related to fenpropimorph was the parent compound. In leaves, three further metabolites could be isolated and identified, BF421-1 glucoside (BF421-1-gluc), BF421-1 diglucoside and BF421-1 glucoside sulphate. The identified metabolites are listed in Table 11.

Table 11 Summary of the identified components in sugar beet samples after treatment with ^{14}C-fenpropimorph M-label (Hamm 2000a 1018643)

	roots			leaves		
	65 DALA 1st	0 DALA 2nd	32 DALA 2nd	65 DALA 1st	0 DALA 2nd	32 DALA 2nd
TRR (mg eq/kg)	0.026	0.029	0.034	0.596	1.885	1.448
			%TRR			
Extracted (CH_3OH)	61.7	73.8	68.9	79.6	89.7	84.5
fenpropimorph	5.9	40.2	14.4	18.0	70.2	15.2
BF421-1 glucoside				3.6		10.3
BF421-1 diglucoside				14.8	4.8	13.7
BF421-1 glucoside sulphate			4.6	21.9	2.8	15.9
Glucose [A]	55.8[C]	33.6[C]	48.1[C]	8.9	6.0	(4.3[B])
Polar unknowns				2.2 (n=2)		14.3 (n=5)
Medium polarity unknowns				10.3 (n=4)		12.4 (n=5)
Non-polar unknowns			1.4 (n=1)		1.6 (n=2)	2.6 (n=4)
unidentified/uncharacterised					4.2	
Unextracted	30.4	20.9	24.6	21.2	7.6	17.2
NH_3 extract				6.7	2.8	5.7
cellulose				2.4	1.0	2.2
Lignin solid				1.4	0.5	1.2
Lignin liquid				8.6	3.1	6.7
Total	92.1	94.7	93.5	100.8	97.3	101.7

[A] Includes glucose identified in precipitate from methanol phase.

[B] The 4.3% was detected in the dichloromethane partition and is not included in the total

[C] remaining after evaporation of CH_3OH, so represents that ^{14}C processed and available for addition to the columns

The residues unextracted by methanol in roots were low (< 0.01 mg eq/kg), and not further characterized.

In leaves the ^{14}C unextracted using methanol reached 0.126–0.218 mg eq/kg (7.6–21.2% TRR), depending on the sampling time. On ammonia extraction, an additional portion could be released, 2.8–6.7% TRR with a further extraction by NaOH releasing and additional 3.4–9.4% TRR.

The additional portions released by more severe treatments suggest some of the ^{14}C is incorporated in, or associated with, natural products such as cellulose and lignin.

In summary, metabolism of ^{14}C-fenpropimorph in sugar beets proceeds with hydroxylation of the dimethylethyl group of the phenyl ring, followed by glucosidation. The presence of polar metabolites (likely to be glucose and further carbohydrates) indicate that the morpholine ring can be degraded with its C_1 fragments utilised during assimilation processes in the plant for the biosynthesis of carbohydrates.

Compounds identified in the metabolism of fenpropimorph in sugar beet are presented in Figure 4.

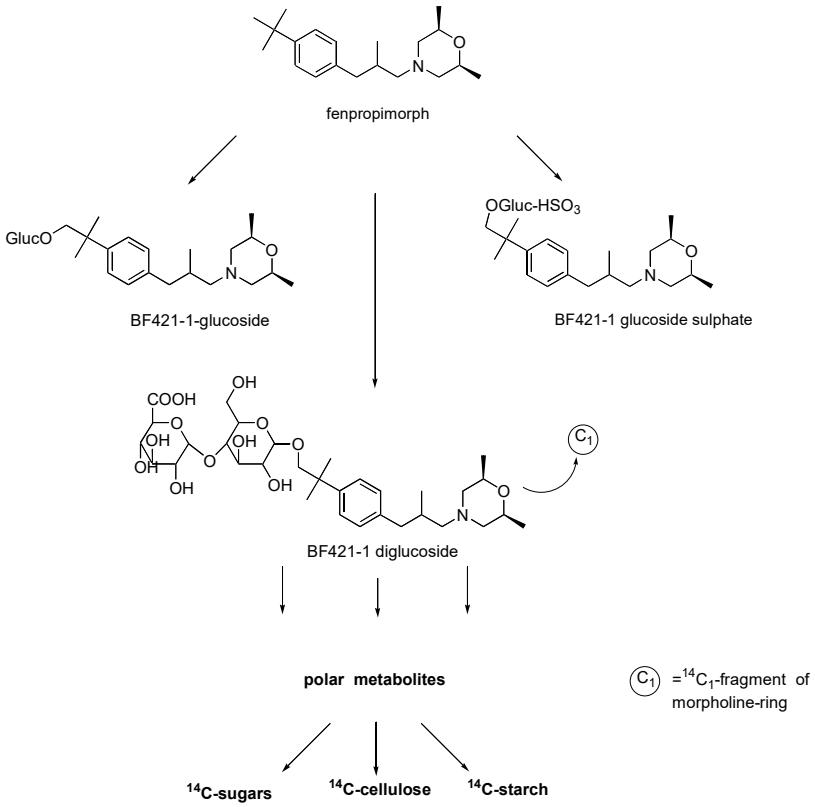


Figure 4 Metabolites of fenpropimorph in sugar beets

In a separate study Rabe and Gläßgen (2005a 1021389) studied the metabolism study for ^{14}C fenpropimorph in sugar beet using [morpholine-2,(6)-^{14}C]- or [phenyl-U-^{14}C]-fenpropimorph. The active substance was applied as an EC formulation to sugar beet plants growing in a **glass house** at an application rate of 1×750 g ai/ha. Samples of sugar beet plants were taken on the day of application and at harvest 112 days after treatment, where the mature plants were separated into leaf and root. All samples were stored at approximately -18 °C during the course of the study.

As expected, the highest levels of TRRs were found in sugar beet plants sampled on the day of application (55.2 mg eq/kg for the phenyl label, 47.2 mg eq/kg for the morpholine label). The TRR values for sugar beet leaves harvested 112 DALA amounted to 0.97 mg eq/kg for the phenyl label or 0.52 mg eq/kg for the morpholine label. In sugar beet root (112 DALA) the ^{14}C levels were much lower, accounting for 0.03 mg eq/kg (phenyl label) to 0.04 mg eq/kg (morpholine label).

The extractability of ^{14}C with methanol was good for sugar beet leaf (92.6 or 88.9% TRR) and quite good for sugar beet root (76.0 or 63.8% TRR) (Table 12).

Table 12 Extraction of radioactivity after treatment of sugar beet with ^{14}C-fenpropimorph

	P-label	M-label

	TRR (mg eq/kg)	Extracted MeOH (%TRR)	Unextracted (%TRR)	TRR (mg eq/kg)	Extracted MeOH (%TRR)	Unextracted (%TRR)
Plant 0 DALA	55.2			47.2		
Leaves at harvest (112 DALA)	0.97	92.6	7.4	0.52	88.9	11.1
Roots at harvest (112 DALA)	0.03	76.0	24.0	0.04	63.8	36.2

In order to classify the radioactive residues extracted with methanol from sugar beet root into organosoluble and water soluble fractions, a partition of the extract on an Extrelut NT3 column was performed for the samples from the P-label experiment, and liquid/liquid partition between ethyl acetate and water was carried out for the morpholine label experiment. Nearly half of the extracted radioactivity was found to be water-soluble in case of the P-label (32.7% TRR), and the major part of the extracted ^{14}C was recovered in the aqueous phase in case of the M-label (39.0% TRR).

Six fractions were identified and quantitated in the combined methanol extract of sugar beet leaves (phenyl label). The major components were a glucosyl conjugate of the N-oxide of fenpropimorph (BF421-36 glucoside, 24.6% TRR), glucosylated hydroxypropylamine derivative (BF421-20 glucoside, 17.9% TRR), fenpropimorph (4.6% TRR) and it's N-oxide BF421-14 (7.0% TRR). A further prominent fraction corresponded to the three metabolites BF421-1 diglucoside and/or BF421-1 glucoside (glucosyl conjugates of the hydroxylated metabolite BF421-1) and/or BF421-20 (hydroxypropylamine derivative with an additional hydroxy group at the *t*-butyl moiety), which were not separated in the HPLC systems used (11.8% TRR). Two further factions were assigned by retention time comparison to represent the metabolite BF421-1 malonylglucoside (5.3% TRR) and the hydroxypropylamine derivative BF421-7 formed by decomposition of the morpholine ring system (4.2% TRR).

Seven fractions were identified in methanol extract of sugar beet leaf from the M-label experiment. The major components were parent compound fenpropimorph (19% TRR) and its N oxide BF421-14 (10.2% TRR), BF421-36 glucoside (19.2% TRR), 2,6 dimethylmorpholine (2,6-DMM, BF421-10; 11.1% TRR), and BF421-20 glucoside (10.3% TRR). One, two, or all of the three non-separated metabolites BF421-1 diglucoside, BF421-1 glucoside and BF421-20 were present at 3.3% TRR. Two further minor fractions were assigned by retention time comparison to correspond to the metabolites BF421-1 malonylglucoside (2.2% TRR) and the hydroxylated derivative BF421-1 (1.5% TRR). The formylated and acetylated derivative of metabolite BF421-7 (1.0% TRR) characterized by its retention time is possibly a product of further non-biotic derivatization of the hydroxypropylamine metabolite. An additional number of small fractions were characterized for both labels based on their chromatographic properties.

In the combined methanol extract of sugar beet root (phenyl label), the major component corresponded to the parent compound (33.5% TRR). Other significant components were a fraction corresponding to one or two or all of the three non-separated metabolites fenpropimorph diglucoside, BF421-1 glucoside and BF421-20, representing 15.3% TRR and the metabolite BF421-20 glucoside (10.6% TRR).

The major component in the combined methanol extract of the root sample with the morpholine label was a sugar with high fructose content (Rabe 2006 1036924). The other major component identified was fenpropimorph (17.4% TRR). A couple of additional minor components were characterized in sugar beet root extracts for both labels by their elution behaviour. The unknown polar derivatives were further characterized in case of the morpholine label by a fermentation experiment with yeast (after enrichment in the aqueous phase by liquid/liquid partition) as not being ^{14}C sugar compounds.

The amounts of ^{14}C unextracted by methanol in the two sugar beet matrices were low.

The solids after methanol extraction of sugar beet leaf accounted for 7.4% TRR for the phenyl label and 11.1% TRR for the morpholine label, respectively. Treatment of the PES of sugar beet leaf with an aqueous ammonia solution solubilized an additional 2.4 to 2.5% TRR. The final radioactive residues in solids after NH_4OH treatment contained less than 7% TRR (< 0.040 mg eq/kg).

In sugar beet root, the relative portions of PES were moderate, but the absolute levels (mg eq/kg) were very low, accounting for 24.0% TRR (0.006 mg eq/kg) for the phenyl label or 36.2% TRR (0.015 mg eq/kg) for the morpholine label. Considerable portions of the residual ^{14}C after methanol extraction of sugar beet root were released by incubation with Macerozyme (an enzyme mixture from *Rhizopus* spp containing pectinase, cellulase, and hemicellulase) and Cellulase (from *Trichoderma viride*) (9.6% TRR released for the phenyl label, 21.2% TRR for the morpholine label), indicating a possible association of fenpropimorph or its degradation products in roots with insoluble natural products like cell wall polymers.

The quantification of the individual metabolites present in the different plant matrices is summarized in Table 13.

Table 13 Summary of the identified components in sugar beet samples (112 DALA) after treatment with ^{14}C-fenpropimorph (Rabe and Gläßgen 2005a 1021389, Rabe 2006 1036924)

	P-label		M-label	
	Leaf	Root	Leaf	Root
TRR (mg eq/kg)	0.971	0.026	0.515	0.042
	%TRR			
Extracted (CH_3OH)	92.6	76.0	88.9	63.8
fenpropimorph	4.6	33.5	19.0	17.4
BF421-14 (N-oxide)	7.0	-	10.2	-
BF421-20 glucoside	17.9	10.6	10.3	-
BF421-1 diglucoside	11.8	15.3	3.3	-
BF421-1 glucoside				
BF421-20				
BF421-36 glucoside	24.6	-	19.2	-
BF421-1 malonylglucoside	5.3	-	2.2	-
BF421-7	4.2	-		
BF421-10 (2,6-DMM)			11.1	
Sugar high in fructose				22.9
BF421-1			1.5	-
Unextracted	7.4	24.0	11.1	36.2
NH_4OH solubilised	2.5	NA	2.4	NA
Enzyme solubilised	NA	9.6	NA	21.2
Total	100	100	100	100

NA = not applicable

- = not detected

In conclusion, the major degradation reactions for fenpropimorph in sugar beet were N oxidation of the morpholine ring, hydroxylation at the *t*-butyl moiety and subsequent conjugation reactions (glucosylation and malonylation), cleavage (detachment of the morpholine ring) or decomposition of the morpholine ring system to form hydroxypropylamine derivatives (Figure 5).

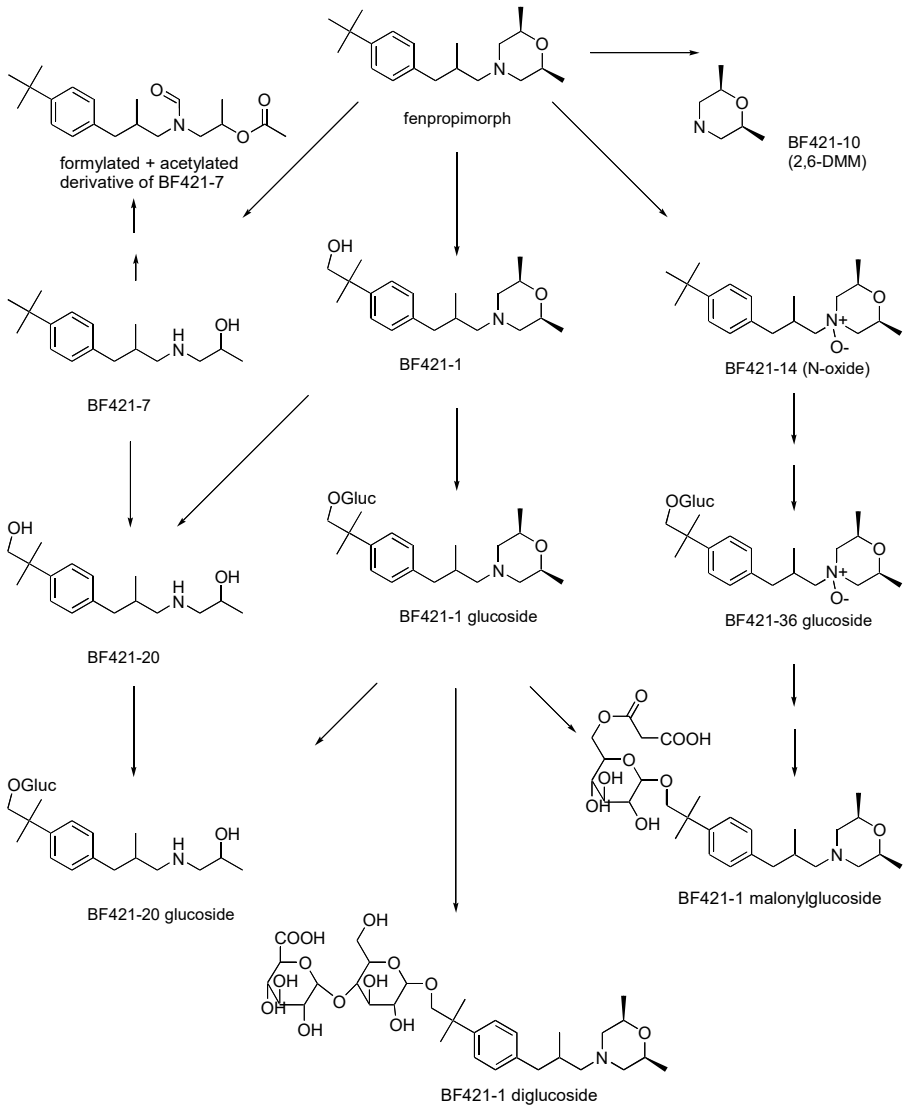


Figure 5 Compounds formed during the metabolism of fenpropimorph in sugar beets.

Note, metabolites BF421-1 diglucoside, BF421-1 glucoside and BF421-20 were not separated in the HPLC systems used and their occurrence has therefore only been established the sum of these

Barley

Pryde and Etterli (1979 10097) studied the dissipation and translocation of fenpropimorph following topical applications to summer barley (cv Herta) maintained under **greenhouse** conditions. Leaves were treated at the five-leaf tillering stage with [benzylic-^{14}C]-fenpropimorph. The application was at a rate equivalent to 0.9 kg free amine/ha. Treated and untreated leaves from the same plant as well as untreated leaves from controls were sampled at various intervals. Surface residues were removed by stirring the leaves with a mixture of HCl 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 and HPLC. Whole plants were examined by autoradiography.

Results are shown in the following table 14.

Table 14 Distribution of total radioactivity and fenpropimorph in or on barley leaves from plants treated with [^{14}C]-fenpropimorph (Pryde and Etterli 1979 10097)

DALA	Treated leaves	Other green plant

	Surface wash		Washed leaves		parts (untreated)
	Wash (%applied)	Fenpropimorph[A] (%applied)	Extracted (%applied)	Fenpropimorph[B] (%applied)	Extracted (%applied)
0	63	59	3.8	3.3	0.03
1	51	40	9.8	6.8	0.025
5	49	35	11	1.9	0.065
8	34	23	12	1.1	0.085
15	26	15	19	1.2	0.175
20	12	7.8	28	2.0	0.67

[A] Average of TLC and HPLC results

[B] TLC

Table 14 shows that up to 63% of the applied radioactivity is on the leaf surface on the day of application and 93% (100×59/63%) of the surface residue is unchanged fenpropimorph. Even after 20 days, up to 12% of the applied ^{14}C is on the surface, 63% (100×7.8/12) of which is fenpropimorph. The proportion of the applied radioactivity remaining in the leaf after washing increased from about 3.8% on the day of application to 28% after 20 days and in the same period the proportion of fenpropimorph decreased from 87% (100×3.3/3.8) to 7% (100×2/28) of the extracted ^{14}C. Although always at low levels, the increase in the ^{14}C in untreated plant parts during the test period indicates some translocation, albeit at low levels, from the leaves to the roots and uptake into other plant parts. Autoradiography of the plants was reported to confirm that a small amount of ^{14}C travelled from treated leaves towards roots and subsequently upwards in the transpiration stream to the remainder of the plant.

Fenpropimorph is a significant component of the residue in barley leaves, declining in proportion of total residues with interval after application.

Wheat

In an additional study on cereals (Huber 1979a 0148), Kolibri variety summer wheat was spray-treated with [morpholine-(2,6)-^{14}C]-fenpropimorph 55 days after seeding at a rate equivalent to 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 15.

Table 15 Radioactive residues in spring wheat treated with morpholine-labelled fenpropimorph (Huber 1979a 0148)

DALA	21 (green plant)	43 (green plant)	84 (straw)	84 (grain)
TRR (mg eq/kg)	6.48	4.33	11.86	0.43
		%TRR		
Extracted MeOH	87	79	62	9
Fenpropimorph	46.6	38.1	22.0	
BF421-1	6.9	7.2	7.8	
BF421-7[A]	5.6 (11.1)	5.3 (10.6)	13.2 (26.3)	
BF421-10	0.21	2.5	2.7	
Unknown MW=103[A]	0.9	0.9 (1.8)	0.4 (0.8)	
Unextracted	12	22	40	95
Lignin			21	
HCl solubilised			7	
Starch				49
Protein				16
Polysaccharides				5

[A] figures in brackets are corrected values taking into account that one labelled C_3 chain from the morpholine ring was lost

TRRs and the extractability with methanol decreased with time after application. By harvest, only about 9% of the ^{14}C in harvested grain was extracted with methanol. The extracted residues in grain were too low to permit identification. Almost half of the unextracted residue was incorporated into the starch (49% TRR), with 16% TRR associated with a protein precipitate and 5% TRR in the polysaccharide fraction. In the case of wheat straw, 40% of the TRR was unextracted by methanol of which 21%TRR was associated with the lignin fraction.

Fenpropimorph was the major component of the residue (38-47% TRR) in green plants with BF421-7 the main metabolite (10–12% TRR). BF421-7 also a significant component of the residue in straw (26% TRR) together with fenpropimorph (22% TRR). BF421-1 and 421-10 were detected in all plant parts except the grain but at < 10% TRR.

In further work on cereals, a trial was conducted under **outdoor conditions** where [phenyl-U-^{14}C]- or [morpholine-2,(6)-^{14}C]-fenpropimorph was applied to spring wheat (variety Ralle) at an application rate of 750 g ai/ha and 5 weeks after sowing (Hoffmann 1986a 1000141, Hoffmann 1986b 1000142, Rüdel 1990a 0524, Rüdel 1990b 0525). Forage samples were taken for analysis 21 days (phenyl label) and 28 days (morpholine label) after application. A further sampling for straw and grains interval was 56 days after application (phenyl label) and 57 days (morpholine label), respectively. Samples were extracted with methanol, the residue transferred to water, acidified and extracted successively with hexane, chloroform, acid ethyl acetate and basic ethyl acetate. Metabolites were identified by comparison with reference substances. Where possible, they were isolated by column chromatography and their structures elucidated by GC-MS.

For characterization of the residues unextracted with methanol in straw, the post-extraction solids were subjected enzymatic hydrolysis (incubation for 2 h at 37 °C with a mixture α-glucosidase, β-glucosidase, β-glucuronidase, cellulase, hemicellulase and pectinase) and a Klason-lignin preparation (66% H_2SO_4, 48h 20 °C followed by 0.5% HCl, 9h reflux).

Methanol extracted 52–97% TRR from forage, 56–61% from straw and 12–13% from grain (Tables 16 and 17).

Characterisation of methanol extracts indicated fenpropimorph is the main component of the residue found in forage 21 days after application at 16 to 26.7% TRR, with no other single component accounting for more than 4.9% TRR. Fenpropimorph was also the major component in straw at 19.8–23.7% TRR with BF421-2 accounting for up to 7.0% TRR. The largest individual unidentified component accounted for 7 to 12.4%TRR. TRR in methanol extracts of grain were too low to permit their characterisation.

Radioactivity unextracted using methanol was subject to further investigation. In grain, only low amounts of ^{14}C were incorporated into polysaccharides and proteins (< 10% TRR) with the majority associated with starch (31-32% TRR). In straw, about 12% TRR was associated with lignin fractions.

Table 16 Radioactive residues in spring wheat treated with phenyl-labelled fenpropimorph (Hoffmann 1986a 1000141, Hoffmann 1986b 1000142, Rüdel 1990b 0524)

DALA	0 (green plant)	21 (green plant)	56 (roots)	56 (straw)	56 (grain)
TRR (mg eq/kg)	65.6	8.81	2.87	10.9	0.106
			%TRR		
Extracted MeOH	97.4	62.5	40.7	60.9	12.2
Fenpropimorph		26.7		23.7	
BF421-1		0.37		0.02	
BF421-1 and/or BF421-7		2.01		0.39	
BF421-2		0.08		6.96	
BF421-13		1.50		0.19	
BF421-15 and/or BF421-2-Me		0.28		0.02	
Unidentified		30.1 (n=25 max 4.9)		30.7 (n=22 max 5.6)	

DALA	0 (green plant)	21 (green plant)	56 (roots)	56 (straw)	56 (grain)
Not analysed by TLC		2.4		3.3	
Unextracted	2.65	37.5	59.3	39.1	87.8
Lignin					11.6
Enzyme solubilised				6.2	
H_2SO_4 solubilised				6.1	
Starch					32
Protein					4.9
Polysaccharides					0.9

Table 17 Radioactive residues in spring wheat treated with morpholine-labelled fenpropimorph (Hoffmann 1986a 1000141, Hoffmann 1986b 1000142, Rüdel 1990b 0524)

DALA	7 (green plant)	28 (green plant)	57 (roots)	57 (straw)	57 (grain)
TRR (mg eq/kg)	40.3	12.2	2.33	24.0	0.372
			%TRR		
Extracted MeOH	94.0	51.9	25.8	56.0	12.9
Fenpropimorph		16.4		19.8	
BF421-1		0.01		0	
BF421-2		0.62			
BF421-7		0.65		0.3	
BF421-13		0.22			
unidentified		34.1 (n=19 max 4.4)		35.7 (n=27 max 12.4)	
Not analysed by TLC		14.3		2.7	
Unextracted	6.04	48.1	74.2	44.0	87.1
Lignin					8.6
Starch					31
Protein					7.8
Polysaccharides					1.1

In summary for wheat, ^{14}C-fenpropimorph is metabolized by two main transformation steps:

- oxidation of the *t*-butyl group to the respective alcohol (BF421-1), followed by a further oxidation to the respective acid (BF421-2) and its methylation product (BF421-2-Me)
- oxidation of the morpholine ring leading to BF421-7.

In forage and straw, unchanged fenpropimorph is the major component of the ^{14}C.

Metabolites of fenpropimorph in wheat are presented in Figure 6.

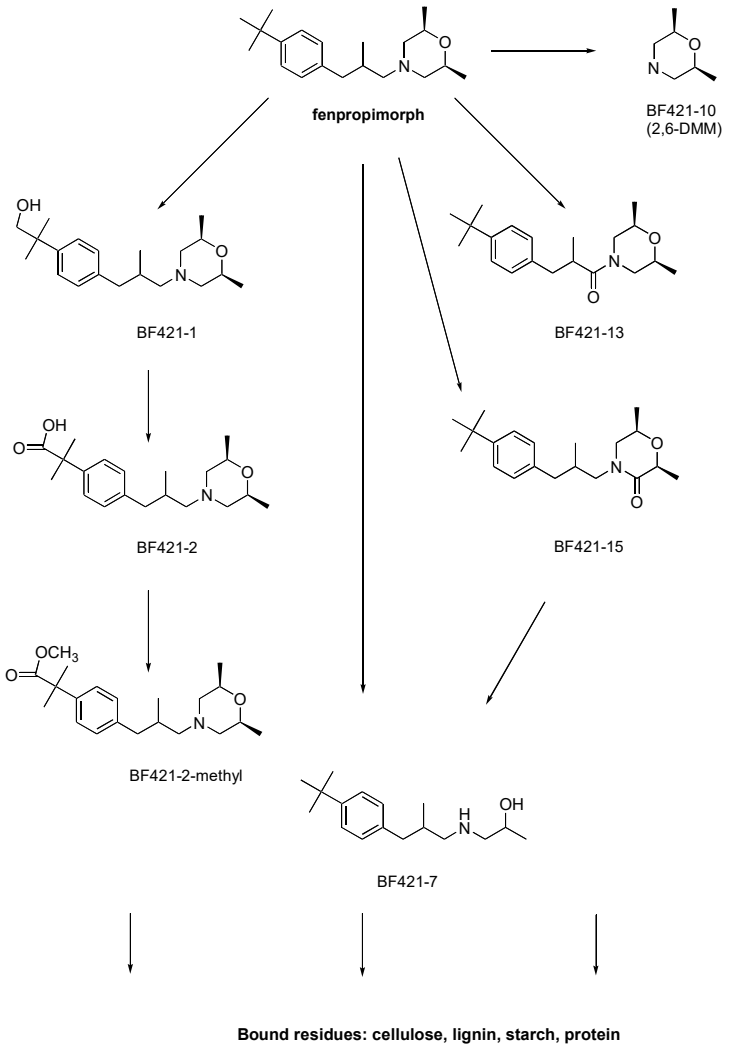


Figure 6 Metabolites of fenpropimorph in wheat

Rabe and Gläßgen (2005b 1001288) also studied the metabolism of [phenyl-U-^{14}C]- or [morpholine-2,(6)-^{14}C]-fenpropimorph in wheat. In some experiments [ethyl-1,2-^{13}C, morpholine-2-^{13}C]-fenpropimorph was used to facilitate metabolite identification using ^{13}C-NMR. EC formulations of labelled fenpropimorph was applied to spring wheat (variety Star) as two foliar applications of 0.75 kg ai/ha each. Wheat forage was sampled 25 days after the first of two applications and dried to hay, and mature wheat plants were harvested 49 days after the second application and separated into grain and straw (including chaff). Plant samples were homogenized and analysed by combustion and radioactivity measurement for the determination of the TRRs.

The plant material of both labels was extracted with methanol. In addition, liquid/liquid partitioning experiments using n-hexane, chloroform and ethylacetate were carried out. For further characterization of the ^{14}C remaining in solids after methanol extraction, proteins and polysaccharides from grains were isolated and the radioactive residues determined (protein solubilisation using 1% aqueous ammonia; cell wall polysaccharide cleavage with Macerozyme (*Rhizopus spp*)/cellulase (*Trichoderma viride*) at 37 °C for 3 h, starch solubilisation using α-amylase (Bacillus spp)/β-amylase (barley)/amyloglucosidase (*Aspergillus niger*) at 37 °C for 2 days and lignin cleavage/solubilisation with tyrosinase (mushroom)/laccase (*Rhus vernificera*) 37 °C for 2 days). For characterization of the residues in straw remaining after methanol extraction, the post-extraction solids were subjected a Klason-lignin preparation.

Samples from the morpholine label experiment were subsequently reanalysed to clarify the identity of a polar metabolite initially identified as dimethylmorpholine (Rabe 2006a 1036924). The results discussed below are a composite of the findings of Rabe and Gläßgen 2005b (1001288), Rabe 2006a 103924, Rabe 2006a 103924, and Rabe 2006b (1034290).

The extractability of the radioactive residues with methanol depended on the matrix under investigation and was good for wheat hay (83.1–83.0% TRR) and quite good for wheat straw (66.6–71.2% TRR). From the wheat grain samples, 14.7% TRR (M-label) and 35.3% TRR (P-label) were extracted with methanol. The extraction behaviour is summarized in Table 18.

Table 18 Extraction of radioactivity after treatment of wheat with ^{14}C-fenpropimorph (Rabe and Gläßgen 2005b 1001288)

	M-label			P-label		
	TRR (mg eq/kg)	extracted MeOH (%TRR)	Unextracted (%TRR)	TRR (mg eq/kg)	extracted MeOH (%TRR)	Unextracted (%TRR)
Spring wheat hay	26.5	83.0	17.0	48.5	83.1	16.9
Spring wheat straw	9.45	71.2	28.8	13.9	66.6	33.4
Spring wheat grain	0.33	14.7	85.3	0.13	35.3	64.7

In order to classify the metabolites extracted with methanol from spring wheat hay into organosoluble and water-soluble fractions, liquid/liquid partition was carried out between ethyl acetate and water. More than one third of the methanol extracted residues were found to be water-soluble (29.0–33.8% TRR).

In the concentrated methanol extract of spring wheat hay (P-label), seven fractions were identified. The major components were parent compound (20.3% TRR) and its N oxide (metabolite BF421-14, 13.6% TRR) and BF421-1 malonylglucoside (22.1% TRR). A further peak that represented the metabolites BF421-1 diglucoside and/or BF421-1 glucoside and/or BF421-20 (hydroxypropylamine derivative of fenpropimorph with an additional hydroxyl group at the *t*-butyl moiety) which were not separated in the HPLC systems used but identified in hay extracts by LC-MS/MS and co chromatography accounted for 7.9% TRR. The hydroxypropylamine derivative itself (BF421-7, 5.2% TRR; formed by decomposition of the morpholine ring system) and the hydroxylated metabolite BF421-1 (2.4% TRR) were found as additional degradation products, accompanied by minor amounts of a formylated and acetylated (possibly non-biotic) derivative of the metabolite BF421-7 (1.8% TRR) and of the conjugate of the hydroxypropylamine derivative, BF421-20 glucoside (0.4% TRR).

In the methanol extract of the hay sample from the M-label experiment seven metabolites were also identified: the BF421-1 malonylglucoside (20.4% TRR), parent compound (20.9% TRR) and its N oxide (BF421-14, 14.0% TRR), 2,6 dimethylmorpholine (2,6-DMM, metabolite BF421-10; 6.5% TRR) which was only detectable with the M-label, the non-separated metabolites BF421-1 diglucoside and/or BF421-1 glucoside and/or BF421 20 (7.2% TRR), and minor amounts of the metabolite BF421-7 (2.3% TRR), its formylated and acetylated derivative (2.7% TRR), and of the metabolite BF421-1 (1.6% TRR). An additional number of small peaks were characterized with both labels based on their chromatographic properties.

The metabolic profiles of the extracts of spring wheat straw showed a very similar composition of degradation products. The methanol extract of the straw sample with the P-label experiment was shown to contain the BF421-1 malonylglucoside (15.4% TRR), the parent compound (6.9% TRR) and its N oxide (BF421-14, 11.8% TRR) the non-separated metabolites BF421-1 diglucoside and/or BF421-1 glucoside and/or BF421-20 (7.8% TRR), and minor amounts of the metabolite BF421-7 (4.3% TRR), the metabolite BF421-1 (2.7% TRR), the glucosyl conjugate of the N oxide of fenpropimorph (BF421-36 glucoside, 1.7% TRR), the BF421-20 glucoside (1.6% TRR), and the (possibly non-biotic) formylated and acetylated derivative of BF421-7 (1.2% TRR).

The methanol extract of the straw sample from the M-label experiment contained the major metabolite BF421-1 malonylglucoside (16.1% TRR), parent compound (7.1% TRR) and its N oxide (metabolite BF421-14, 14.3% TRR), 2,6-DMM (metabolite BF421-10, 6.4% TRR), the non-separated metabolites BF421-1 diglucoside and/or BF421-1 glucoside and/or BF421-20 (6.7% TRR), and minor amounts of the formylated and acetylated derivative of BF421-7 (3.3% TRR), the metabolite BF421-1 (1.4% TRR), the metabolite BF421-7 (1.2% TRR), the BF421-36 glucoside (1.2% TRR), and the BF421-20 glucoside (1.0% TRR). A few further small fractions of unknowns were characterized for both labels based on their elution behaviour.

In the methanol extract of spring wheat grain (P-label), the major components were parent compound (16.4% TRR) and its N oxide (metabolite BF421-14, 4.1% TRR) together with the BF421-1 malonylglucoside (4.7% TRR), and BF421-1 diglucoside and/or BF421-1 glucoside and/or BF421-20 (3.2% TRR), the metabolite BF421-7 (2.3% TRR) and the metabolite BF421-1 (1.3% TRR).

The major components identified in the methanol extract of the grain sample from the morpholine label experiment were the parent compound (1.7% TRR) and its N oxide (metabolite BF421-14, 0.6% TRR) together with 2,6-DMM (metabolite BF421-10, 0.6%TRR). Further significant components, in comparison to parent, were BF421-1 malonylglucoside (0.3% TRR) and the non-separated metabolites BF421-1 diglucoside and/or BF421-1 glucoside and/or BF421-20 (0.3% TRR). Sugar, predominantly fructose, accounted for 6.5% TRR. A couple of additional minor fractions were characterized in wheat grain extracts with both labels by their elution behaviour.

Different proportions of residues unextracted using methanol were observed in the three wheat matrices. The PES after methanol extraction of wheat hay accounted for 16.9% TRR for the P-label or 17.0% TRR for the M-label experiments, respectively. In wheat straw, the ^{14}C in PES were moderate at 33.4% TRR for the P-label or 28.8% TRR for the M-label experiments. The relative amount of unextracted residues of fenpropimorph in spring wheat grain were comparably high and amounted to 64.7% TRR with the P-label or 85.3% TRR in case of the M-label experiment.

More than 90% of the ^{14}C in PES were released from each matrix by sequential solubilisation procedures including treatments with aqueous ammonia, Macerozyme and cellulase, amylases and amyloglucosidase, tyrosinase and laccase, or reflux with NaOH. The most effective solubilisation step applied for hay and straw was the treatment with aqueous ammonia which released additional portions of the more polar metabolites (particularly of glucoside conjugates and of 2,6-DMM). The incubations of the solids with Macerozyme/cellulase and amylase/amyloglucosidase (which were applied for all three matrices) mainly released the hydroxylated metabolite BF421-1 and the metabolite BF421-10 (2,6-DMM, which was detected only with the M-label) and in some cases minor amounts of the parent compound and the metabolite BF421-7. Major parts of the radioactive residues were also solubilized with NaOH, and minor portions of these solubilized residues could be precipitated with HCl. Considerable parts of the ^{14}C in PES were thus concluded to consist of fenpropimorph or its degradation products associated with or embedded/incorporated in insoluble natural polymers (starch and cell wall components like hemicelluloses, lignin, lignin-carbohydrate complexes or pectin). The final ^{14}C remaining in solids after the sequential solubilisation procedures contained less than 2.5% TRR for all matrices.

The quantification of the individual metabolites present in the different plant matrices is summarized in Table 19 and Table 20.

Table 19 Summary of the identified components in wheat samples after treatment with ^{14}C-fenpropimorph (Rabe and Gläßgen 2005b 1001288, Rabe 2006a 103924, Rabe 2006b 1034290)

	M-label			P-label		
	Hay 25 DALA	Straw 49 DALA	Grain 49 DALA	Hay 25 DALA	Straw 49 DALA	Grain 49 DALA
TRR (mg eq/kg)	48.013	14.626	0.131	25.446	9.641	0.345
	%TRR					
Extracted (CH_3OH)	83.0	71.2	14.7	83.1	66.6	35.3
fenpropimorph	20.9	7.1	1.7	20.3	6.9	16.4
BF421-14 (N-oxide)	14.0	14.3	0.6	13.6	11.8	4.1
BF421-20 glucoside	-	1.0	-	0.4	1.6	-

	M-label			P-label		
	Hay 25 DALA	Straw 49 DALA	Grain 49 DALA	Hay 25 DALA	Straw 49 DALA	Grain 49 DALA
BF421-1 diglucoside BF421-1 glucoside BF421-20	7.2	6.7	0.3	7.9	7.8	3.2
BF421-36 glucoside	-	1.2	-	-	1.7	-
BF421-1 malonylglucoside	20.4	16.1	0.3	22.1	15.4	4.7
BF421-7	2.3	1.2	-	5.2	4.3	2.3
BF421-10 (2,6-DMM) Sugar [A]	6.5	6.4	0.6 6.5			
BF421-1	1.6	1.4	-	2.4	2.7	1.3
Formylated and acetylated derivative of BF421-7	2.7	3.3	-	1.8	1.2	-
Unresolved						
Polar region	1.1	5.3	7.7	-	0.2	2.3
Medium polar region	-	2.9	-	3.7	10.2	-
Non-polar region	1.2	1.3	0.3	1.6	2.5	-
Unextracted	17.0	28.8	85.3	16.9	33.4	64.7
NH_4OH solubilised	8.4	15.8	NA	8.6	20.9	NA
Macerozyme/cellulase	2.9	3.8	35.8	2.1	3.4	25.4
Amylase/amyloglucosidase	0.8	1.2	20.3	0.8	1.3	15.1
Tyrosinase/laccase	0.5	0.5	5.1	0.4	0.6	3.6
NaOH reflux	2.3	3.6	16.4	2.2	2.8	16.1
HCl precipitate from NaOH solubles	0.5	1.3	4.0	0.6	1.0	3.2
Total	100	100	100	100	100	100

NA = not applicable

- = not detected

[A] *predominantly fructose*

Table 20 Identification of radioactivity remaining in solids after methanol extraction that are solubilised by the sequential treatment with NH_4OH, Macerozyme/cellulose and amylase/amyloglucosidase (Rabe and Gläßgen 2005b 1001288, Rabe 2006a 103924, Rabe 2006b 1034290)

	M-label			P-label		
	Hay 25 DALA	Straw 49 DALA	Grain 49 DALA	Hay 25 DALA	Straw 49 DALA	Grain 49 DALA
	%TRR					
Solubilised PES	**12.1**	**20.8**	**56.1**	**11.5**	**25.6**	**40.5**
fenpropimorph	-	-	-	*0.4*	*0.4*	
BF421-14 (N-oxide)						
BF421-20 glucoside	*0.1*	-	-	*0.2*	*0.9*	
BF421-1 diglucoside	*5.1*	*8.3*	-	*5.7*	*9.4*	
BF421-1 glucoside						
BF421-20						
BF421-36 glucoside	-	-	-	*0.8*	*2.1*	
BF421-1 malonylglucoside	*0.3*	-	-	*0.5*	-	
BF421-7	-	-	-	*0.3*	*0.8*	
BF421-10 (2,6-DMM)	*1.5*	*6.1*	-			
Sugar [A]			*(67.2)*[B]			
BF421-1	*1.6*	*1.0*	-	*2.0*	*2.7*	
Polar region	*3.1*	*3.5*	-	-	-	
Medium polar region	*0.3*	*1.9*	-	*1.7*	*9.2*	
Non-polar region	-	-	-	-	-	
Total	**12**	**20.8**	**56.1**	**11.6**	**25.5**	

NA = not applicable

- = not detected

[A] *predominantly fructose*

[B] *reanalysis of PES solubilised 64.1% TRR with sugars accounting for 67.2% TRR*

In summary, the major degradation reactions for fenpropimorph in spring wheat were hydroxylation at the *t*-butyl moiety and subsequent conjugation reactions (glucosylation and malonylation), decomposition of the morpholine ring system to form hydroxypropylamine derivatives, and N oxidation of the morpholine ring.

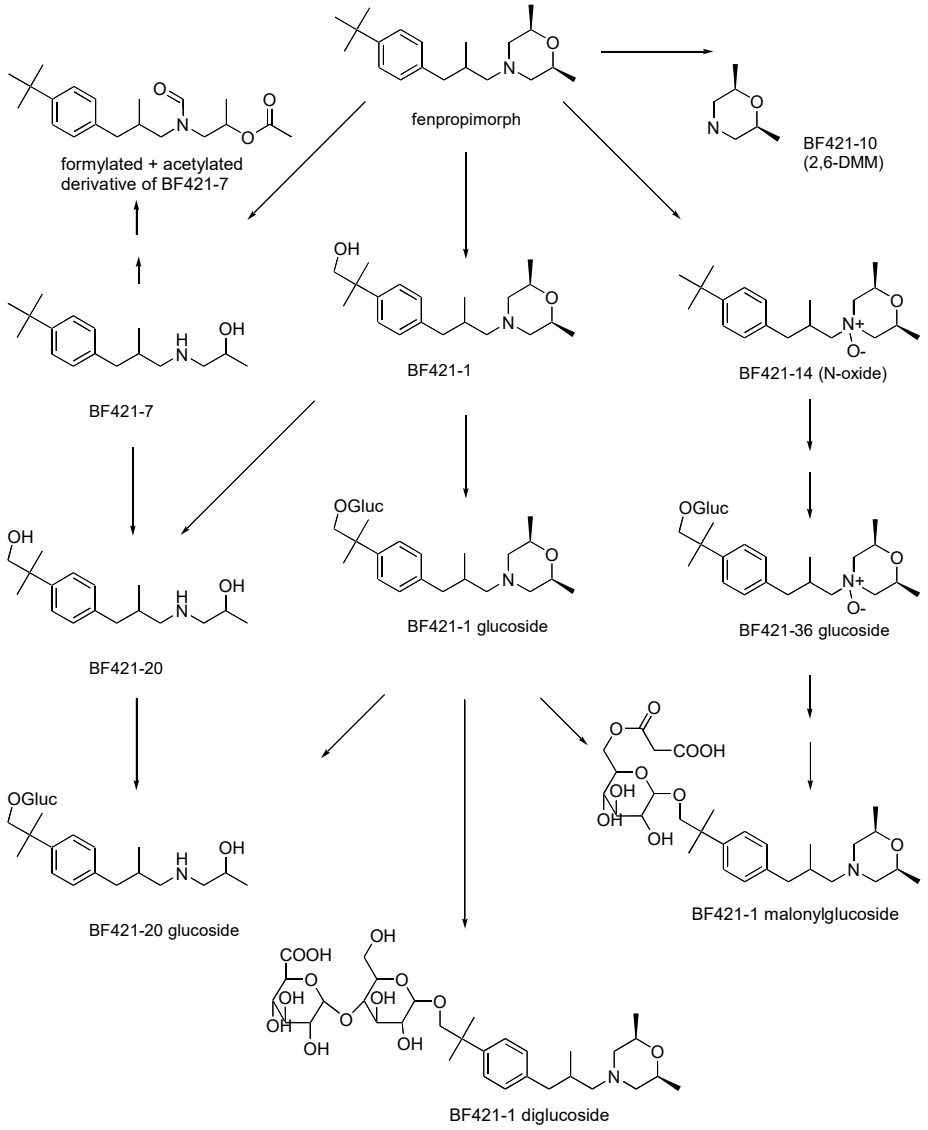


Figure 7 Metabolites of fenpropimorph in wheat, note the scheme is the same as for sugar beet

Animal metabolism

Laboratory animal studies

Metabolism of fenpropimorph in rats was evaluated by the WHO Core Assessment Group of the 2016 JMPR. Metabolites identified in rats included BF421-2, BF421-3 and its conjugates, BF421-4, BF421-16 and BF421-17.

Lactating goat

The metabolism and distribution of ^{14}C-fenpropimorph was investigated in two lactating goats (Saanenziege, white, 40 and 60 kg bw) following repeated oral administration by intubation (Ritter 1989a 10215). Fenpropimorph was administered daily on five consecutive days at a nominal dose level of 56 mg/kg bw (corresponding to approximately 2336 ppm in the diet for the P-label and

1421 ppm for the M-label based on daily feed consumption during the acclimatisation period of 1.4 and 1.5 kg/day respectively). Mean milk production during the dosing period was 0.6 and 0.7 L/day respectively. Animals were euthanized approximately 5 hours after that last dose.

Following the administration of ^{14}C fenpropimorph, radioactivity is excreted via faeces (20–29%) and urine (14–21%). Only small amounts are excreted via milk or remain in tissues (Table 21). The material balance was low in this study (40–55%) which is likely due to material remaining in the intestine, which was not analysed.

Table 21 Distribution of ^{14}C following administration of [^{14}C]-fenpropimorph for five days

	Phenyl label		Morpholine label	
	%AD	mg eq/kg	%AD	mg eq/kg
Tissues	**2.65** [A]		**3.13** [A]	
Liver		*140.6*		*124.5*
Kidney		*232.7*		*53.4*
Fat		*4.3*		*18.7*
Mammary		*24.0*		*10.6*
Muscle		*8.9*		*6.3*
Brain		*5.6*		*9.0*
Spleen		*12.6*		*14.9*
Heart		*21.7*		*14.5*
Bile	**1.8**	**10128**	**1.08**	**4240**
Milk	**0.06** [B]	**0.68-9.7**	**0.32**	**0.7-23.1**
Excreta	**34.92**		**50.29**	
Faeces	*20.43*		*28.95*	
Urine	*14.49*		*21.34*	
Cage wash	**1.02**		**0.44**	
Total	**40.45**		**55.26**	

[A] Assumes fat and muscle are 12 and 40% respectively of body weight

[B] P-label dosed goat only produced 1 and 7 mL of milk at 1 and 4 hours after the last dose, the sample collected for further analysis.

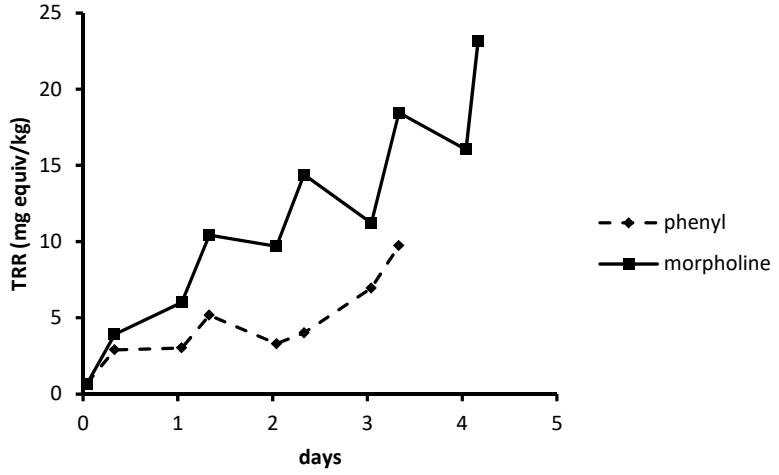


Figure 8 Residues in milk following dosing with ^{14}C-fenpropimorph

During the course of the study milk production for the goat dosed with the P-label declined from 0.8 L/day on day 3 to 0.12 and 0.01 L/d on days 4 and 5 and may in part explain the higher levels of ^{14}C observed in milk for the P-label. Residues in milk did not reach a plateau by day five of dosing. There were significant differences in ^{14}C levels between milk collected one hour after dosing compared to eight hours, suggesting fenpropimorph residues are rapidly eliminated following dosing.

Radioactivity in blood ranged from 24.3–32.6 mg/L for the phenyl label and 13.6–17.7 mg/L for the morpholine label for the period between the 2nd and 4th daily dose and was 54.4 and 29.1 mg/L

at 5 hours after the last dose. No decline in plasma levels were observed in the period to 5 hours after the last dose.

A range of extraction schemes were used to further characterise residues in milk and tissues.

For milk, proteins were precipitated (P-label) by addition of acetone and ^{14}C in the protein and whey fractions determined. In the case of milk from the M-label experiment, milk was first diluted with water and then extracted with n-hexane (2×) prior to precipitation of proteins with addition of acetone. The whey fraction was extracted with *n*-hexane, dichloromethane. The protein fraction was extracted with methanol.

Fat samples were extracted with 80% methanol (4–5×) followed by methanol (3×) and the combined extracts concentrated by rotary evaporator at 50 °C prior to partitioning with hexane (4×). The remaining aqueous phase was partitioned with dichloromethane (4×) and the aqueous phase and pooled organic extracts analysed by TLC. An aliquot of the aqueous phase from the M-label sample was lyophilised, re-suspended in methanol or was hydrolysed by reaction with 2N HCl for 72 hours at 70 °C prior to analysis by TLC.

Muscle samples were extracted with 80% methanol (3×), the extracts filtered and an aliquot partitioned with *n*-hexane to remove dissolved lipids. Polar residues, isolated from TLC plates, were hydrolysed by reaction with 2N HCl for 72 hours at 70 °C.

Liver samples were extracted with 80% methanol (5×), the extracts were centrifuged and the supernatant filtered using filter paper and a membrane filter. Polar residues, isolated from TLC plates, were hydrolysed by reaction with 2N HCl for 48 hours at 70 °C and the resulting compound, together with reference compounds, were derivatized by methylation with diazomethane prior to analysis by TLC. Additionally a sample of whole liver extract was hydrolysed with 0.5N HCl for 48 hours at 70 °C

Kidney samples were extracted with 80% methanol; the extracts were filtered using filter paper. Two major metabolites isolated using TLC were derivatized by methylation with diazomethane to aid identification. Polar residues, isolated from TLC plates, were hydrolysed by reaction with 4N HCl for 120 hours at 70 °C, and derivatized by methylation with diazomethane. However, these compounds proved to be unstable.

The extracts obtained from the different matrices were analysed by thin layer chromatography (TLC) in several mobile phases and compounds identified by comparison with synthetic reference standards.

Radioactivity in milk from the P-label experiment was predominantly found in the whey fraction at 76.2% TRR with 23.8% in the protein fraction. The whey fraction of milk from the M-label experiment contained 60% TRR (9.8% *n*-hexane soluble; 7.5% CH_2Cl_2 soluble; 42.7% TRR water soluble) and the protein fraction 33.4% TRR (4.7%TRR CH_3OH soluble).

Extractability of kidney, liver and muscle tissues with methanol was good at >83% TRR (Table 22).

Parent fenpropimorph was not detected in any tissue or in milk. The major components of the ^{14}C were BF421-2 (10.8–67.8% TRR) and BF421-1 (24.9–26.5% TRR).

Table 22 Characterisation and identification of ^{14}C residues in solvent extracts from milk and tissues

	Milk	kidney	liver	muscle	fat
M-label					
TRR (mg eq/kg)	39.156	53.39	124.53	6.34	18.74
			%TRR		
Extracted (80% CH_3OH)	**71.3** [B]	**90.3**	**84.1**	**86.0**	**54.6** [C]
BF421-1					*26.5*
BF421-2	*16.6*	*26.7*	*40.0*	*64.2*	
BF421-2 conjugate			*32.3*		
BF421-3	*7.2*	*11.0*			
Unidentified	*41.7 (n=3, 5.8-23.6)*	*52.6 (n=5, 2.2-28)*	*11.8(n=2, 4.2-7.6)*	*21.8 (n=3, 4.2-11.9)*	*28.1 (n=3, 2.9-21.5)*

	Milk	kidney	liver	muscle	fat
Extracted H_2O					**9.5**
BF421-2					*3.0*
BF421-3					*0.9*
Unidentified					*5.6 (n=3, 0.9-3.3)*
Unextracted	**28.7** [D]	**9.7**	**15.9**	**14.0**	**35.9**
P-label					
TRR (mg eq/kg)	2.835	232.73	140.57	8.91	4.34
			%TRR		
Extracted (80% CH_3OH)	**76.2 (whey fraction)**	**98.6**	**92.1**	**95.6**	**77.2** [A]
BF421-1					*24.9*
BF421-2		*10.8*	*33.3*	*67.8*	*35.0*
BF421-2 conjugate			*37.8 (6.7, 31.1)*		
BF421-3		*6.3*			
Unidentified	*76.2 (n=6, 6.1-22.0)*	*81.5 (n=4, 10.7-33.7)*	*21 (n=2, 9.0-12)*	*27.8 (n=5, 3.4-6.5)*	*17.3*
Extracted H_2O					**19.0**
BF421-2					*2.6*
Unidentified					*16.4 (n=4, 1.9-8.4)*
Unextracted	**23.8 (protein fraction)**	**1.4**	**7.9**	**4.4**	**3.8**

[A] combined n-hexane (37.7%TRR) and CH_2Cl_2 (39.5% TRR) extracts

[B] sum of n-hexane (6.6+9.8%TRR), CH_2Cl_2 (7.5% TRR), CH_3OH (4.7% TRR) and H_2O (42.7% TRR) extracts with 5.8% TRR not accounted for

[C] sum of n-hexane (39.0% TRR) and CH_2Cl_2 (15.6%TRR)

[D] 33.4%TRR protein fraction minus 4.7% TRR extracted MeOH)

Note milk was separated into protein and whey by addition of acetone. P-label milk, whey and protein fractions were not solvent extracted. Morpholine label whey and protein fractions were extracted as indicated.

Radioactivity present in urine, plasma and bile was also identified. BF421-3 was the only metabolite identified in urine (8 unknowns). BF421-2 and BF421-3 were identified in plasma while BF421-2 was identified as the main compound present in extracts from bile on hydrolysis suggesting conjugates of BF421-2 were present.

In summary, hydroxylation of the *t*-butyl- moiety leads to formation of BF421-1 which can be oxidised to give the corresponding acid BF421-2. Hydroxylation at the 2-methyl group of the dimethylmorpholine ring gives BF421-3. BF421-2 may be present as conjugates. Parent fenpropimorph was not detected in tissues or milk.

In a subsequent study, Leibold and Hoffmann (2000a 1013272, also Fabian and Knoell 2003a 1014911) studied the metabolism and distribution of ^{14}C- fenpropimorph in lactating goats (Bunte deutsche Edelziege, 2-4 year old, 35, 38 kg bw phenyl, 34, 38 kg bw morpholine) following repeated oral administration by gavage of [phenyl-U-^{14}C]- or [morpholine-2,(6)-^{14}C]-fenpropimorph in olive oil. ^{14}C-Fenpropimorph was administered daily on seven consecutive days at approximately 12 ppm feed (12.4 ppm for P-label and 12.6 ppm for the morpholine label). Feed consumption was 1.8 kg/day (phenyl) and 1.8–1.9 kg/day (morpholine). Milk production was 1.7 kg/d for the P-label dosed goats and 1.3–1.5 kg/d for the M-label dosed goats. Milk samples were collected twice daily, urine and faeces once daily. Animals were sacrificed 23 hours after the last dose and muscle, fat, liver and kidney were collected for analysis. Prior to analysis, the tissue samples of both goats for each label were pooled. In the case of milk, pooling was performed for milk of each application period. The TRR of milk, muscle, fat, liver and kidney were determined by LSC-measurement.

Following administration of ^{14}C-fenpropimorph to lactating goats, the radioactivity was rapidly and almost completely excreted. Excretion mainly occurred via the faeces (faeces M-label 46–54% AD; P-label: 37–49% AD: urine M-label 21–32% AD; P-label: 19–30% AD). Radioactivity recovered from urine and faeces together with cage wash amounted to 79–96% of the total while the total recovery of radioactivity was in the range of 79–98% (Table 23).

Table 23 Distribution of ^{14}C following administration of [^{14}C]-fenpropimorph for seven days, mean of two goats (Leibold and Hoffmann (2000a 1013272)

	M-label		P-label	
	%AD	mg eq/kg	%AD	mg eq/kg
Tissues	**0.485**		**0.36**	
Liver	*0.3*	*0.777*	*0.285*	*0.637*
Kidney	*0.025*	*0.257*	*0.01*	*0.153*
Fat	*0.02*	*0.238*	*0.015*	*0.194*
Muscle	*0.14*	*0.055*	*0.05*	*0.021*
Milk	**0.98**	**0.16**	**0.105**	**0.016**
Excreta	**76.68**		**67.125**	
Faeces	*50.23*		*42.655*	
Urine	*26.45*		*24.47*	
Cage wash	**19.37**		**11.645**	
Total	**97.515**		**79.235**	

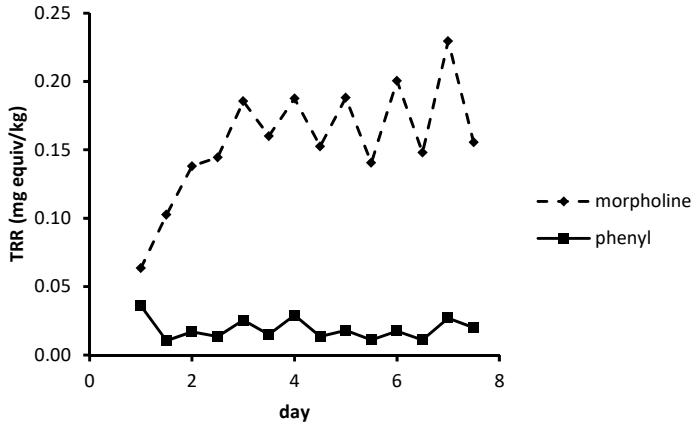


Figure 9 Residues in milk following dosing with ^{14}C-fenpropimorph..

Residues in milk appeared to reach plateau levels by day four of dosing with significant differences in ^{14}C levels between milk collected in the morning prior to dosing compared to evening milk suggesting fenpropimorph residues are rapidly eliminated following dosing. The rapid elimination is confirmed by ^{14}C in plasma which peaked at 1–6 hours after last dosing and thereafter declined with a half-life of 13.8-18.3 hours (M-label) and 43.3–66.7 hours (P-label).

TRRs in milk, muscle, fat and kidney were low ranging from 0.016 to 0.257 mg eq/kg. The TRR that in liver was 0.777 and 0.637 mg eq/kg (M-Label and P-Label).

A range of extraction schemes were used to further characterise residues in milk and tissues.

Milk: Work-up one involved precipitation of proteins with acetone/acetonitrile (1/1; v/v). The precipitate was separated and the supernatant (extract) subject to analysis by HPLC. The precipitate was further extracted with methanol. Work-up two involved adjusting the pH of the milk to pH 4.2 using 0.5 M H_3PO_4 to precipitate proteins. Following separation of the precipitate, acetone was added to the solution phase which resulted in precipitation of lactose. The acetone phase was separated from the lactose and subject to HPLC.

Liver, kidney, muscle: samples were extracted with methanol (3×) and the pooled methanol extracts analysed by HPLC. The residue following methanol extraction was further extracted with water or an aliquot also extracted sequentially with isohexane and water and finally incubated with pronase (pH 7, 37 °C overnight) and in the case of muscle an aliquot also extracted sequentially with isohexane, ethyl acetate, reflux with methanol and finally aliquots refluxed with 5M HCl or 5M NaOH.

Fat: homogenised samples were extracted with methanol (3×) and the pooled extracts analysed by HPLC or an aliquot partitioned with isohexane. The methanol phase following partitioning was analysed by HPLC. The residue following the initial methanol extraction was further extracted with water or with isohexane.

Extractability was good with >70% TRR in kidney, liver and muscle samples extracted with methanol (≥ 93% P-label, 71–78% M-label). For fat, methanol extracted 49% (M-label) to 57% TRR (phenyl) with iso-hexane extracting an additional 19% (P-label) to 21% (M-label) TRR. In the case of milk, acetone/CH_3CN extracted 22.0% (M-label) to 60.2% TRR (P-label) with methanol extracting an additional 9.7% (M-label) to 17.0% (P-label) TRR.

In solvent extracts of milk, fenpropimorph (1.4–10.0% TRR) and the metabolites BF421-2 (2.9–26.8% TRR) and BF421-3 (1.1–10.0% TRR) were the main components identified. Beside these compounds, several minor metabolites were detected.

In liver extracts, the predominant metabolite was identified as BF421-2 (56.8–72.6% TRR) together with smaller amounts of BF421-3 (5.1–6.4% TRR) and BF421-1 (1.9–10.0% TRR) and also the parent fenpropimorph (2.3–6.9% TRR). Minor metabolites identified in liver extract were the sulphate of BF421-1 and the glucuronic acid conjugates of BF421-2 and BF421-3.

The main components identified in kidney extracts were BF421-2 (25.3–40.3% TRR) and BF421-3 (20.8–40.1% TRR). The metabolites BF421-10 (1.7% TRR) and BF421-19 (5.0% TRR) were identified exclusively in M-label extract. Fenpropimorph was also present at 10.2–14.9% TRR.

The main radioactive residue in muscle extract was BF421-2 (30.0–78.6% TRR).

Identified radioactive residues in fat extract were mainly BF421-2 (28.7–33.5% TRR), GlcA of BF421-2 (14.4–15.4% TRR) and fenpropimorph (2.9–14.5% TRR).

Table 24 Characterisation and identification of ^{14}C residues in solvent extracts from milk and tissues (M-label) (Leibold and Hoffmann 2000a 1013272, Fabian and Knoell 2003a 1014911)

	Milk 1	Milk 2	Kidney	Liver	Muscle	Fat
TRR (mg eq/kg)	0.160		0.257	0.777	0.056	0.238
			%TRR			
Extracted	**22.0 (acetone /ACN)**	**66.2 (acid pH 4.2)**				
fenpropimorph	*1.4*	*2.1*				
BF421-1	-	-				
BF421-2	*2.9*	*3.1*				
BF421-2 GlcA conjugate	*0.4*					
BF421-3	*1.1*	*1.2*				
BF421-3 GlcA conjugate	*0.5*					
BF421-10	+					
BF421-19	*2.2*					
BF421-21	*0.4*					
BF421-24	*0.3*					
lactose		*19.1*				
unknowns	*12.8*	*40.7*				
Extracted CH_3OH	**9.7**		**71.1**	**77.5**	**73.2**	**73.2** [A]
fenpropimorph			*10.2*	*6.9*	*4.9*	*14.5*
BF421-1			-	*1.9*	-	-
BF421-2			*25.3*	*56.8*	*30.0*	*33.5*
BF421-2 GlcA conjugate			-	-	-	*15.4*
BF421-3			*20.8*	*6.4*	*5.9*	*5.9*
BF421-3 GlcA conjugate			-	-	-	*1.6*
BF421-4			-	+	-	-
BF421-10			*1.7*	-	-	*2.3*
BF421-19			*5.0*	-	-	-
unknowns			*8.1*	*5.5*	*32.4*	-
Unextracted	**58.5**	**22.2**	**23.9**	**19.8**	**29.3** [B]	**13.9**
Solubilised by pronase			*17.6*	*16.4*	*27.5*	
Soluble in iso-hexane						*7.1*
Water soluble			*1.9*	*6.3*		

	Milk 1	Milk 2	Kidney	Liver	Muscle	Fat
Total	**90.2**	**88.4**	**96.9**	**103.6**	**102.5**	**87.1**

+ = trace

[A] combined methanol (48.7%TRR) and hexane (21.3%TRR) extracts

[B] reflux with HCl released 17.5% TRR from muscle PES.

Table 25 Characterisation and identification of ^{14}C residues in solvent extracts from milk and tissues (P-label) (Leibold and Hoffmann 2000a 1013272, Fabian and Knoell 2003a 1014911)

	Milk 1	Milk 2	Kidney	Liver	Muscle	Fat
TRR (mg eq/kg)	0.016	0.016	0.153	0.637	0.021	0.194
			%TRR			
Extracted	**60.2 (acetone /ACN)**	**50.8 (acid pH 4.2)**				
fenpropimorph	*10.0*	*14.2*				
BF421-1	-	-				
BF421-2	*26.8*	*22.1*				
BF421-2 GlcA conjugate	*3.7*					
BF421-3	*10.0*	*7.2*				
BF421-3 GlcA conjugate	*4.3*					
BF421-21	-	*6.3*				
Lactose	-	*1.0*				
Unknowns	*5.4*	-				
Extracted CH_3OH	**17.0**		**96.2**	**93.9**	**107.1**	**75.5** [A]
fenpropimorph			*14.9*	*2.3*	-	*2.9*
BF421-1			-	*10.0*	-	-
Sulphate of BF421-1			-	*1.2*	-	-
BF421-2			*40.3*	*72.6*	*78.6*	*28.7*
BF421-2 GlcA conjugate			-	*1.0*	*7.0*	*14.4*
BF421-3			*41.0*	*5.1*	*21.5*	*8.6*
BF421-3 GlcA conjugate			-	*0.8*	-	-
Unknowns				*0.9*	-	*20.9*
Unextracted	**24.4**	**32.8**	**5.2**	**6.4**	**2.7**	**5.8**
Water soluble			*1.2*	*1.4*	*0.6*	*0.9*
Total	**101.6**	**83.6**	**102.6**	**101.7**	**110.4**	**82.3**

+ = trace

[A] combined methanol (56.8%TRR) and hexane (18.8%TRR) extracts

Residues in solids unextracted with the solvent system used (M-label) were further characterised by enzymatic digestion with pronase which released 83% of unextracted ^{14}C in liver, 74% in kidney, and 94% muscle. A comparable proportion of unextracted ^{14}C (85%) was released by acid hydrolysis of muscle. The results suggest degradation of the dimethylmorpholine moiety and incorporation of ^{14}C via endogenous metabolism into proteins.

Radioactivity in urine and faeces were also identified with the results summarised below.

Urine M-label contained BF421-2 5.9%, BF421-3 51.7%, BF421-2 GlcA conjugate 6.8%, BF421-3 GlcA conjugate 0.9%, BF421-10 2.0%, sulphate of BF421-1 1.0%, BF421-19 19%, BF421-21 2.1%, BF421-24 2.8%, BF421-26 1.1%, BF421-30 1.6%.

Urine P-label contained fenpropimorph 0.4%, BF421-2 4.5%, BF421-3 51.9%, BF421-2 GlcA conjugate 7.1%, BF421-3 GlcA conjugate 1.7%, sulphate of BF421-1 2.5%, BF421-21 3.3%, BF421-22 4.6%, BF421-24 2.8%, BF421-26 0.8%, BF421-30 1.8%.

Faeces M-label contained fenpropimorph 9.8%, BF421-2 35.0%, BF421-3 20.1%, BF421-2 GlcA conjugate 1.5%, BF421-21 7.8%.

Faeces P-label contained fenpropimorph 14.3%, BF421-2 29.5%, BF421-3 16.6%, BF421-2 GlcA conjugate 1.1%, BF421-21 3.7%.

In summary, the main metabolites of fenpropimorph in goats occurred following hydroxylation of the *t*-butyl- moiety (BF421-1) which can be oxidised to give a carboxylated *t*-butyl moiety (BF421-2). Subsequently, BF421-2 can be hydroxylated at the 2-methyl group of the dimethylmorpholine ring (BF421-3). BF421-1 may be conjugated to sulphate and to glucuronic acid (sulphate or GlcA of BF421-1). Also, BF421-2 may be conjugated to glucuronic acid (GlcA of BF421-2). BF421-2 may undergo hydroxylation (BF421-24) and degradation of the t-butyl group to BF421-26. In addition, BF421-2 can undergo hydrolysis of the dimethylmorpholine-ring (BF421-21) followed by further degradation of the side chains (BF421-4). The hydrolysation of the dimethylmorpholine moiety at the ring-ether bridge followed by further metabolism of the resulting hydroxypropylamine moieties leads to the formation of C_3- and C_2-fragments, which possibly enter anabolic pathways and be incorporated into proteins or fatty acids.

BF421-3 can be oxidized to a dicarbon acid (carboxylated at the *t*-butyl moiety and the dimethylmorpholine ring, BF421-22) or hydroxylated and degraded at the *t*-butyl moiety (BF421-26). Finally, BF421-3 can be hydroxylated and conjugated to glucuronic acid (GlcA of BF421-3).

A second and minor metabolic route is cleavage of fenpropimorph, generating the dimethylmorpholine ring, which subsequently may be hydroxylated and conjugated to sulphate.

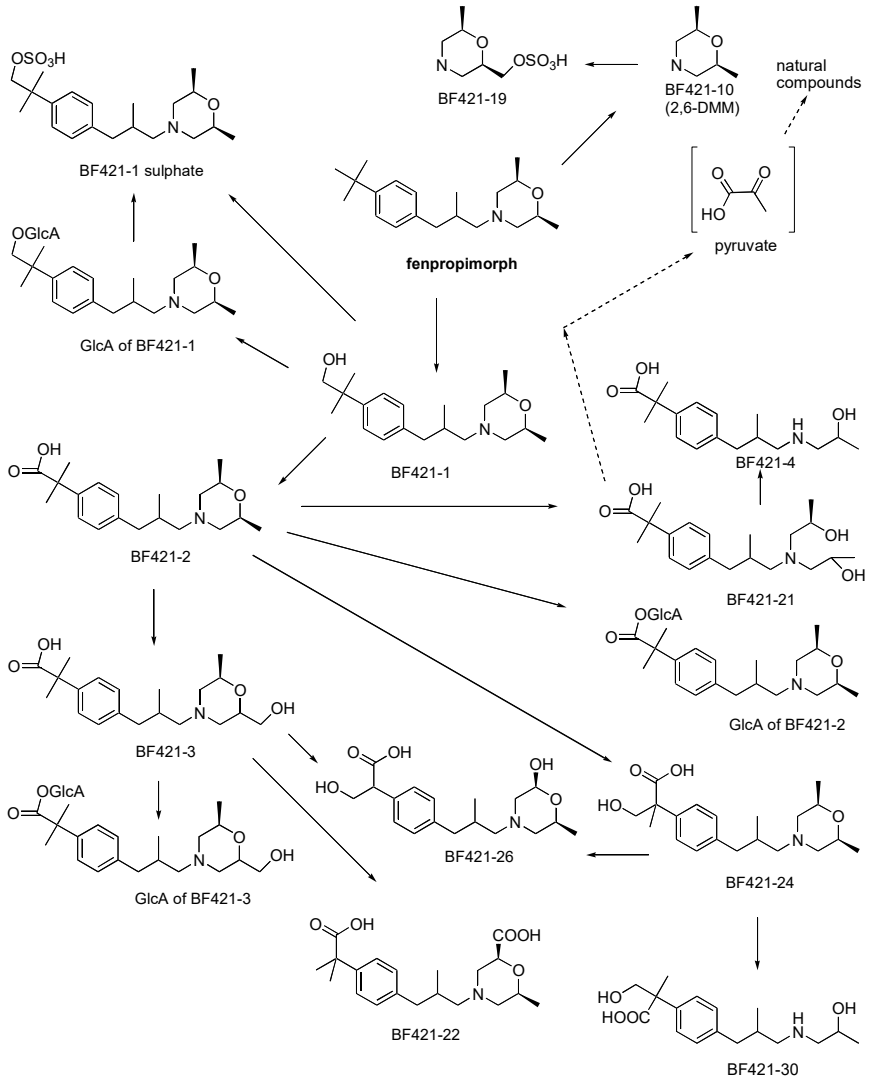


Figure 10 Metabolites of fenpropimorph identified in tissues and milk of lactating goats.

Laying hen

Ritter (1989b 0595) studied the metabolism of fenpropimorph in laying hens. Leghorn LSL Blanches hens (22 wks old, 1.50–1.59 kg bw) were dosed orally *via* intubation, once a day for a total of five doses, with [phenyl-U-^{14}C]- or [morpholine-2,6-^{14}C]-fenpropimorph at doses equivalent to 55.5 (phenyl) and 41.3 (morpholine) ppm in the diet (4 mg/kg bw). Feed consumption was 116 g/d. Laying efficiency during the dosing period was 80–90%. Egg samples were collected twice a day; excreta samples were also collected twice daily. Eggs were separated into egg whites and yolks. Hens were sacrificed five hours after the final dose, and samples of skin with subcutaneous fat and peritoneal fat, thigh and breast muscle, kidneys, liver, and gastrointestinal tract and contents were collected. Total radioactivity was determined by liquid scintillation counting. Characterisation and identification of metabolites was by 2D-TLC, HPLC and high voltage electrophoresis.

Excretion of fenpropimorph was fast, with 79.1% AD (M-label) and 83.1% AD (P-label) found in the excreta by 5 hours after the last dose. Only a small portion of the administered radioactivity was excreted in the eggs (0.2–0.4% AD) or found in tissues and blood (3.1–3.6% AD).

Table 26 Recovery of administered dose in eggs, tissues, and excreta of laying hens following dosing for four consecutive days (five doses) with [^{14}C]-fenpropimorph [A] (Ritter 1989b 0595)

Sample	M-label % AD	Residue (mg eq/kg)	P-label % AD	Residue (mg eq/kg)
Tissues/blood	**3.6**		**3.1**	
Liver		*3.92*		*2.75*
Kidneys		*2.37*		*2.81*
Fat: stomach/skin		*1.081/0.678*		*1.44/0.974*
Heart		*0.712*		*0.925*
Muscle: chest/leg		*0.243/0.337*		*0.277/0.416*
Gizzard		*1.056*		*0.404*
Brain		*0.309*		*0.157*
Ovaries		*4.161*		*1.257*
Spleen		*1.450*		*0.822*
Blood		*2.01*		*1.43*
Eggs [B]	**0.4**		**0.2**	
Yolk (5 hr after last dose)		*2.96*		*0.832*
White (5 hr after last dose)		*0.417*		*0.166*
Excreta	**79.1**		**83.1**	
Other = cage wash/debris	**2.7**		**3.6**	
Total	**85.8**		**90.0**	

[A] Assumed muscle, fat and blood represent 40, 12 and 8% bodyweight respectively

[B] whole eggs values, assuming yolk represents 1/3 and white 2/3 were 1.3 and 0.39 mg eq/kg for the M- and P-label respectively.

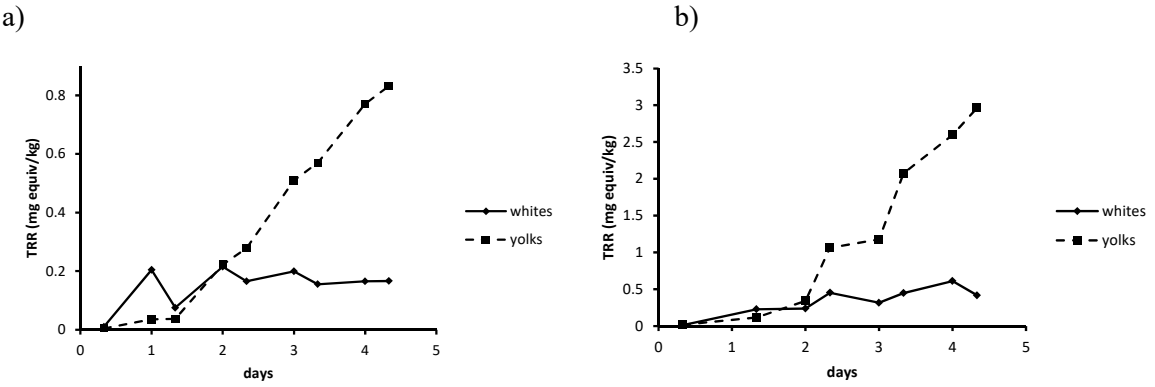


Figure 11 Residues in eggs following dosing with ^{14}C-fenpropimorph, a) P-label, b) M-label

For hens dosed with the P-label, TRR were highest in liver and kidneys, followed by fat and muscle and eggs (Table 26). TRR for the M-label were greatest in liver, egg yolk and kidneys with lower levels in the fat and 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, a pattern consistent with the physiology of egg formation (Figure 11).

A range of extraction schemes were used to further characterise residues in milk and tissues.

Eggs: White samples: proteins were precipitated by shaking with acetone and solids removed by centrifugation and filtration. The supernatant was extracted with acetone (4×) the extracts concentrated and the concentrated residue partitioned with dichloromethane.

Yolk samples: yolk was suspended in water and proteins were precipitated by shaking with acetone and solids removed by centrifugation and filtration. The supernatant was concentrated and the concentrated residue partitioned with hexane.

Liver samples were extracted with 80% methanol (4×), methanol (1×) and acetone (2×) and the pooled extracts concentrated and partitioned with hexane (1×), dichloromethane (2×) and ethyl acetate (1×), the organic phase dried over anhydrous Na_2SO_4 and analysed by TLC. Metabolites isolated from TLC plates were subjected to acid hydrolysis (2N HCl, 70 °C, 48 h).

Kidney samples were extracted with 80% methanol (4×), methanol (1×) and acetone (2×) and the pooled extracts concentrated and partitioned with hexane (2×), dichloromethane (1×) and ethyl acetate (1×), the organic phase concentrated and analysed by TLC. Metabolites isolated from TLC plates were subjected to acid hydrolysis (2N HCl, 70 °C, 48 h).

Muscle samples were extracted with 80% methanol (4×), methanol (1×) followed by Soxhlet extraction with methanol. Combined extracts were concentrated and partitioned with hexane (1×), dichloromethane (1×) and ethyl acetate (1×), the organic phase concentrated and analysed by TLC. Metabolites isolated from TLC plates were subjected to acid hydrolysis (2N HCl, 70 °C, 96 h).

Fat samples were extracted with methanol (6×) and ethyl acetate (2×). Combined extracts were concentrated and partitioned with acetone, the acetone phase concentrated and analysed by TLC. Alternatively, fat samples were extracted using chloroform/methanol (1:1 v/v). The chloroform phase was concentrated and subjected to enzymatic hydrolysis with lipase.

Skin+subcutaneous fat samples were extracted with 80% methanol (2×) and acetone (4×). Combined extracts were concentrated, separating into an upper lipid phase and a lower aqueous phase.

The ^{14}C residues remaining in solids following methanol extraction (PES) were further extracted with water or an aliquot also extracted sequentially with isohexane and water and finally incubated with pronase (pH 7, 37 °C overnight) and in the case of muscle an aliquot also extracted sequentially with isohexane, ethyl acetate, reflux with methanol and finally aliquots refluxed with 5M HCl or 5M NaOH.

Extractability with solvents and water was good for most tissues and eggs.

Organosoluble or water-soluble fractions of eggs, muscle and fat/skin contained 3–10 unidentified metabolites, however compounds were only identified for extracts of the tissues that contained the highest levels of radioactivity, liver and kidneys as well as plasma.

In liver, fenpropimorph was extensively metabolised with up to 10 metabolites identified in the organosoluble extracts but no parent compound. Fenpropimorph was detected in kidney for birds dosed with the P-label. Metabolites identified (by co-chromatography with authentic standards) were BF421-1 (plasma), BF421-2 (plasma, liver and kidney), and BF421-3 (kidney) but only at levels < 10% TRR.

Table 27 Characterisation and distribution of ^{14}C residues in eggs and tissues from laying hens dosed with [^{14}C]-fenpropimorph (mean values) (M-label) (Ritter 1989b 0595)

	Egg yolk	Egg white	Liver	Kidney	Muscle	Fat
TRR (mg eq/kg)	2.64	0.611	3.92	2.37	0.465	
			%TRR			
Extract [A]	**67.9**	**21.6**	**76.2**	**70.1**	**66.8**	**97.0**
organosoluble	58.7	1.9	54.0	32.2	35.6	54.9
fenpropimorph	-	-	-	-	-	-
BF421-2	-	-	*3.9*	-	-	-
unidentified		*1.9 (4)*	*54.0 (n=6 21.1)*		*35.6 (n=5 8.8)*	
Water soluble	8.3	19.7	22.2	37.9	31.2	42.1
unidentified			*22.2 (n=8 5.9)*	*37.9 (n=3 15)*		
Unextracted Protein pellet	**33.0**	**78.4**	**23.8**	**29.9**	**33.2**	**3.0**

[A] extraction solvents: Eggs: acetone; liver, kidney: 80% methanol+methanol+acetone; muscle 80% methanol, methanol, Soxhlet; fat methanol+ethyl acetate; skin+subcutaneous fat 80% methanol+acetone

Table 28 Characterisation and distribution of ^{14}C residues in eggs and tissues from laying hens dosed with [^{14}C]-fenpropimorph (mean values) (P-label) (Ritter 1989b 0595)

	Egg yolk	Egg white	Liver	Kidney	Muscle (leg)	Fat
TRR (mg eq/kg)	0.832	0.215	2.75	2.81	0.277	1.44
			%TRR			
Extract [A]	**62.8**	**79.7**	**87.1**	**87.4**	**97.1**	**98.9**
organosoluble	56.9	4.3	62.8	34.4	72.3	66.2
fenpropimorph	-	-	-	*11*	-	-
BF421-2	-	-	-	*3.5*	-	-
BF421-3	-	-	-	*1.9*	-	-
unidentified		*4.3 (4)*	*62.8 (n=10 13)*	*34.4 (n=6 4.7)*	*72.3 (n=7 23.6)*	*76.1 (n=3 45.4)*
Water soluble	5.9	75.4	24.3	53.0	24.8	32.7
unidentified			*24.3 (n=8 6.3)*	*53.0 (n=5 21.2)*	*24.8 (n=5 7.4)*	
Unextracted Protein pellet	**37.2**	**20.3**	**12.9**	**12.6**	**2.9**	**1.1**

[A] extraction solvents: Eggs: acetone; liver, kidney: 80% methanol+methanol+acetone; muscle 80% methanol, methanol, Soxhlet; fat methanol+ethyl acetate; skin+subcutaneous fat 80% methanol+acetone

In a separate study Leibold and Hoffmann (2000b 1013365, also Hartl 2002a 1014901) assessed the rates and routes of excretion and distribution of ^{14}C-fenpropimorph in excreta, blood, eggs, and tissues of laying hens after oral dosing. The metabolism and distribution of fenpropimorph was investigated in laying hens (Brown Leghorn, 27 wks, 1.9 kg bw) following repeated oral administration of [phenyl-U-^{14}C]- or [morpholine-2,(6)-^{14}C]-fenpropimorph. ^{14}C- Fenpropimorph was administered daily on 10 consecutive days at a nominal dose level of 12.3-14 ppm feed, which corresponded to an actual daily dose of 0.81 mg/kg body weight for both dose groups. Mean feed consumption was 118–127 g/d. Laying efficiency was 93–96%. The test substance was dissolved in olive oil and administered orally by gavage with a syringe. Excreta were collected in 24 hour intervals until sacrifice. Eggs were sampled twice a day, in the morning prior to dose administration and in the late afternoon (except for the weekend when records on egg production were made only once per day). The animals were sacrificed 23 hours after the last administration and samples of liver, blood, adipose tissue, chest and leg muscle, and the gastrointestinal tract (skin and content) were taken and pooled according to dose group.

Following administration of ^{14}C-fenpropimorph to laying hens, the radioactivity was rapidly and almost completely excreted within 24 hours (Table 29)

Radioactivity recovered from excreta together with cage wash amounted to 84.9% and 106.5% of administered radioactivity for the M-label and the P-label, respectively. Radioactivity in eggs was found to be in the range of 0.28% (P-label) to 1.65% (M-label) of the administered dose. At sacrifice, the highest concentrations of radioactivity were found in the contents of the gastrointestinal tract. Accountability was 87.6% (M-label) and 107.4% of the total administered dose (P-label).

Table 29 Material balance after administration of ^{14}C- fenpropimorph to laying hens

Matrix	Material balance in % of total administered dose	
	M-label	P-label
Tissues and blood	**0.55**	**0.24**
Blood	*0.01*	*0.00*
Liver	*0.16*	*0.06*
Leg muscle	*0.14*	*0.05*
Chest muscle	*0.09*	*0.03*
Adipose tissue	*0.15*	*0.10*
Eggs	**1.65**	**0.28**
Excreta	**83.66**	**105.86**
Other	**1.74**	**1.05**
GI Tract (Skin)	*0.22*	*0.18*
GI Tract (Contents)	*0.27*	*0.20*
Cage wash	*1.25*	*0.67*
Total	**87.60**	**107.41**

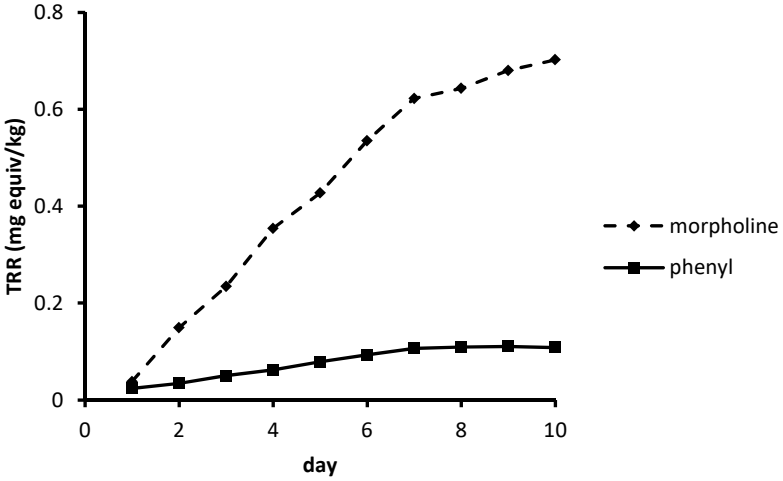


Figure 12 Residues in eggs following dosing with ^{14}C-fenpropimorph,

The TRR in tissues ranged from 0.097 mg eq/kg to 0.630 mg eq/kg for the M-label and from 0.036 mg/kg to 0.337 mg eq/kg for the P-label with highest residues in liver and fat and lowest residues in muscle. In eggs, by the end of dosing the TRRs were 0.639 mg eq/kg and 0.107 mg eq/kg, for the M- and P-label, respectively (Figure 12).

Tissue samples were extracted with methanol and iso-hexane or in some cases with a mixture of methanol/iso-hexane and in the case of M-label eggs also with acetonitrile. The methanol extracts were further purified by partitioning with iso-hexane. For metabolite identification, the resulting iso-hexane extracts and phases were subjected to a combined saponification/esterification procedure (reflux with ethanol/H_2SO_4). Radioactivity unextracted using solvent was investigated further by subjecting samples to pronase digestion (37 °C overnight) with the resulting enzyme incubates directly analysed by radio-HPLC.

The extractability of all matrices with methanol and iso-hexane is presented in Table 30. Extractability with methanol was generally low to moderate, ranging between 18 and 56% TRR depending on label and tissue type. The subsequent extraction with iso-hexane was especially

successful for fat of both labels (extracting 60–83% TRR), for the P-label muscle (66% TRR) and the M-label egg (45% TRR). The overall extracted radioactivity using methanol + iso-hexane ranged from 46 to 101%TRR in edible matrices for the M-label and from 63 to 92% TRR for the P-label.

Table 30 Extractability of edible matrices after dosing of laying hens with ^{14}C-fenpropimorph

Matrix	TRR (mg eq/kg)	Extracted (%TRR)				Unextracted (%TRR)
		Methanol	Iso-hexane	Acetonitrile	Total	
M-label						
Eggs	0.639	28.3	44.5	2.8	75.6	24.8
Muscle	0.097	35.2	10.5	n.p.	45.7	58.8
Fat	0.435	18.8	82.6	n.p.	101.4	1.3
Liver	0.630	49.4	10.4	n.p.	59.8	42.3
P-label						
Eggs	0.107	53.3	24.8	n.p.	78.1	20.8
Muscle	0.036	18.0	65.5	n.p.	83.5	10.9
Fat	0.337	31.7	60.1	n.p.	91.8	2.4
Liver	0.250	55.6	7.7	n.p.	63.3	36.0

n.p.: not performed

Parent fenpropimorph was a major component of radioactivity in eggs, fat and liver of the P-label, accounting for 17.7, 18.0 and 28.8% TRR, respectively, whereas in the M-label tissues fenpropimorph accounted for only 2.7% TRR in egg, 13.3% TRR in fat and 11.2% TRR in liver. In muscle, fenpropimorph represented 5.1% (M-label) and 5.5% (P-label) of the TRRs. Other compounds identified were the acid metabolite BF421-2 (eggs, muscle and liver) which was a minor component in all tissues of the morpholine label (egg 1.8% TRR; muscle 0.5% TRR; liver: 1.8% TRR), but accounting for 16% TRR in eggs, 3.4% TRR in muscle and 7.5% TRR in the liver for the P-label. BF421-3 was also identified in hens in amounts similar to BF421-2, representing 13.4%, 2.9% and 8.1% TRR in eggs, muscle and liver of the phenyl label and 0.8%, 1.4% and 1.3% TRR in the respective matrices of the M-label.

The P-label specific metabolites BF421-12 and BF421-16, were identified in eggs, muscle and fat, accounting for 5.1%, 2.4% and 4.5% of the TRR. Another metabolite exclusively found in the P-label tissues was BF421-18, which was detected in the muscle in free form (4.5% TRR) and identified in all tissues as its ethyl ester after saponification/esterification, indicating an incorporation into/conjugation with endogenous lipids. Radioactive triacyl glycerides represented the major metabolites in muscle and fat, accounting for 52.5% and 52.4% TRR. They were also detected in eggs and liver, at 9.5% TRR and 1.8% TRR, respectively.

Of the M-label specific metabolites that could be unambiguously identified, cis-dimethyl morpholine BF421-10 was found in eggs (1.5% TRR), muscle (3.4% TRR) and liver (3.3% TRR). By far the most predominant metabolites identified in the morpholine label experiment were lipids in fat (79.5% TRR) and egg (45.2% TRR) with lower levels in muscle (7.6% TRR) and liver (9.8% TRR). These lipid metabolites were identified after a saponification/esterification reaction via their fatty acid methyl esters to represent triacyl glycerides primarily containing the most abundant endogenous fatty acids in hens, palmitic, oleic, and stearic acid.

The ^{14}C unextracted with solvent of all matrices except fat contained considerable amounts of radioactivity and were subjected to enzymatic digestion with pronase which solubilised 82 to 100% of the unextracted radioactivity, indicating the unextracted ^{14}C was associated with proteins. In the case of the P-label liver, pronase treatment liberated only 51% of the “bound” radioactivity. The remainder was subjected to acid hydrolysis which released all the remaining radioactivity.

Analysis of the ^{14}C liberated by pronase treatment allowed the identification of minor amounts of parent fenpropimorph, and metabolites BF421-3, BF421-12 and BF421-16 in the egg and the identification of BF421-2 and BF421-3 in the liver of the P-label experiment. Solubilised residues of the morpholine label egg, muscle and liver could not be unambiguously identified, however they

were comprised of numerous compounds, presumably small polar molecules and it is likely that a considerable portion being endogenous molecules.

Table 31 Identification of ^{14}C residues in eggs and tissues from laying hens dosed with [^{14}C]-fenpropimorph (mean values) (M-label)

	Egg	Liver	Muscle	Fat
TRR (mg eq/kg)	0.639	0.630	0.097	0.435
		%TRR		
Extracted				
fenpropimorph	*2.7*	*11.2*	*5.1*	*13.3*
BF421-2	*1.8*	*1.8*	*0.5*	-
BF421-3	*0.8*	*1.3*	*1.4*	-
BF421-10	*1.5*	*3.3*	*3.4*	-
Triacyl glycerides [A]	*45.2*	*9.8*	*7.6*	*79.5*
Unextracted				

[A] Containing palmitic, stearic and oleic acid, identified as ethyl esters after hydrolysis and esterification

Table 32 Identification of ^{14}C residues in eggs and tissues from laying hens dosed with [^{14}C]-fenpropimorph (mean values) (P-label)

	Egg [A]	Liver [A]	Muscle	Fat
TRR (mg eq/kg)	0.107	0.250	0.036	0.337
		%TRR		
Extracted				
fenpropimorph	*17.7*	*28.8*	*5.5*	*18.0*
BF421-2	*16.0*	*7.5*	*3.4*	-
BF421-3	*13.4*	*8.1*	*2.9*	-
BF421-12	*5.1* [C]	-	*2.4* [C]	*4.5* [C]
BF421-16				
BF421-18	-	-	*4.5*	-
Triacyl glycerides [B]	*9.5*	*1.8*	*52.5*	*52.4*
Unextracted				

[A] Quantities represent the sum of metabolites in extracts and metabolites released from the post extraction solids by pronase digestion

[B] Containing BF421-18, identified as ethyl esters after hydrolysis and esterification

[C] Quantified as sum since they are not resolved with HPLC Method

In summary, after administration of the test compound, most of the radioactivity was excreted. There was no indication of accumulation of ^{14}C-fenpropimorph in hen eggs and tissues. Besides endogenous lipids, the main component was the parent fenpropimorph and to some extent the metabolite BF421-2.

Metabolism of fenpropimorph in laying hen proceeds via:

- Hydroxylation/oxidation of the *t*-butyl group of the phenyl ring
- Cleavage between the methylpropyl group and the morpholine ring
- Cleavage between the methylpropyl group and the phenyl ring
- Cleavage of the morpholine ring
- Hydroxylation of the methyl group of the morpholine ring

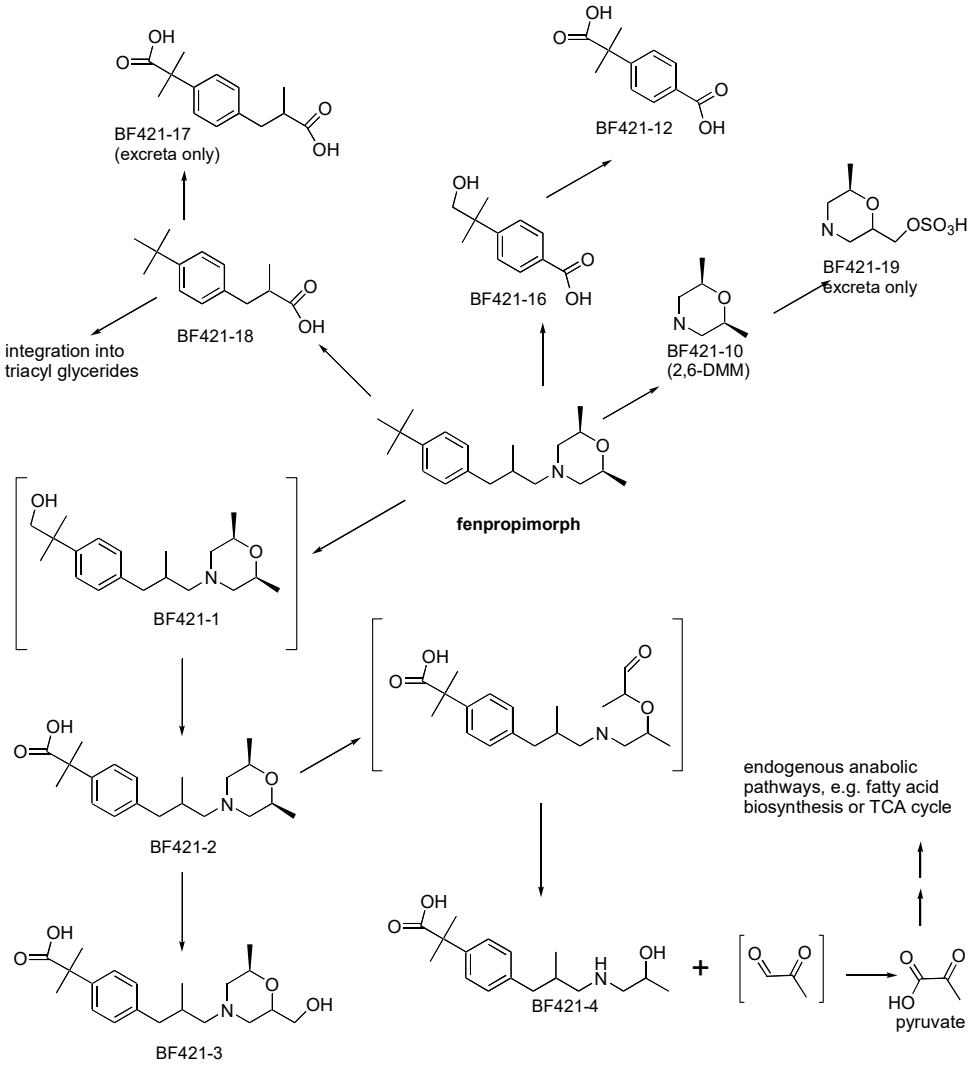


Figure 13 Metabolites of fenpropimorph identified in laying hen tissues and eggs

The metabolism of fenpropimorph in plants is similar to that in animals to the extent that oxidation of the phenyl ring *t*-butyl group is the first step followed by degradation of the morpholine ring. There are differences in that fenpropimorph is generally the main residue in plants but relatively minor in animals. A number of plant metabolites and/or their conjugates, BF421-2-Me, BF421-7, BF 421-13, BF421-14 and BF421-15 as well as BF421-36 have not been reported in animals although in edible plant parts most of these would be at relatively low levels.

ENVIRONMENTAL FATE

The FAO Manual on the Submission and Evaluation of Pesticide Residues Data for the Estimation of Maximum Residue Levels in Food and Feed (2009) explains the data requirements for studies of environmental fate. The focus should be on those aspects that are most relevant to MRL setting. For fenpropimorph, supervised residue trials data are available for banana, sugar beet and cereal crops. Aerobic degradation in soil is relevant, as well as the normal requirements for hydrolysis, photolysis and rotational crop studies.

The Meeting received information on soil aerobic metabolism, aqueous hydrolysis and soil photolysis properties of fenpropimorph. Studies were also received on the behaviour of [^{14}C]-fenpropimorph in a confined rotational crop situation.

Fenpropimorph residues are not persistent in soils and it is unlikely that fenpropimorph residues in soils resulting from recommended uses make a significant contribution to the residues in succeeding crops.

Confined rotational crop studies

A confined rotational crop study was conducted on a sandy loam soil (6.6% sand, 35.7% silt, 57.7% clay; pH 6.6; 3.0% organic matter; 42.2 mEq/100 g CEC) on which wheat plants (cv *Probus* winter wheat) had been treated with [benzylic-^{14}C]-fenpropimorph at 720 g ai/ha (Pryde and Etterli, 1980 10102). The top 5–10 cm depth of soil was used to grow rotational crops (spinach cv *Glares*, sugar beet cv *Monohil* and wheat cv *Probus*) sown into the soil at 30, 113, 141 and 337 days after the application (DAA). The treated boxes were maintained in a **greenhouse** with both plants and soil collected at various intervals for measurement of radioactivity by combustion analysis. For wheat, only mature plants were analysed as no grain was produced under the greenhouse conditions. The results are shown in Table 33.

Table 33 Distribution of radioactivity in soil and rotational crops after treatment of soil with [^{14}C]-fenpropimorph (Pryde, Etterli, 1980 10102)

DAA	Age of crops (days)	Average total residue (mg eq/kg)			
		Soil	Spinach[A]	Sugar beet[B]	Wheat mature plants[C]
7	-	0.42	-	-	-
102	10	-	0.01	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[D] 0.07[E]		0.01 green plant 0.004 mature beet	< 0.02

[A] Fresh wt. basis, 90.6% moisture content

[B] Fresh wt. green plant 19.1% or mature beet (without leaves or roots), 20.6% moisture content

[C] Fresh wt., 19.6% moisture content

[D] Soil from sugar beet pot

[E] Soil from wheat pot

Rabe (2003a, 1001380) conducted a confined rotational crop study on a loamy sand soil (60% sand, 29% silt, 11% clay; pH 7.7; 0.7% organic matter; 6.9 mEq/100 g CEC) treated with [phenyl-U-^{14}C]- and [morpholine-2,(6)-^{14}C]-fenpropimorph at 1500 g ai/ha. Radish (var *Cherry Belle*), lettuce (var not specified) and wheat (var *Yecora Rojo*) were sown into the soil at 30, 120 and 365 days after the soil application (DAA). The treated boxes were maintained in a screened enclosure. Mature crops were harvested, processed and analysed by combustion and radioactivity measurement for the determination of the TRRs in the raw agricultural commodities. In addition, soil samples were taken after application, ploughing and after harvest of mature crops.

Plant samples were extracted with methanol and solids remaining after extraction were further treated with 1% aqueous ammonia and treated with different enzymes (Macerozyme, amylase, tyrosinase/laccase, baker's yeast), depending on the level of the radioactive residues, to release part of the remaining radioactivity. Methanol extracts of all samples, and where possible, the ammonia and enzyme extracts were analysed by HPLC.

TRRs in the soil after application (8.86–12.05 mg eq/kg), decreased after a soil aging period of 30 days and ploughing (0.696–0.850 mg eq/kg) (Table 34). During the next 5 months until harvest

of the mature crops the residues in the soil declined significantly to a level of about 0.1–0.3 mg eq/kg in both labels. After longer plant back intervals (PBIs) of 120 and 365 days, only a slight further decrease of levels of ^{14}C in soil was observed.

Table 34 Quantitative distribution of radioactive residues in soil after treatment with ^{14}C-fenpropimorph (M- and P-label)

Sample /experiment	TRR P-label (mg eq/kg)	TRR M-label (mg eq/kg)
After application		
0 DAA	8.858	12.046
Soil after aging and ploughing		
30 DAA	0.850	0.697
120 DAA	0.158	0.278
365 DAA	0.214	0.212
Soil after harvest of crop		
Plant back interval 30 days		
Lettuce	0.218	0.213
Radish	NS	0.218
Wheat	0.116	0.262
Plant back interval 120 days		
Lettuce	0.185	0.151
Radish	0.130	0.285
Wheat	0.166	0.177
Plant back interval 365 days		
Lettuce	0.155	0.181
Radish	0.093	0.140
Wheat	0.118	0.196

NS = not sampled

The distribution of the TRRs, the methanol extracted radioactive residues are summarized in Table 35 and Table 36. A comparison of the two labels showed that over all plant back intervals and rotational crop matrices the TRRs in the M-label were significantly higher than for the P-label.

The TRRs in lettuce head were highest in the samples of the 30 days PBI (0.067–0.209 mg eq/kg) declining with subsequent plant back periods. TRR in radish leaves (30 PBI: 0.060–0.306 mg eq/kg,) was higher than in radish roots (30 PBI: 0.043–0.101 mg eq/kg). In both matrices and both labels, the TRRs were lower for the longer PBIs. In wheat forage residues (highest 30 PBI 0.210–0.892 mg eq/kg) declined for the longer PBIs. In wheat straw and chaff, the TRRs were higher (0.555–3.065 mg eq/kg, straw) in the samples of the 30 days PBI and were 0.015–0.364 mg eq/kg for the 365 days PBI. In wheat grain TRRs at the different PBIs followed the order 120 > 30 > 365 days.

Table 35 Summary of major components in the follow crops grown in soil previously treated with ^{14}C fenpropimorph (M-label)

	Lettuce 56 DAA	Radish leaf 64 DAA	Radish root 64 DAA	Wheat forage 56 DAA	Wheat grain 116 DAA	Wheat straw 116 DAA	Wheat chaff 116 DAA
30 day PBI							
TRR (mg eq/kg)	0.126	0.227	0.044	0.647	0.021	1.168	0.375
				%TRR			
Extracted CH_3OH	**60.3**	**75.8**	**43.7**	**76.3**	**14.0**	**38.1**	**25.3**
Fenpropimorph	-	-	*8.2*	*0.3*			
Sugar	*7.5*	*2.0*	*8.9*	*0.4*	*4.1*	*1.1*	*1.5*
2,6-DMM [B]	*26.8*	*34.2*	*10.8*	*49.2*	*3.5*	*18.9*	*9.2*
BF421-1-Glc	*3.4*					*0.7*	
BF421-1	*0.2*		*1.1*	*1.8*		*0.3*	
Metabolite 12				*3.8*		*1.1*	
Metabolite 3 [C]	*4.5*	*4.4*		*3.9*	*1.5*	*5.1*	*4.7*
Polar region	*9.8*	*18.6*	*3.8*	*5.8*	*0.7*	*7.4*	*6.0*
Medium polar	*9.1*	*16.7*	*12.1*	*10.8*	*3.7*	*3.5*	*3.8*
Non-polar					*0.5*		

	Lettuce 56 DAA	Radish leaf 64 DAA	Radish root 64 DAA	Wheat forage 56 DAA	Wheat grain 116 DAA	Wheat straw 116 DAA	Wheat chaff 116 DAA
Unextracted	**39.7**	**16.5**	**53.7**	**14.1**	**86.0**	**58.7**	**72.6**
NH_4OH	*14.1*	*7.1*	*0.1*	*4.2*	*22.8*	*13.7*	*19.8*
Macerozyme	*7.5*	*5.0*	*25.5*	*2.8*	*44.6*	*8.3*	*12.4*
Amylase	*3.0*	*0.9*	*5.2*	*1.1*	*11.2*	*2.9*	*4.3*
Sugar	*8.5*	*4.5*	*29.4*	*0.6*	*63.3*	*1.9*	*2.4*
2,6-DMM [B]	*16.1*	*7.7*	*1.4*	*6.0*	*6.7*	*18.4*	*19.0*
BF421-1-Glc						*0.4*	*0.5*
BF421-1						*0.5*	*0.3*
Metabolite 3 [C]						*0.5*	*3.7*
Polar					*4.6*	*1.4*	*4.3*
Medium polar				*1.8*		*2.0*	*4.5*
NaOH	*NA*	*NA*	*NA*	*NA*	*NA*	*17.0*	*21.4*
Lignin super	*NA*	*NA*	*NA*	*NA*	*NA*	*10.3*	*14.0*
Lignin precipitate	*NA*	*NA*	*NA*	*NA*	*NA*	*7.2*	*8.0*
120 day PBI							
TRR (mg eq/kg)	0.027	0.039	0.021	0.052	0.032	0.239	0.454
				%TRR			
Extracted CH_3OH	**38.5**	**46.9**	**48.1**	**38.0**	**5.3**	**27.4**	**41.1**
Fenpropimorph							
Sugar	*7.3*	*3.9*	*49.0*	*3.7*	*2.8*	*2.9*	*4.1*
2,6-DMM [B]	*25.2*	*31.3*		*29.1*	*0.8*	*14.6*	*24.9*
Metabolite 3 [C]	*2.2*	*1.3*		*1.5*	*0.4*	*2.9*	*4.9*
Polar region	*0.9*	*4.0*		*0.5*	*0.4*	*4.3*	*4.5*
Medium polar	*2.8*	*6.5*		*3.1*	*1.0*	*2.6*	*2.7*
Unextracted	**60.0**	**53.1**	**51.9**	**62.0**	**94.7**	**72.6**	**57.4**
NH_4OH	*9.6*	*NA*	*4.5*	*3.1*	*11.6*	*4.5*	*12.2*
Macerozyme	*11.9*	*NA*	*11.4*	*11.6*	*46.4*	*7.2*	*6.6*
Amylase	*4.4*	*NA*	*5.0*	*5.8*	*11.8*	*3.4*	*3.4*
sugar	*23.5*		*10.5*	*11.3*	*69.8*	*5.7*	*9.6*
2,6-DMM [B]	*2.3*			*5.0*		*9.4*	*10.2*
Metabolite 3 [C]							*0.4*
Polar				*4.3*			*0.2*
Medium polar							*1.8*
NaOH	*NA*	*NA*	*NA*	*28.2*	*1.3*	*39.2*	*2.3*
Lignin super	*NA*	*NA*	*NA*	*20.2*	*5.0*	*20.1*	*33.3*
Lignin precipitate	*NA*	*NA*	*NA*	*9.6*	*0.8*	*20.6*	
365 day PBI							
TRR (mg eq/kg)	0.007	0.006	0.006	0.024	0.005	0.156	0.060
				%TRR			
Extracted CH_3OH	**50.3**	**41.2**	**57.7**	**55.8**	**3.7**	**42.8**	**31.3**
Fenpropimorph							
Sugar	*37.2*	*5.9*	*57.7*	*3.3*	*3.7*	*2.4*	*8.1*
Metabolite 3 [C]		*2.5*		*3.8*		*6.2*	*5.7*
Polar region		*2.2*		*1.9*		*5.8*	*7.6*
Medium polar		*11.1*		*4.9*		*3.4*	*2.5*
Unextracted	**49.7**	**58.8**	**42.3**	**44.2**	**96.3**	**57.2**	**68.7**
NH_4OH	*NA*	*NA*	*NA*	*NA*	*14.1*	*6.7*	*20.6*
Macerozyme	*NA*	*NA*	*NA*	*NA*	*49.1*	*3.1*	*8.5*
Amylase	*NA*	*NA*	*NA*	*NA*	*16.1*	*1.7*	*7.7*
sugar					*65.2*	*6.5*	*9.3*
2,6-DMM [B]						*4.8*	*20.4*
Polar						*0.2*	
Laccase/Tyrosinase	*NA*	*NA*	*NA*	*NA*	*NA*	*2.2*	*3.9*
NaOH	*NA*	*NA*	*NA*	*NA*	*NA*	*5.5*	*6.7*
Lignin super	*NA*	*NA*	*NA*	*NA*	*NA*	*27.6*	*38.0*
Lignin precipitate	*NA*	*NA*	*NA*	*NA*	*NA*		

[A] Solubilised ^{14}C after treatment with NH_4OH, Macerozyme and Amylase were subject to HPLC

[B] 2,6-DMM is only tentatively identified as reanalysis of the wheat and sugar beet metabolism samples showed the HPLC system used (HPLC 1 in the metabolism study and referred to as HPLC 2 in the rotational crop study) does not allow unambiguous identification. Using the same HPLC system as for the rotational crop study the polar peak attributed to 2,6-DMM was sometimes found to be 2,6-DMM (wheat hay and straw), fructose (sugar beet roots) or a mixture of 2,6-DMM and fructose (wheat grain).

[C] "Metabolite 3" is thought to be an amino acid based on ^{13}C NMR.

Table 36 Summary of major components in the follow crops grown in soil previously treated with ^{14}C fenpropimorph (P-label)

	Lettuce 50 DAA	Radish leaf 76 DAA	Radish root 76 DAA	Wheat forage 60 DAA	Wheat grain 95 DAA	Wheat straw 95 DAA	Wheat chaff 95 DAA
30 day PBI							
TRR (mg eq/kg)	0.055	0.027	0.015	0.131	0.005	0.358	0.078
				%TRR			
Extracted CH_3OH	**82.3**	**45.2**	**33.7**	**62.1**	**11.0**	**64.4**	**57.6**
Fenpropimorph	*22.7*	*1.7*	*15.4*	*23.7*	*1.5*	*1.4*	
Sugar	*20.5*	*32.2*	*15.8*		*5.4*	*0.9*	*11.1*
Metabolite 9		*3.4*					
BF421-1-Glc	*18.9*			*14.0*		*21.5*	*18.0*
BF421-1		*2.5*		*15.5*		*29.6*	*19.2*
Metabolite 14	*1.4*	*3.6*					
Polar region	*2.4*						
Medium polar	*5.9*	*1.5*	*1.2*			*8.6*	*9.3*
Non-polar	*10.5*	*0.3*	*1.4*	*9.0*	*4.1*	*2.4*	
Unextracted	**14.9**	**50.4**	**61.0**	**38.1**	**89.0**	**41.2**	**46.0**
NH_4OH	*NA*	*12.3*	*NA*	*9.9*	*NA*	*12.2*	*8.8*
Macerozyme	*NA*	*8.8*	*NA*	*5.5*	*NA*	*4.0*	*3.7*
Amylase	*NA*	*3.2*	*NA*	*2.8*	*NA*	*2.4*	*1.4*
Sugar		*23.7*		*12.6*		*01.3*	*12.0*
BF421-1-glc						*3.7*	
BF421-1				*0.7*		*6.2*	
polar				*0.6*		*0.6*	
Medium polar		*0.6*		*1.4*		*1.0*	
Non-polar				*0.8*		*1.1*	
Laccase/Tyrosinase	*NA*	*NA*	*NA*	*NA*	*NA*	*1.6*	*NA*
NaOH	*NA*	*NA*	*NA*	*NA*	*NA*	*2.1*	*NA*
120 day PBI							
TRR (mg eq/kg)	0.026	0.009	0.008	0.023	0.012	0.060	0.083
				%TRR			
Extracted CH_3OH	**43.4**	**23.1**	**35.8**	**22.2**	**2.7**	**12.7**	**14.8**
Fenpropimorph			6.6				
Sugar	40.9	23.1	29.2	13.3	2.7	7.2	12.5
BF421-1-Glc						1.5	1.1
Metabolite 14				3.5			
Polar region						1.7	1.7
Medium polar				6.4		2.4	0.9
Non-polar						2.4	
Unextracted	**56.8**	**76.9**	**64.2**	**77.8**	**97.3**	**87.3**	**85.2**
NH_4OH	14.2	NA	NA	7.9	15.4	7.6	10.0
Macerozyme	4.7	NA	NA	12.9	49.8	4.7	4.1
Amylase	3.0	NA	NA	5.0	3.1	3.5	3.1
Sugar	*8.6*			*23.8*	*62.7*	*15.8*	*17.2*
BF421-1-glc	*4.4*						
BF421-1	*7.2*						
polar	*1.0*			*2.0*	*2.5*		
Medium polar	*0.7*						
Laccase/Tyrosinase	NA	NA	NA	2.4	NA	6.7	2.8
NaOH	NA	NA	NA	2.5	3.8	5.4	3.9
Lignin super	NA	NA	NA	34.3	NA	51.8	51.5
Lignin precipitate	NA	NA	NA	NA	NA	NA	NA
365 day PBI							
TRR (mg eq/kg)	**0.014**	**0.015**	**0.011**	**0.043**	**0.138**	**0.364**	**0.191**
				%TRR			
Extracted CH_3OH	**50.3**	**41.2**	**57.7**	**55.8**	**3.7**	**42.8**	**31.3**
Unextracted	**49.7**	**58.8**	**42.3**	**44.2**	**96.3**	**57.2**	**68.7**

Solubilised ^{14}C after treatment with NH_4OH, Macerozyme and Amylase were subject to HPLC

Lettuce

In the extract of the 30 day PBI sample of the P-label, fenpropimorph (0.015 mg/kg, 22.7% TRR), a sugar (0.014 mg/kg, 20.5% TRR), the glycoside of the BF421-1 (0.013 mg/kg, 18.9% TRR) and the malonylglucoside of BF421-1 (0.001 mg/kg, 1.4% TRR) were the main components identified. In the methanol extract of the 120-day PBI sample only the sugar compound (40.9% TRR) was identified. In the methanol extracts of M-label samples at the 30 day PBI 2,6-DMM (0.056 mg/kg, 26.8% TRR), a sugar (0.016 mg/kg, 7.5% TRR), BF421-1-Glc (0.007 mg/kg, 3.4% TRR) and "Metabolite 3" (0.009 mg/kg, 4.5% TRR) were the main components identified.

On further examination of ^{14}C unextracted with methanol, 40–61.4% of the ^{14}C present was released following sequential ammonia, Macerozyme and amylase treatment. The greatest part of which consisted of the sugar compound and 2,6-dimethylmorpholine (M-label experiment only).

Radish

In the extract radish leaves of the 30 day PBI P-label five metabolites accounted for 93% of the extracted ^{14}C; a sugar compound (0.019 mg/kg, 32.2% TRR), fenpropimorph (0.001 mg/kg, 1.7% TRR), BF421-1 (0.001 mg/kg, 2.5% TRR), the malonylglucoside of BF421-1 (0.002 mg/kg, 3.6% TRR) and the malonyldiglucoside BF421-1 (0.002 mg/kg, 3.4% TRR).

In the methanol extracts of the M-label, 2,6-DMM (0.102 mg/kg, 34.2% TRR), the sugar compound (0.006 mg/kg, 2.0% TRR) and "Metabolite 3" (0.013 mg/kg, 4.4% TRR) were the main components. For both labels, the treatment of the solids remaining after extraction sequentially with ammonia and Macerozyme and amylase released mainly the sugar compound with 2,6-DMM (M-label only). "Metabolite 3" was proposed to be an amino acid containing natural compound based on its NMR spectra though its identity was not established.

In the radish root extracts of the P-label fenpropimorph (0.001–0.007 mg/kg, 6.6–15.4% TRR) and the sugar compound (0.007 mg/kg, 15.8–29.2% TRR) were the main components found while for the morpholine label the major part of the extracted ^{14}C was 2,6-DMM (0.011 mg/kg, 10.8% TRR) and the sugar compound (0.009 mg/kg, 8.9% TRR) at the 30 day PBI, and the sugar compound at both the 120 day PBI (0.021 mg/kg, 49.0% TRR) and 365 day PBI (0.006 mg/kg, 57.7% TRR). Treatment of the solids remaining after extraction with ammonia and Macerozyme and amylase released mainly the sugar compound.

Wheat

In the methanol extract of the 30 day PBI wheat forage sample of the P-label fenpropimorph (0.050 mg/kg, 23.7% TRR), the metabolite BF421-1 (0.033 mg/kg, 15.5% TRR) and the glucoside of BF421-1 (0.029 mg/kg, 14.0% TRR) were the main components. Nearly the half of the methanol extract of the 30 PBI sample of the M-label consisted of 2,6-DMM (0.417 mg/kg, 49.2% TRR) with the sugar compound, fenpropimorph, BF421-1 and the diglucoside of BF421-1 present in small amounts. 2,6-DMM (29.1–41.8% TRR) was also the main metabolite in the methanol extracts of the samples of the longer PBIs. The sugar compound and "Metabolite 3" were found in very small amounts in these extracts. Treatment of the solids remaining after extraction with ammonia and Macerozyme and amylase released mainly the sugar compound with 2,6-DMM also found in the case of M-label samples.

In the case of straw the extract of the 30 day PBI sample of the P-label contained two main components, BF412-1 (0.164 mg/kg 29.6% TRR) and its glucoside (0.119 mg/kg, 21.5% TRR), with small amounts of fenpropimorph (0.008 mg/kg, 1.4% TRR) and the sugar compound (0.005 mg/kg, 0.9% TRR). The extract of the 120-day PBI sample contained the sugar compound (0.038 mg/kg, 7.2% TRR) and the glucoside of BF421-1 (0.008 mg/kg, 1.5% TRR) and these represented about 60% of the radioactivity in the extract.

The main components in the methanol extract of the 30-day PBI sample (M-label) were dimethylmorpholine (0.579 mg/kg, 18.9% TRR) as well as small amounts of BF421-1, its glucoside and its diglucoside and "Metabolite 3". The methanol extracts of the 120 and 365 day PBI samples

contained the sugar compound (120 PBI: 0.025 mg/kg, 2.9% TRR; 365 PBI 0.009 mg/kg, 2.4% TRR), 2,6-DMM (120 PBI: 0.127 mg/kg, 14.6% TRR; 365 PBI: 0.088 mg/kg, 24.3% TRR) and "Metabolite 3" (120 PBI: 0.025 mg/kg, 2.9% TRR; 365 PBI: 0.023 mg/kg, 6.2% TRR). Further examination of the remaining solids released an additional 18.2–42.7% of the radioactive residues present, which consisted of the same metabolites as found in the methanol extracts. It is possible the high residual radioactivity after harsh treatment of post-extraction solids is due to the incorporation of some small degradation products of fenpropimorph in natural polymers.

The extractability with methanol of wheat grain samples was poor (2.7–14.0%). The methanol extracts of the samples of the P-label consisted in all three PBIs mostly of the sugar compound. Besides this, a small amount of fenpropimorph was found. The samples of the methanol extracts of the M-label for the 30 PBI sample consisted of 2,6-DMM and the sugar compound in nearly equal amounts together with "Metabolite 3". In the methanol extracts of the two other PBIs, the sugar compound was the main metabolite. Additional treatments of solids after extraction released 67.3–92.4% of the radioactivity present with 65–74% identified as the sugar compound.

From the wheat chaff samples, 14.8–54.0% of the TRR was extracted with methanol. In the extract of the P-label samples three metabolites were identified: BF421-1 (30 PBI: 0.026 mg/kg, 19.2% TRR), its glucoside (30 PBI: 0.024 mg/kg, 18% TRR; 120 PBI: 0.06 mg/ kg, 1.1% TRR) and the sugar compound (30 PBI: 0.015 mg/kg, 11.1% TRR; 120 PBI: 0.07 mg/kg, 12.5% TRR). In the methanol extracts of all three samples of the M-label, 2,6-DMM was the main metabolite (30 PBI: 0.136 mg/kg, TRR 9.2%; 120 PBI: 0.266 mg/kg, TRR 24.9% and 365 PBI: 0.016 mg /kg, TRR 8.4%) with small amounts of the sugar compound (30 PBI: 0.023 mg/kg, TRR 1.5%; 120 PBI: 0.043 mg/kg, TRR 4.1% and 365 PBI: 0.015 mg/kg, TRR 8.1%) and "Metabolite 3" (30 PBI: 0.070 mg/kg, TRR 4.7%; 120 PBI: 0.053 mg/kg, TRR 4.9% and 365 PBI: 0.011 mg /kg, TRR 5.7%) detected. The same components were identified in radioactivity released following further treatment of post extraction solids of the M-label whereas for the P-labelled samples only the sugar compound was released (30 and 120 day PBI).

Metabolites of fenpropimorph in the rotational crop matrices are presented in Figure 14. In addition to the alcohol (BF421-1), its conjugates, and the degradation product BF421-10 (2,6-DMM), other compounds identified were natural sugars like glucose, fructose and saccharose as well as starch.

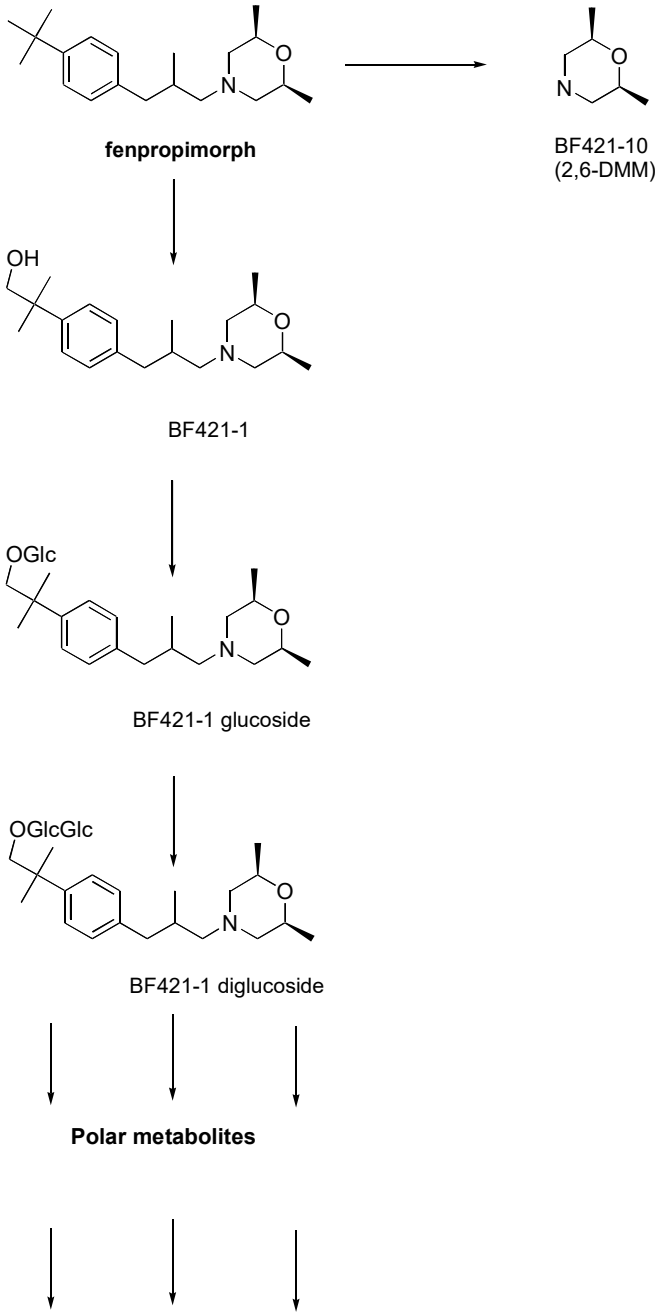


Figure 14 Compounds identified in rotational crops

Field rotational crop trials on representative crops

Metabolism in confined rotational crops shows that no detectable residues would be expected at the targeted maximal annual application rate.

ENVIRONMENTAL FATE IN SOIL

Route of degradation in soil

A number of studies were made available on the aerobic soil metabolism of fenpropimorph and metabolites.

Aerobic degradation of fenpropimorph in soil

Huber (1979 10024) investigated the aerobic soil metabolism of [2,6-^{14}C-morpholine]-fenpropimorph on a sandy loam soil (sand 26.8%, silt 34.7%, clay 38.5%, organic C 1.12%, pH 7.1, CEC meq/100g 8.6) maintained at 22 °C, 40% MWHC in the dark for 12 months. Fenpropimorph was incorporated at a nominal concentration of 6 mg/kg. Samples were extracted with methanol followed by liquid-liquid partition on the extracts with chloroform.

The total radioactivity expressed as fenpropimorph decreased to 57% AD 12 months. The proportion extracted by methanol decreased from 87% TRR at the start of the experiment to 17% TRR after 12 months. Fenpropimorph decreased from 85% AD at day 0 to 6.6% AD after 12 months, while degradation products increased. Further analysis of chloroform extracts (months 2 and 3 combined) identified fenpropimorph (18% AD), BF 421-2 (2.9% AD), and BF 421-10 (2,6-DMM) (1.9% AD).

The DT_{50} for degradation of fenpropimorph was originally reported to be 20 days but was subsequently recalculated by Platz (2002 1007746) to be 24.4 days.

In another study Beutel (1978 1000021) studied the aerobic degradation of fenpropimorph on two soils (Neuhofen, pH 6.1, organic C 2.66% and Hatzenbühl pH 7.1, organic C 1.12%) incubated in the dark at 40% MWHC at room temperature for 153 days. Fenpropimorph was incorporated at about 5 mg/kg and residues of fenpropimorph and BF421-10 (2,6-DMM) measured.

B the end of the study fenpropimorph residues had declined to 42% and 8% of their original concentrations. The DT_{50} values for fenpropimorph were stated as 90 days for the Neuhofen soil and 19 days for the Hatzenbühl soil. Platz (2002 1007746) recalculated the DT_{50} values to be 123.8 and 18.1 days respectively for Neuhofen and Hatzenbühl soils.

Keller (1985 10061) studied the rate of aerobic degradation of fenpropimorph in the dark at various temperatures and water holding capacities for two soils; a clayey loam from Kirchheim (pH 7.2, 2.4% humic material) and a sandy loam from Limburgerhof (pH 6, 1.5% humic material). Fenpropimorph was incorporated at a concentration of about 5 mg/kg.

The DT_{50} values (recalculated by Platz 2002 1007746) ranged from 11–187 days for the clayey loam and 9.4–104 days for the sandy loam.

Table 37 Summary of DT_{50} values for aerobic soil degradation of fenpropimorph under a variety of conditions

Soil	Location	Temperature (C)	%MWHC	DT_{50} (days)	DT_{50} (days) recalculated [A]
Clayey loam	Kirchheim	5	40	181	187.3
		20	20	66	73.7
		20	40	19	21.8
		20	60	11	11.3
		30	40	12	12.4
Sandy loam	Limburgerhof	5	40	55	103.5
		20	20	12	15
		20	40	14	14.9
		20	60	14	15.2
		30	40	9	9.4

%maximum water holding capacity

[A] DT_{50} values recalculated by Platz 2002 1007746 using non-linear first–order regression

Almvik *et al*. (2015) studied the degradation of fenpropimorph in soils from Norway as an example of soils from a cold temperate climate. The degradation of fenpropimorph was studied at two temperatures in the laboratory (10 °C and 20 °C) in topsoil (0–10 cm) from agricultural fields (Klepp sandy loam, Ås loam, Stjørdal sandy loam and Tromsø sandy loam) at three climatic zones in Norway (from latitudes 58–69°). A study was also carried out in the field. Fenpropimorph was applied to the soil as formulated products. Fenpropimorph was applied to give a concentration of 0.5–5 mg/kg dry soil. Duplicate samples were sampled after 0, 3, 7, 14, 28, 56, 84 and 112 days and stored at -20 °C until acetone extraction and LC-MS/MS analysis.

Table 38 DT_{50} values for fenpropimorph in soils from Norway.

	DT_{50} (days)			
	Klepp	Ås	Stjørdal	Tromsø
Laboratory 20 °C	126	68	78	249
Laboratory 10 °C	295	278	450	>1000

The corresponding laboratory DT_{90} values ranged from 1.8 to 9.7 years for fenpropimorph and indicated a long-term persistence of fenpropimorph in the soils. At low soil temperature (10 °C), average DT_{50} ranged from 2 to 4 years.

In another study Hassink and Stephan (2005 1022507) studied the aerobic soil degradation of [morpholine-2,(6)-^{14}C]- and [phenyl-U-^{14}C]-fenpropimorph on a loamy sand soil from Bruch West, Germany at 20 °C and 40% MWHC in the dark for 149 days. The nominal initial concentration of fenpropimorph in soil was 2 mg/kg which is equivalent to a single application rate at 750 g ai/ha. A closed incubation system was used with constant aeration and trapping of evolved gases. Samples were extracted with methanol (3×) followed by methanol/water (3×). Fulvic acids were determined by treatment of solvent extracted soils with 0.5 M NaOH (3×) and acidification of the extracts with concentrated HCl to precipitate the humic acids. Identification of compounds was by LC-MS.

Only one degradation product, BF421-2, was formed at levels above 5% AR reaching a maximum of 7.8 to 9.7% AR at 10 to 14 days after application for the morpholine and phenyl labels respectively.

For the M-label, unextracted residues reached a maximum of 37% AR after 119 days. The fulvic acid fraction was a maximum at 22 days at 11.7% AR while humic acid was a maximum at 58 days at 1.9% AR. For the P-label, unextracted residues reached a maximum of 55.6% AR after 91 days. The fulvic acid fraction was a maximum at 27 days at 4.9% AR while humic acid reached 1.4% AR at the end of incubation (149 days). Mineralisation to CO_2 reached 52.5% AR for the M-label and 38.6% for the P-label by 149 days with no other volatile products detected.

The DT_{50} for fenpropimorph was 11.8 days for the M-label and 13.2 days for the P-label. The DT_{50} for the main metabolite BF421-2 was 3.8 days for the M-label and 4.4 days for the P-label.

Table 39 summarises the DT50 values recalculated using data reported in the original studies (Platz 2002 1007746, Platz 2005a 1020649) together with results of more recent studies are summarised. The values have been normalised to 20 °C and soil moisture at pF2 using standard procedures. The overall geometric mean of the different normalised DT_{50} values in soil of fenpropimorph is 16.2 days.

Table 39. DT_{50} values (days) of fenpropimorph, normalised to 20 °C and soil moisture at pF2

Soil location	Soil type	Experimental $\Theta_{act.}$ /temperature (%MWHC/°C)	DT_{50}	$DT_{50\ norm}$	$DT_{50\ norm}$ geometric mean [A]	Reference
	Sandy loam	40/22	24.4	19.9	19.9	1979/10024
Neuhofen	Loamy sand	40/20	123.8	134.0	134.0	1978/1000021
Hatzenbühl	Loamy sand	40/20	18.1	17.1	17.1	1978/1000021

Soil location	Soil type	Experimental $\Theta_{act.}$ /temperature (%MWHC/°C)	DT_{50}	$DT_{50\ norm}$	$DT_{50\ norm}$ geometric mean [A]	Reference
Kirchheim	Clay loam	40/5 20/20 40/20 60/20 40/30	187.3 73.7 21.8 11.3 12.4	25.4 [B] 25.5 12.2 8.4 18.0 [B]	13.8	1985/10061
Limburgerhof	Sandy loam	40/5 20/20 40/20 60/20 40/30	103.5 15 14.9 15.2 9.4	16.7 [B] 6.2 10.0 13.5 16.2 [B]	9.4	1985/10061
Bruch West (morpholine label)	Sandy loam	40/20	11.8	10.2	10.8	2004/1022507
Bruch West (phenyl label)		40/20	13.2	11.4		
Overall geometric mean					16.2	

[A] geometric mean of normalised DT_{50} values calculated for identical soils

[B] experiments at 5 °C and 30 °C were not considered for averaging

Aerobic soil degradation of BF421-2

In a study by Ebert and Harder (2005 1010873) BF421-2 was applied as test substance to three soils and incubated in the laboratory (aerobic dark, 20 °C, 40% MWHC). The soils were a sandy loam (Bruch West, pH 7.3, % organic C 2.7), a loam (LUFA 3A, pH 7.2, % organic C 2.4) and a loamy sand (Li10, pH 6.3, % organic C 0.8). BF421-2 was incorporated at 0.53 mg/kg. Soil samples were extracted with methanol (3×) and methanol water 1:1 v:v, 3×) and the extracts analysed by LC-MS.

In all three soils, residues of BF421-2 declined rapidly. Single first-order DT_{50} values were calculated to be 4.4–9.4 days.

The rapid decline observed in these three soils is in agreement with the results of Hassink and Stephan (2005 1022507) who estimated a DT_{50} of 4.1 days for BF421-2 as part of a study on the soil metabolism of fenpropimorph.

Normalised laboratory DT_{50} values in soil of BF421-2 are summarised in Table 40. The geometric mean of the normalised DT_{50} values is 4.6 days.

Table 40 Summary of DT_{50} values of BF421-2 (SFO), normalised to 20 °C and soil moisture at pF2

Soil location	Soil type	Experimental $\Theta_{act.}$ /temperature (%MWHC/°C)	DT_{50}	$DT_{50\ norm}$	$DT_{50\ norm}$ geometric mean	Reference
Bruch West (morpholine label)	Sandy loam	40/20	3.8	3.3		2004/1022507
Bruch West (phenyl label)		40/20	4.4	3.8	3.5	2004/1022507
Bruch West	Sandy loam	40/20	4.7	5.2	5.2	2005/1010873
Lufa 3A	Loam	40/20	4.4	3.4	3.4	2005/1010873
Li10	Loamy sand	40/20	9.4	8.9	8.9	2005/1010873
Overall geometric mean					**4.6**	

Aerobic soil degradation of BF421-7

Ebert (2010a 1005601) studied the rate of degradation of [phenyl-U-^{14}C]-BF421-7 in three soils incubated under aerobic conditions at 20 °C and 40% MWHC in the dark for 120 days. The soils loamy sand L10 from Limburgerhof, loamy sand LUFA 5M from Mechtersheim and a silty sand Speyerer Wald 2 from Schifferstadt; all originated from Rhineland Palatinate, Germany. They were treated in a bulk application at a concentration of about 0.27 mg/kg dry soil. A closed incubation

system was used with constant aeration and trapping of evolved gases. Samples were extracted four times with acetonitrile/water (2:1, v:v) and extracts analysed using LC-MS.

The amount of extractable radioactivity decreased moderately fast in soil Li 10, fast in soil Lufa 5M and rather slow in soil Speyerer Wald 2, declining to 24, 3, and 59% TAR after 120 days, respectively. The unextracted residues increased slowly to 25% TAR in soil Li 10. In soil Lufa 5M, unextracted radioactivity reached its maximum after 14 days at about 40% TAR and then declined very slowly to 35% TAR after 120 days. In soil Speyerer Wald 2, the formation of bound residues was slowest reaching only about 18% TAR at the end of incubation. The mineralisation to CO_2 reached about 39% TAR in Li 10, 47% TAR in Lufa 5M and only about 15% TAR in Speyerer Wald 2. No other volatile products were detected.

^{14}C-BF421-7 represented the only major radioactive fraction in the soil extracts. No other degradation product exceeded 3.5% TAR at any sampling time. At the end of incubation, BF421-7 was degraded reaching finally 22% TAR in Li 10, about 1% TAR in Lufa 5M and about 54% TAR in Speyerer Wald 2.

Kinetic analysis and calculations of DT_{50} and DT_{90} values was performed. The estimated parameter values and the corresponding DT_{50} and DT_{90} values are reported for the best fit kinetic model in Table 41.

Table 41 DT_{50} and DT_{90} values (days) for BF421-7

Soil	DT_{50}	DT_{90}	Kinetic model	χ^2
Li 10	50.3	166.9	SFO	4.42
LUFA 5M	2.5	13.9	FOMC	4.54
Speyerer Wald 2	209.1	>1000	FOMC	2.24

In all three soils, the degradation occurred mainly by mineralisation to CO_2 and formation of residues not extracted with acetonitrile. No other metabolite appeared in significant amounts. The formation of $^{14}CO_2$ in all three soils shows that the aromatic ring was opened and further degraded.

Aerobic soil degradation of BF421-10

Elsom (1998 10250) studied the aerobic soil metabolism of [2,6-^{14}C]-BF421-10 (=cis-2,6-dimethylmorpholine) in a sandy loam soil from Arrow (sand 65.6%, silt 22.4%, clay 11.9%, organic C 2.2%, microbial biomass 39.5 mg C/100g, CEC 13.9 meq/100 g, pH 7.1, MWHC 43%) at 20 °C in the dark for 120 days. BF421-1 was mixed with soil at a concentration of 0.2 mg/kg. A closed incubation system was used with constant aeration and trapping of evolved gases. Samples were extracted with acetonitrile/water, acetonitrile/0.1 M HCl and methanol/0.1 M HCl mixtures. Analysis of extracts was by LC-MS and TLC.

BF421-10 was the main component at all sampling times representing 82.6% TAR at day 2 declining to 42.7% TAR at day 120, the final sampling point. CO_2 accounted for 11.7% TAR at termination with bound residues accounting for 29.7% TAR. Bound residues were fractionated into humin, humic acid and fulvic acid and by day 120 these fractions accounted for 9.0, 8.8 and 9.9% AR respectively. BF421-10 was detected in the fulvic acid fraction in addition to very polar material.

The DT_{50} for BF421-10 was 149 days (SFO).

Field studies

In a series of studies, the degradation of fenpropimorph was studied following a single application to fallow land or bare soil (Hesse and Tilting 1991 10760, Hesse and Tilting 1992 1000183, von der Mühll *et al*. 1980 10042, Stockmaier *et al.* 1996 10365). The results are summarised in Table 42.

Table 42 Summary of field dissipation studies for fenpropimorph.

Reference	Location	Soil	Soil properties %organic C	pH	DT_{50} (days)	DT_{90} (days)
1992/1000183	Oberding, Germany	Sandy loam	1.48	6.3	10	35
	Brockhausen, Germany	Sandy loam	1.19	5.7	29	324
	Birkenheide, Germany	Loamy sand	0.8	6.1	-	>360
1991/10760	Hoheneggelsen-Mölme, Germany	Loam	0.98	7.5	40	131
	Stetten a. , Germany	Loamy clay	1.13	6.8	90	>2× study duration
1996/10365	Nienwohlde, Germany	Silty sand	1.2	6.3	47	-
	Neuenkirchen, Germany	Clayey silt	0.9	7.3	36	-
1980/10042	Dielsdorf, Switzerland	Sandy loam	-	6.2	42	-
Almvik *et al.* 2015 (0-50 cm)	Klepp, Norway	Sandy loam			54	-
	Ås, Norway	Loam			150	-
	Stjørdal, Norway	Sandy loam			218	-
	Tromsø, Norway	Sandy loam			173	-

One of the studies, (von der Mühll *et al*. 1980 10042) used [benzylic-^{14}C]- fenpropimorph and investigated the presence of metabolites in the top 0–5 cm of soil where the majority of radioactivity was located. The only significant metabolite identified was BF421-7 which reached a maximum of 9.8% of the applied dose at 4 weeks after the start of the experiment. A DT_{50} value was estimated for BF421-7 as 82 days and is consistent with the laboratory degradation studies.

In summary, degradation in soil proceeds by oxidation and opening of the morpholine ring to give BF421-2 (the acid), BF421-7 and BF421-10 (dimethylmorpholine). In laboratory studies DT_{50} values for aerobic degradation of fenpropimorph, normalised to 20 °C and 40% MWHC, ranged from 6.2–134 days with a geometric mean of 16.2 days. BF421-2, BF421-7 and BF421-10 are formed but do not exceed 10% TAR. DT_{50} values for BF421-2, BF421-7 and BF421-10 are estimated as 4.6 days, 2.5–2.09 days and 149 days respectively.

In field studies (outdoor) DT_{50} values for fenpropimorph ranged from 10 to 218 days with the longer DT_{50} values observed in colder climates, and for BF421-7 a DT_{50} value of 82 days was reported.

ENVIRONMENTAL FATE IN WATER

Hydrolysis

Rüdel (1988a 0443) studied the hydrolytic stability of [^{14}C]-fenpropimorph (0.93 mg/L) at 25 °C for 32 days in dark, sterile, aqueous buffered solutions at pH 3, 5, 7 and 9.

At no pH-value hydrolysis products were detected by TLC-analyses. At neutral (pH 7) and alkaline (pH 9) pH values the recovery of the test substance decreased with time due to adsorption effects and the low solubility of the active substance at these pH values. A second peak (besides the test substance) in the TLC chromatograms of samples incubated at pH 3 could be characterised as the hydrochloride-adduct of fenpropimorph. In the rinsing solutions of the glass stoppers of the incubation flasks from solutions incubated at pH 9, besides fenpropimorph another compound was characterised by TLC co-chromatography. This compound had a Rf-value similar to the reference substance BF421-13. An assessment of the concentration of this compound as a reasonable worst-case calculation resulted in amounts up to 7.7% TAR. However, this substance is not considered as a hydrolysis product.

It can be concluded that fenpropimorph is hydrolytically stable in the whole investigated pH-range (pH 3 to pH 9). No half-lives were calculated.

Aqueous hydrolysis is not expected to contribute to the degradation of fenpropimorph in the environment.

Photochemical degradation

In general, photolysis of fenpropimorph is not a relevant degradation pathway since the absorption coefficient of fenpropimorph is < 10 L/(mol × cm) above 290 nm.

Herrchen (1988a 0433) studied the soil photolysis of fenpropimorph on sterile loamy sand (sand 55.2%, silt 36.9%, clay 7.9%, organic C 2.3%, pH 5.8, CEC mval/100 g 2.39) at 25 °C. Samples were irradiated using a Xe lamp (100000 lux, >290 nm). The radioactivity extracted using methylene chloride decreased with time as the 'bound' or unextracted radioactivity increased. As well as parent fenpropimorph, the main compounds detected were BF421-13 (max 9.3% TRR) and BF421-15 (max 5.3% TRR). The half-life for the degradation of parent compound was estimated to be 30 days.

Photochemical degradation is not expected to contribute significantly to the degradation of fenpropimorph in the environment.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The Meeting received descriptions and validation data for analytical methods for residues of fenpropimorph in plant matrices and fenpropimorph and BF421-2 in animal matrices. The methods are suitable for analysis of fenpropimorph in plant and fenpropimorph and BF421-2 in animal matrices.

Table 43 Overview of the methods for determination of fenpropimorph in crops

		Method 137 (1978/10076)	Method 156 (1979/10088)
Extraction and clean-up	Analytes	fenpropimorph	fenpropimorph
	Matrix	Grain, straw	Grain, straw, forage
	Extraction	Samples are homogenized with dry ice, extracted with methanol, filtered, acidified with HCl and methanol removed (50 °C). The water phase is partitioned against chloroform, the chloroform phase filtered with Na_2SO_4 and evaporated to dryness, the residue is dissolved in acetone and transferred to an ion exchange column (1), eluted, the acetone removed and the water phase extracted with n-hexane, then chloroform. The chloroform phase is evaporated and the residue dissolved in tolunene/acetone for clean-up on a aluminium oxide column (2). The eluant is evaporated to dryness and redissolved in acetone for quantitation using GC-FID	Samples are homogenized with H_2O and extracted with chloroform using a Bleidner apparatus (reflux 2 h), the chloroform is dried with sodium sulphate and solvent removed. The residue is dissolved in hexane, evaporated to dryness and redissolved in hexane for GC analysis.
	Column	(1). ion exchange resin (Dowex 50WX4) (2). aluminium oxide	
	Eluent	(1). acetone/2M HCl (8:2) (2). toluene/acetone (1:3)	
Chromatography	Type	GC	GC
	Analytical column	5% Dexsil on Chromosorb G	1.05% OV17 + 1.95% OV210 on Gaschrom Q 100-200 mesh
	Dimensions	25 cm, 8 mm id or 20 cm, 20 mm id	2.5 m, 2.5mm id
	Particle size	60-80 mesh	
	Injection volume	5 µL	Dissolved in iso-octane, 2 µL (or 5 µL if packed column)
	Flow rate		60 mL He/min
	Instrument	Perkin Elmer 3920	Perkin Elmer 3920 if packed column, Dani 3900 if capillary
Detection	Quantitative detection	N-FID	N-FID
	LOQ	0.05 mg/kg grain, 0.1 mg/kg straw	0.05 mg/kg grain, 0.1 mg/kg forage, straw
	Whole method linearity (r^2)		0.25-1 µg/mL, ≥0.998

Table 43: cont

		Method 241 (87/10142)	Method 241/1 (1993/11464)	Method 241/3 (2000/1012400)
Extraction and clean-up	Analytes	fenpropimorph	fenpropimorph	fenpropimorph
	Matrix	Wheat forage, straw, grain, rape forage, seed, Brussels sprouts, tangerines, oranges	banana	Cereal grain, forage, straw
	Extraction	Samples are homogenized with H_2O, saturated with NaCl for rape seed, and extracted with chloroform using a Bleidner apparatus (reflux 2h). The chloroform extract is filtered through a phase separation filter and the solvent removed (40 °C), the residue dissolved in methylene chloride with clean-up on a silica gel column, eluted, the solvent removed, the residue dissolved in *n*-hexane, solvent removed, residue dissolved iso-octance for analysis	Samples are homogenised in H_2O, and extracted with chloroform using a Bleidner apparatus (reflux 2h). The chloroform extract is acidified (0.5M HCl), the organic phase separated and solvent removed. The residue is dissolved in methanol with ion-exchange clean-up and the eluant acidified (1M HCl), partitioned agianst dichloromethane (2×), the dichloromethane removed, the residue dissolved in acetone, evaporated to dryness again and redissolved in iso-octane/acetone (9:1) for analysis.	Samples mixed with methanol are macerated and the solvent recovered by filtration through celite. After addition of water and 2M HCl, the methanol is removed, additional water and methanol added and the solution is partitioned against iso-hexane. The aqueous phase is extracted with dichloromethane (2×) and the combined dichloromethane extract evaporated to dryness (40 °C) before dissolving in dichloromethane, loading on column (1), washing, eluting, removing the solvent and redissolving in methanol for ion-exchange clean-up (2). The eluant from (2) is acidified with HCl and extracted with dichloromethane, the dichloromethane phase evaporated to dryness, the residue taken up in acetone, evaporated to dryness and re-dissolved in acetone for analysis
	SPE column	Non-activated silica gel	Ion exchange resin (sulphuric acid form)	(1). Silica gel (2). Ion exchange (sulphonic acid)
	Washing solvent	Methylene chloride	methanol	(1). dichloromethane (2). methanol
	Eluent	98% methylene chloride, 2% methanol	1 M NaOH/methanol (2.5/8 v/v)	(1). dichloromethane/methanol (96:4) (2). 1M NaOH + methanol
Chromatography	Type	GC	GC	GC
	Analytical column	1.05% OV17 + 1.95% OV210 on Gaschrom Q 100-200 mesh if packed column	SE14	DB5, XLB
	Dimensions	2.5 m, 2.5mm id if packed column or 0.28 mm id for GC capillary	18 m, 0.27mm id	30 m, 0.25 mm id
	Injection volume	2 µL or 5 µL if packed column	2 µL	1 µL MS or 5 µL ion trap
	Flow rate	60 mL He/min		20 mL/min
	Instrument	Perkin Elmer 3920 if packed column, Dani 3900 if capillary	Perkin Elmer 8320	HP 5890 GC, HP 5971A or GC Varian 3700, Ion-trap Finnigan IST 40
Detection	Quantitative detection	N-FID	PN detector or GC-MS (m/e 128 & 303)	MS (EI mode), m/z 128
	LOQ	0.05 mg/kg	0.05 mg/kg	0.05 mg/kg
	Whole method linearity (r^2)	0.5-2 µg/mL, $r^2 \geq 0.998$	0.5-2 µg/mL, 0.0025-0.025 µg/mL $r^2 \geq 0.998$	0.025-5 µg/mL, $r^2 \geq 0.996$

Table 43: cont

		Method 456/0 (2001/1000985)	Method 535/1 (2001/1039427)
Extraction and clean-up	Analytes	fenpropimorph	fenpropimorph
	Matrix	Cereals forage, straw, grain	Wheat, rape seed, tomato, onion, lemon, lettuce
	Extraction	Add methanol/water/2M HCl (70/25/5 v/v/v) and macerate. Centrifuge and take an aliquot. Add 0.2M NaOH and partition against cyclohexane/0.2M NaOH. Evaporate the cyclohexane phase to dryness (40 °C) and dissolve the residue in methanol/Baker ® water	Add methanol/water/2M HCl (70/25/5 v/v/v) and macerate. Centrifuge and take an aliquot. Add 0.2M NaOH and partition against cyclohexane/0.2M NaOH and add 2M HCl. Evaporate the cyclohexane phase to dryness (40 °C) and dissolve the residue in methanol/Baker ® water
Chromatography	Type	LC	LC
	Analytical column	Betasil C18	Betasil C18
	Dimensions	100 mm, 2 mm id	100 mm, 2 mm id
	Particle size	20 µL	20 µL
	Injection volume	≥20 µL	50 µL
	Mobile phase	methanol/Baker ® water/formic acid A: 90/10/0.1 B: 10/90/0.1	A: Baker ® water/formic acid 1000/1 v/v B: methanol/formic acid 1000/1 v/v
	Flow rate	600 µL/min	600 µL/min
	Instrument	PE Sciex API 3000 MS	PE Sciex API 3000 MS
Detection	Quantitative detection	Positive ionization mode 304→147, 304→130, 304→116	Positive ionization mode 304→147, 304→132, 304→116
	LOQ	0.05 mg/kg	0.01 mg/kg
	Whole method linearity (r^2)	0.025-10 ng/mL ≥0.99	0.05-0.5 ng/mL ≥0.99

Method No. 137 (Anonymous 1978 10076)

Plant material is chopped with dry ice, extracted with methanol, and the extracts are filtrated. After addition of water and HCl, the methanol is removed with a rotary evaporator (50 °C). The remaining water phase is partitioned against chloroform, the chloroform phase filtered with sodium sulphate and evaporated to dryness. The residue is dissolved in acetone and transferred to an ion exchange column. The eluate is discarded and the active substance is eluted from the column with an acetone/2M HCl (8:2 v/v). After evaporation of acetone, the remaining water phase is firstly extracted with n-hexane (clean-up, is discarded), then with chloroform. The chloroform phase is evaporated and the residue redissolved in a toluene/acetone (1:3 v/v). Finally, the toluene/acetone mixture is cleaned up by an aluminium oxide column, the eluate evaporated to dryness and the residue dissolved in acetone for determination by GC with an alkali flame detector.

Recovery and repeatability data for the determination of fenpropimorph residues in crops are presented in Table 44. Average recoveries ranged from 70.5 to 77.8%. The LOQ was 0.05 mg/kg for grain and 0.1 mg/kg for straw. The %RSDs ranged from 0.8 to 6.7. Linearity was not reported.

Table 44 Recovery data for method 137 for the determination of fenpropimorph residues in cereals

Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
Straw	0.1	4	78	2.7	75-80
	0.4	4	74	2.8	72-77
	1	4	74	4.8	70-78
Grain	0.05	4	77	6.7	72-84
	0.2	4	75	1.9	73-76
	1	4	70	0.8	70-71

Method No. 156 (Anonymous 1979a 10088)

Plant plant material is macerated with water and extracted with chloroform using a Bleidner apparatus (reflux 2h). The chloroform phase is dried with sodium sulphate and solvent removed. The residue is

dissolved in hexane, again evaporated to dryness and the residue redissolved in hexane for GC analysis.

Recovery and repeatability data for the determination of fenpropimorph residues in crops are presented in Table 45. Average recoveries ranged from 74 to 92%. The LOQ was 0.05 mg/kg for grain and 0.1 mg/kg for forage and straw. The %RSDs ranged from 2.1 to 5.0. Linearity was good for injections between 0.25 and 1 µg/mL ($r^2 \geq 0.9998$)

Table 45 Recovery data for method 156 for the determination of fenpropimorph residues in cereals

Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recover range
Forage	0.1	4	88	2.2	86-90
	0.5	4	74	4.6	70-78
	5	4	82	3.1	79-85
Straw	0.1	4	88	5.0	84-94
	0.5	4	78	4.3	74-82
	5	4	92	2.1	89-93
Grain	0.05	4	79	3.3	76-82
	0.5	4	78	4.4	74-82
	5	4	78	3.2	74-80

Method No. 241 (Maxwell 1993a 10384, Beutel and Tilting 1987a 10142)

Samples are homogenised and water, or in the case of rape seed a saturated NaCl solution, added prior to extraction of residues with chloroform by means of a Bleidner apparatus (reflux 2h). The chloroform extract is filtered through a phase separation filter and the solvent removed using a rotary evaporator (40 °C). The residue is redissolved in methylene chloride with clean-up on a non-activated silica gel column. Elution is with 98% methylene chloride, 2% methanol. The solvent is removed by rotary evaporation, the residue taken up in n-hexane and the solvent removed once more before the residue is redissolved in iso-octane for analysis by GC with nitrogen detector.

Recovery and repeatability data for the determination of fenpropimorph residues in crops are presented in Table 46. Average recoveries ranged from 72 to 104%. The LOQ was 0.05 mg/kg for all crops investigated. The %RSDs ranged from 2.0 to 8.8. Linearity was good for injections between 0.5 and 2 µg/mL ($r^2 \geq 0.998$)

Table 46 Recovery data obtained during validation of analytical method 241 for the determination of fenpropimorph residues

Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
Wheat forage	0.05	4	97	2.6	94-100
	0.5	4	78	5.4	74-81
Wheat straw	0.05	4	83	6.4	80-91
Wheat grain	0.05	4	82	4.9	78-87
Brussels sprouts	0.05	4	104	6.9	99-114
Rape seed	0.05	4	89	7.8	82-98
	5	4	77	6.0	72-82
Rape forage	0.05	4	76	8.8	69-83
	5	4	92	4.9	86-96
Oranges	0.05	4	98	2.0	96-101
	5	4	72	3.4	70-75
Tangerines	0.05	4	94	5.4	89-101
	5	4	84	3.0	81-86

The stability of standard solutions stored at 4 °C in the dark or room temperature in the light was also investigated. Samples stored at 4 °C in the dark were stable for 90 days (102% of concentration in fresh sample) but declined rapidly on storage at room temperature with exposure to daylight (6.8% concentration of fresh sample after 90 days).

In a study describing the independent laboratory validation of the method average recoveries ranged from 77 to 84%. The LOQ was 0.05 mg/kg for grain and straw. The %RSDs ranged from 6.7 to 29. Linearity was good for injections between 0.1–1 ng and 1-4 ng) ($r^2 \geq 0.98$).

Recovery and repeatability data for the determination of fenpropimorph residues in crops are presented in Table 47.

Table 47 Recovery data obtained during independent laboratory validation of analytical method 241 for the determination of fenpropimorph residues

Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
Wheat straw	0.05	5	84	21	60-103
	5	4	78	6.7	75-86
Wheat grain	0.05	10	82	23	54-109
	5	5	77	29	45-102

Method 241 was modified to allow quantification of fenpropimorph in banana matrices (Tilting 1993a 11464). Samples are homogenised and water, added prior to extraction of residues with chloroform by means of a Bleidner apparatus (2 h reflux). The chloroform extract is acidified with HCl and the organic phase separated, and the solvent removed using a rotary evaporator. The residue is redissolved in methanol with clean-up on an ion exchange column (sulphonic acid form). The residue is eluted using 1 M NaOH and methanol (2.5/8 v/v). Water is added to the eluant and the solution acidified with HCl and partitioned against dichloromethane (2×). The dichloromethane phase is evaporated to dryness and the residue dissolved in acetone, evaporated to dryness once more before dissolving in iso-octane/acetone (9:1 v/v) for analysis by GC-NPD or GC-MS.

Recovery and repeatability data for the determination of fenpropimorph residues in crops are presented in Table 48. Average recoveries ranged from 87 to 97%. The LOQ was 0.05 mg/kg for banana peel, pulp and whole fruit. The %RSDs ranged from 2.4 to 17. Linearity was good for injections between 0.5 and 2 µg/mL ($r^2 \geq 0.99$)

Table 48 Recovery data for method 241/1 for the determination of fenpropimorph residues in banana

Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
Pulp	0.05	4	96	6.4	88-103
	5	4	91	16	77-112
Peel	0.05	4	92	2.4	90-94
	5	4	87	17	69-100
Whole fruit	0.05	4	97	6.4	90-102
	5	4	96	3.3	93-99

Artz and Malinsky (1997a 5077) provided an independent laboratory validation of the modified method 241/1 was slightly modified. Recovery and repeatability data for the determination of fenpropimorph residues in crops are presented in Table 49. Average recoveries ranged from 70 to 75%. The LOQ was 0.05 mg/kg for banana fruit. Linearity was good for injections between 0.2 and 2 µg/mL ($r^2 \geq 0.99$)

Table 49 Recovery data obtained during independent laboratory validation of analytical method 241/1 for the determination of fenpropimorph residues in bananas

Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
Whole fruit	0.05	2	70	-	70-70
	0.25	2	75	-	73-77

Thirty-three pesticides registered in the USA were analysed using the method with no indication of interference (Shaffer and Artz 1997a 5003).

Radiovalidation of the extraction step for fenpropimorph in banana

A comparison of the extractability of residues in banana by CH_3OH versus Bleidner extraction showed that ^{14}C-fenpropimorph could be recovered by methanol extraction just as well as by the Bleidner extraction used in the metabolism study.

Table 50 comparison of ^{14}C extracted using methanol versus a Bleidner extraction

	TRR	CH_3OH extraction		Bleidner extraction	
	(mg eq/kg)	mg eq/kg	%TRR	mg eq/kg	%TRR
Peel	0.218	0.135	62	0.101	46
Pulp	0.042	0.025	60	0.022	52
Whole fruit	0.105	0.064	59	0.050	50

Additional modifications to method 241 were made by Tilting (1994a 10550) allow analysis of asparagus, lemon, grapes, cabbage, sugar beet (including green material), oat (grain, forage and straw) and other cereals. Fenpropimorph is extracted with chloroform from plant sample material by means of Bleidner distillation. Pre-extraction with acetone and water (2 + 1) is required in the case of cereal grains. After purification by means of a silica gel or ion-exchange resin (sulphonic acid form) column, the active ingredient is determined by GC (N-FID).

Plant material other than grain: Water is added, the material homogenised, $NaHCO_3$ added, and the material transferred to a Bleidner apparatus.

Cereal grain: Ground cereal grain is boiled with acetone:water (2:1) for 1 hour, after which the solution is acidified with HCl and filtered through celite. The acetone is removed by evaporation and water and $NaHCO_3$ added before transfer to a Bleidner apparatus.

Recovery and repeatability data for the determination of fenpropimorph residues in crops are presented in Table 51. Average recoveries ranged from 72 to 105%. The LOQ was 0.05 mg/kg for banana peel, pulp and whole fruit. The %RSDs ranged from 1.3 to 12. Linearity was good for injections between 0.5 and 2 µg/mL ($r^2 \geq 0.99$)

Table 51 Recovery data for analytical method 241/2 for the determination of fenpropimorph residues in asparagus, lemon, grapes, cabbage, sugar beet (including green material), oat (grain, forage and straw) and other cereals

Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
Asparagus	0.05	4	87	4.8	83-92
	5.0	4	84	8.1	75-90
Lemon	0.05	4	72	2.5	70-73
	5	4	77	8.0	74-87
Vine	0.05	4	98	6.7	88-103
	5	4	94	3.3	91-98
Cabbage [A]	0.05	4	105	11	89-114
	5	4	94	2.1	91-96
Sugar beet green matter	0.05	4	80	6.1	73-84
	5	4	79	4.7	74-82
Sugar beet	0.05	4	82	4.9	79-87
	5	4	74	8.3	71-83
Oat green matter	0.05	4	98	5.4	94-102
	5	4	89	11	80-102
Oat straw	0.05	4	94	3.3	90-97
	5	4	103	6.4	99-113
Oat grain [B]	0.05	4	99	7.6	90-109
	5	4	82	1.3	81-84
Wheat grain [B]	0.05	4	84	11	74-96
	5	4	80	6.4	74-86
Barley grain [B]	0.05	4	89	10	79-101
	5	4	79	10	67-86

Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
Rye grain [B]	0.05	4	78	12	71-90
	5	4	81	7.0	74-87

[A] With ion exchange column clean-up

[B] with pre-extraction and ion-exchange column clean-up

Method 241 was further modified to allow the use of GC-MS detection (Sasturain and Mackenroth 2000 1012400). Samples are mixed with methanol are macerated and the solvent recovered by filtration through celite. After addition of water and 2M HCl, the methanol is removed, additional water and methanol added and the solution is partitioned against iso-hexane. The aqueous phase is extracted with dichloromethane (2×) and the combined dichloromethane extract evaporated to dryness (40 °C) before redissolving in dichloromethane, loading onto a silica gel column, washing with dichloromethane and eluting with dichloromethane/methanol (96/4 v/v). The solvent is removed and the residue dissolved in methanol for ion-exchange clean-up (sulphonic acid form). The column is washed with methanol and eluted with 1M NaOH/methanol, the eluant is acidified with HCl and extracted with dichloromethane, the dichloromethane phase is evaporated to dryness and the residue dissolved in acetone, evaporated to dryness and re-dissolved in acetone for analysis by GC-MS (m/z 128).

Recovery and repeatability data for the determination of fenpropimorph residues in crops and processed grain products are presented in Tables 52 and 53. Average recoveries ranged from 71 to 97%. The LOQ was 0.05 mg/kg for all matrices. The %RSDs ranged from 2.8 to 14. Linearity was good for injections between 0.0025 and 0.025 µg/mL ($r^2 \geq 0.99$).

Table 52 Recovery data for method 241/3 for the determination of fenpropimorph residues in cereals and processed cereal products using mass selective detection (Sasturain and Mackenroth 2000 1012400, Schultz 2002 1004082)

Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
Plant	0.05	5	82	6.1	77-88
	0.5	5	81	3.4	77-85
	5.0	5	97	6.3	88-103
Grain	0.05	5	80	7.3	72-87
	0.5	5	80	4.2	76-84
	5	5	94	11	79-102
Straw	0.05	5	84	5.5	80-91
	0.5	5	74	4.0	70-77
	5	5	78	2.8	75-81
Fine bran (middlings)	0.05	5	80	9.0	69-89
	0.5	5	77	10	70-91
Coarse bran	0.05	5	74	12	63-86
	0.5	5	73	5.6	70-80
Flour (type 550)	0.05	5	79	5.5	74-84
	0.5	5	75	8.9	66-81
Wholemeal flour	0.05	5	72	13	57-81
	0.5	5	82	10	71-92
Wholemeal bread	0.05	5	75	9.1	67-79
	0.5	5	71	9.2	60-76
Beer	0.05	5	85	14	70-99
	0.5	5	73	5.0	69-77
Brewing malt	0.05	5	75	10	68-88
	0.5	5	82	8.7	74-90

Table 53 Recovery data for method 241/3 for the determination of fenpropimorph residues in cereals using ion trap detection (Sasturain and Mackenroth 2000 1012400)

Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
Plant	0.05	5	77	3.1	74-81
Grain	0.05	5	85	2.8	81-89
Straw	0.05	5	84	8.0	76-97

Method No. 456/0 (Benz and Mackenroth 2001 1000985)

Benz and Mackenroth (2001 1000985) developed an LC-MS/MS method for determination of fenpropimorph (method 456/0). Methanol/H_2O/2M HCl (70:25:5 v/v), is added to samples and the mixture macerated. An aliquot is centrifuged and the supernatant is partitioned against cyclohexane/0.2 N NaOH. The cyclohexane phase is evaporated to dryness (40 °C, stream N_2) and the residue dissolved in methanol/Baker ® water (80:20 v/v). The final determination is performed by LC-MS/MS (m/z 304 → 147 or 304 → 130 or 304 → 116).

Recovery and repeatability data for the determination of fenpropimorph residues in crops using method 456/0 are presented in Table 54. Average recoveries ranged from 76 to 96%. The LOQ was 0.05 mg/kg for all matrices. The %RSDs ranged from 1.5 to 5.8. Linearity was good for injections between 0.025 and 10 ng/mL ($r^2 \geq 0.99$)

Table 54 Recovery data for method 456/0 for the determination of fenpropimorph residues in crops (m/z 304 → 147)

Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
Wheat forage	0.05	5	89	5.7	83-95
	0.5	5	89	2.4	87-91
Wheat grain	0.05	5	96	2.3	94-99
	0.5	5	84	3.8	80-87
Wheat straw	0.05	5	85	5.8	79-90
	0.5	5	84	3.2	81-87
Oranges	0.05	5	76	3.7	71-78
	0.5	5	79	1.5	77-80
Sunflower seed	0.05	5	80	2.1	78-82
	0.5	5	90	2.1	87-91

The LC-MS/MS final determination for fenpropimorph is a highly selective detection technique with quantitation possible at three different transitions and no confirmatory technique is required.

Method 535/1 (Mackenroth and Lehmann 2006 1039427)

Another LC-MS/MS method was developed by Mackenroth and Lehmann (2006 1039427), Method 535/1 which is a modification of method 456/0. Macerated samples are extracted with a mixture of methanol, water and 2M hydrochloric acid. An aliquot of the extract is centrifuged and partitioned at alkaline conditions against cyclohexane. The final determination of the analytes is performed by LC-MS/MS.

Recovery and repeatability data for the determination of fenpropimorph residues in crops are presented in Table 55. In all matrices tested, the mean recovery values were between 86 and 111%, except for rapeseed at mass transition m/z 304 →132 at 0.1 mg/kg with 117%. The LOQ was 0.01 mg/kg for all matrices. The %RSDs ranged from 1.8 to 8.4. Linearity was good for injections between of 0.05 to 0.5 ng/mL ($r^2 \geq 0.99$).

Table 55 Recovery data for analytical method 535/1 for the determination of fenpropimorph residues

Matrix	Transition	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range (%)
Wheat plant w/o root	m/z 304 →147	0.01	5	96	2.1	93-99
		0.1	5	108	2.7	104-111
	m/z 304 →132	0.01	5	97	5.5	89-104
		0.1	5	105	5.2	97-111
Wheat grain	m/z 304 →147	0.01	5	102	3.9	98-108
		0.1	5	109	1.8	107-112
	m/z 304 →132	0.01	5	107	6.7	99-117
		0.1	5	111	3.6	104-114

Matrix	Transition	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range (%)
Wheat straw	m/z 304 →147	0.01	5	94	4.5	88-99
		0.1	5	105	4.6	101-111
	m/z 304 →132	0.01	5	101	5.3	95-109
		0.1	5	105	8.4	92-116
Lemon	m/z 304 →147	0.01	5	86	3.8	82-90
		0.1	5	88	4.1	83-93
	m/z 304 →132	0.01	5	88	7.3	98-112
		0.1	5	87	3.0	100-110
Lettuce	m/z 304 →147	0.01	5	104	5.1	95-108
		0.1	5	109	2.0	107-112
	m/z 304 →132	0.01	5	106	5.2	98-112
		0.1	5	105	4.2	100-110
Rape seed	m/z 304 →147	0.01	5	106	3.7	102-112
		0.1	5	111	2.7	108-114
	m/z 304 →132	0.01	5	110	1.9	107-112
		0.1	5	117	3.9	111-120
Tomato	m/z 304 →147	0.01	5	105	2.4	102-106
		0.1	5	106	2.4	103-110
	m/z 304 →132	0.01	5	104	7.1	96-114
		0.1	5	104	2.4	102-108
Onion	m/z 304 →147	0.01	5	102	3.7	98-106
		0.1	5	104	4.7	103-110
	m/z 304 →132	0.01	5	106	4.5	100-113
		0.1	5	102	3.2	98-107

Multiresidue methods for plant commodities

DFG S8 for plant commodities

Tilting (1989 10207) investigated the suitability of DFG multiresidue methods S8 and S19 for the determination of fenpropimorph in plant matrices.

Method S19 was found to be unacceptable as recoveries were below 50% and is not discussed further.

In multiresidue method S8, plant material is extracted with acetone, filtered and the acetone is diluted with water and extracted with dichloromethane. The organic phase evaporated to dryness, redissolved in dichloromethane and cleaned-up by charcoal/silica gel column chromatography. The eluate is reduced and subsequently made up to a defined volume with hexane for GC analysis (NPD or ECD detector).

The recoveries using method S 8 are shown in Table 56.

Table 56 Recovery data for multiresidue method DFG S8 for the determination of fenpropimorph

Matrix	Fortification Level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
Wheat straw	0.5	4	67	6.0	64-72
Wheat grain	0.5	4	81	10	70-87

From published data (DFG Method S 8), desmetryn might interfere with the fenpropimorph determination.

DFG S19 for plant commodities

Weeren and Pelz (1999 11462) examined further the applicability of DFG method S19 for the determination of fenpropimorph in lemon, tomato and wheat (grain). Multiresidue method S19 involves extraction of plant material with an acetone-water-mixture (2 + 1 v/v), after which sodium chloride is added and the solution extracted with dichloromethane. The organic phase evaporated to dryness, dissolved in a small volume of ethyl acetate, cleaned-up by gel permeation chromatography

and the eluate concentrated. The concentrate is analysed by GC or, in cases where the sample shows interferences, subject to further clean up on a silica gel column prior to chromatography. Due to the loss of recovery when extracting lemons at their natural pH values, prior to extraction with acetone sodium hydrogen carbonate is added to lemon samples to obtain a slightly alkaline medium. The extraction of fenpropimorph from rapeseed was performed according to DFG Clean-Up Method 5 followed by clean-up procedures according to DFG method S19.

Multiresidue method DFG S19 proved to be suitable for analysis of fenpropimorph in wheat (grain), rape (seed), tomato and lemon with LOQs of 0.01 mg/kg in wheat grain, tomato and lemon and 0.02 mg/kg in rapeseed. It was suggested metozachlor could be an interference in determining fenpropimorph in rapeseed using this method.

Table 57 Recovery data for multiresidue method DFG S19 for the determination of fenpropimorph residues in various crops

Matrix	Fortification Level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
Wheat Grain	0.01	5	95	9.2	89-110
	0.1	5	89	8.8	80-99
Rape seed	0.02	5	80.0	11	69-90
	0.2	5	93.4	14	81-116
Tomato	0.01	5	98.6	14	81-120
	0.1	5	101	9.2	91-115
Lemon	0.01	5	83.8	4.2	78-87
	0.1	5	80.2	7.5	76-89

Multi residue method 1 (Baumann et al., 1996 a 10470).

A brief overview of multi residue method 1 was made available. Fenpropimorph is extracted from fruits and vegetables with an acetone extraction and it is recommended that this is followed by a dichloromethane/petroleum ether partition. The organic phase is evaporated to dryness and the residue redissolved in iso-octane for the GC-analysis. Recoveries for the determination of fenpropimorph residues using GC-MS (ion trap detector) in samples (matrix not stated) fortified at 0.29 mg/kg were reported to average 98% with an RSD of 7.8% (n=10).

Residues in food and feedstuffs of Animal origin

Table 58 Overview of methods for the analysis of fenpropimorph and BF421-2 in animal commodities

		Method REM 167.03 (1995/11081)	Method 573/0 (2005/1026504)
Extraction and clean-up	Analytes	BF421-2	Fenpropimorph + BF421-2
	Matrix	Milk, muscle, liver, fat, kidney, egg	Milk, muscle, liver, fat, kidney, egg
	Extraction	Tissues: Add CH_3OH/H_2O buffer pH 9 (4:1) and macerate. An aliquot is diluted with CH_3OH/H_2O buffer pH 9 (4:1) and partitioned against hexane. The aqueous phase is retained. Egg: Extract/macerate with CH_3CN/buffer pH 9 (5:1 v/v), filter, add toluene to the filtrate, remove the solvent by evaporation to leave an aqueous residue, dissolve the residue in methanol and dilute with water/pH 9 buffer and partition with hexane. Retain the aqueous phase. Milk: Extract/macerate with CH_3CN/buffer pH 9 (90:5 v/v), filter, remove the CH_3CN by evaporation, dissolve the residue in methanol and dilute with water/pH 9 buffer and partition with hexane. Retain the aqueous phase. For tissues, milk and eggs, the aqueous phase is passed through a silica cartridge, eluted and the volume of the eluant reduced (N_2 flow, 40-50 °C). The volume is adjusted with 1%	Fat: add CH_3CN, HCl, isohexane (50 mL: 0.1M HCl 0.2 mL: isohexane 50 mL) and homogenise. Centrifuge and an aliquot of the CH_3CN phase is evaporated to dryness followed by liquid/liquid partition against dichloromethane and cyclohexane. Milk: add CH_3CN, HCl, isohexane (40 mL: H_2O 5 mL: 5M HCl 0.1 mL: isohexane 50 mL), homogenise, centrifuged and evaporated an aliquot to dryness. Re-dissolve in a mixture of CH_3OH/water (70/30 v/v) and add an equal volume of saturated NaCl. Liquid/liquid partition, 2× with CH_2Cl_2, add 0.05% NH_3 and partition against cyclohexane. Combine the organic phases, acidify with 1M HCl and evaporated to dryness. Other tissues, eggs: add CH_3OH/H_2O (70:30 v:v) homogenise, centrifuge and mix the supernatant with equal volume saturated

		Method REM 167.03 (1995/11081)	Method 573/0 (2005/1026504)
		phosphoric acid and if necessary filtered through a syringe filter disc for analysis by LC-UV or LC-MS	NaCl solution. Muscle, liver, kidney: partition against CH_2Cl_2 (2×), add 0.05% NH_3 and partition against cyclohexane. Combine the organic phases, add HCl and evaporate to dryness. Eggs: liquid/liquid partition, against CH_2Cl_2 (2×), the combined organic phases are acidified with 1M HCl and evaporated to dryness In all cases, the residue is dissolved in CH_3OH/Baker® water (50/50 v/v for determination of fenpropimorph and BF421-2 by LC-MS/MS.
	SPE column	C-18 bonded silica gel	
	Washing solvent	Buffer pH 9 diluted 1:10 with water	
	Eluent	pH9 buffer diluted with water 1:10/methanol (4:6 v/v)	
Chromatography	Type	LC	LC
	Analytical column	1. Sperisorb CN 2. Inertsil ODS II	Betasil C18
	Dimensions	100 mm, 2 mm id	100 mm, 2 mmm id
	Particle size	5 µm	
	Injection volume	20 µL	50 µL
	Mobile phase	water/CH_3CN/85% H_3PO_4 A: 840:160:1 v/v B: 780:220:1 v/v	A: methanol/formic acid 1000/1 v/v B: Baker® Millipore water/formic acid 1000/1 v/v
	Flow rate		0.6 mL/min
	Instrument	Linear UVIS 204 (Linear instruments , Reno, NE, USA)	PE Sciex API 4000 Mass Spectrometer
Detection	Quantitative detection	UV 200 nm 304→147, 304→130, 304→116	Positive ionization mode Fenpropimorph: 304→132, 304→147, 304→117 BF421-1: 334→91, 334→107
	LOQ	Tissues and eggs 0.01 mg/kg, Milk 0.002 mg	Tissues and eggs 0.005 mg/kg Milk: 0.001 mg/kg
	Whole method linearity (r^2)	0.01-0.2 µL/mL $r^2 \geq 0.99$	0.01-1 ng/mL $r^2 \geq 0.99$

Method REM 167.03 for animal commodities (Tribolet 1995a 11081)

Methods have been developed for BF421-2 (fenpropimorph acid) in animal matrices. Tissues: Methanol/H_2O buffer pH 9 (4:1) is added and the mixture macerated. An aliquot is diluted with methanol/H_2O buffer pH 9 (4:1) and partitioned against hexane. The aqueous phase is retained.

Egg: Acetonitrile/buffer pH 9 (5:1 v/v) is added and the mixture macerated, filtered, toluene added to the filtrate, and the solvent removed evaporation to leave an aqueous residue. The residue is dissolved in methanol and diluted with water/pH 9 buffer and partitioned against hexane. The aqueous phase is retained.

Milk: Extract/macerate with acetonitrile/buffer pH 9 (90:5 v/v), filter, and remove the solvent by evaporation, dissolve the residue in methanol and dilute with water/pH 9 buffer, partition against hexane and retain the aqueous phase.

For tissues, milk and eggs, the aqueous phase is passed through a SPE C18 silica cartridge, eluted and the volume of the eluant reduced (N_2 flow, 40–50 °C). The volume is adjusted with 1% phosphoric acid and if necessary filtered through a syringe filter disc for analysis by LC-UV after column switching from Spherisorb CN to Hypersil ODSII or by LC-MS

Recovery and repeatability data for the determination of BF421-2 (fenpropimorph-acid) residues using REM 167.03 in animal matrices are presented in Table 59. Average recoveries (lab 1) ranged from 72 to 105%. The LOQ was 0.01 mg/kg for all tissues and eggs and 0.002 mg/L for milk.

The %RSDs (lab 1) ranged from 1.2 to 10. Linearity was good for injections between 0.01 and 0.2 µg/mL ($r^2 \geq 0.99$)

The method was independently validated by a laboratory not involved in method development.

Table 59 Recovery data for analytical method REM 167.03 for the determination of BF-421-2 (fenpropimorph acid) in animal matrices

Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
Muscle	0.01	8	105	5.4	97-116
	0.1	8	97	2.3	94-101
Milk	0.002	8	100	5.7	94-107
	0.02	8	86	5.7	75-91
Liver	0.01	3	90	9.5	81-98
	0.1	3	103	2.4	101-106
Kidney	0.01	3	91	3.2	89-94
	0.1	3	97	1.2	96-98
Egg	0.01	3	72	10	66-80
	0.1	3	76	2.7	74-78
Muscle (Lab 2)	0.01	3	89	11	83-100
Milk (Lab 2)	0.002	3	63	2.4	62-65

Tilting (2001a 1014760) conducted an independent validation of REM 167.03 and describe a confirmatory method for BF421-2. The confirmatory method used the same clean up as REM 167.03, but determination was achieved by LC-MS using HPLC without column switching (m/z 334).

Recovery and repeatability data for the determination of BF421-2 (fenpropimorph-acid) residues in animal matrices are presented in Table 60. Average recoveries ranged from 74 to 110%. The LOQ was 0.01 mg/kg for all tissues and eggs and 0.002 mg/kg for milk. The %RSDs ranged from 1.4 to 12. Linearity was good for injections between 0.01 and 0.2 µg/mL for method 987 and 0.002 to 0.01 µg/mL for the confirmatory method ($r^2 \geq 0.99$).

The use of an LC-MS detection system proved to be suitable for the confirmation of residue results obtained by LC-UV.

Table 60 Recovery data for analytical methods for the determination of the metabolite BF-421-2

Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
LC-UV					
Milk (cow)	0.002	5	103	5.4	95-108
	0.02	5	90	5.1	83-95
Muscle (cow)	0.01	5	110	1.4	108-112
	0.1	5	94	3.0	90-97
Egg (hen)	0.01	5	107	8.9	95-118
	0.1	5	74	12	60-83
Confirmatory LC-MS m/z 334					
Milk (cow)	0.002	3	81	6.7	77-88
Muscle (cow)	0.01	3	97	6.7	90-103
Egg (hen)	0.01	3	88	5.0	84-93

Method 573/0 for animal commodities (Benz and Mackenroth 2005 1026504)

Method 573/0 was developed by Benz and Mackenroth (2005 1026504) to enable determination of both fenpropimorph and BF471-2. In this method, fenpropimorph and its metabolite BF421-2 are extracted from muscle, liver, kidney and eggs with a mixture of methanol and water (70/30 v/v). An aliquot of the extract is centrifuged and a sample of the supernatant mixed with an equal volume of saturated NaCl solution. Clean-up in the case of muscle, liver, kidney is by liquid/liquid partition, 2× with dichloromethane and on the final partition 0.05% NH_3 solution is added prior to partitioning against cyclohexane. The combined dichloromethane and cyclohexane phases are acidified with 1N HCl and evaporated to dryness at 30 °C under a stream of N_2.

Clean-up in the case of eggs is by liquid/liquid partition, 2× with dichloromethane, the combined dichloromethane phase is acidified with 1N HCl and evaporated to dryness at 40 °C under a stream of N_2.

In case of fat, the extraction solvent is a mixture of acetonitrile, hydrochloric acid and isohexane (acetonitrile 50 mL, 0.1 N HCl 0.2 mL, isohexane 50 mL). An aliquot of the extract is centrifuged and a sample of the acetonitrile phase evaporated to dryness at 30 °C under a stream of N_2. Clean-up is by liquid/liquid partition against dichloromethane and cyclohexane.

For milk, the extraction solvent is a mixture of acetonitrile, hydrochloric acid and isohexane (acetonitrile 40 mL, water 5 mL, 5 N HCl 0.1 mL, isohexane 50 mL). The extract is centrifuged and an aliquot of the acetonitrile extract is evaporated to dryness at 40 °C under a stream of N_2 and the residue redissolved in a mixture of methanol and water (70/30 v/v) and an equal volume of saturated NaCl solution added. Clean-up is by liquid/liquid partition, 2× with dichloromethane and on the final partition 0.05% NH_3 solution is added prior to partitioning against cyclohexane. The combined dichloromethane and cyclohexane phases are acidified with 1N HCl and evaporated to dryness at 40 °C under a stream of N_2.

In all cases, the residue is redissolved in methanol/Baker® water (50/50 v/v for final determination of fenpropimorph and the metabolite BF421-2 by LC-MS/MS.

Recovery and repeatability data for the determination of fenpropimorph and BF421-2 residues in animal matrices are presented in Tables 61 and 62.

For fenpropimorph average recoveries ranged from 73 to 89% for m/z 304→147. The LOQ was 0.005 mg/kg for all tissues and eggs and 0.001 mg/kg for milk. The %RSDs ranged from 2.5 to 13.5. Linearity was good ($r^2 \geq 0.99$) for injections between 0.01 and 1.0 ng/mL.

For BF421-2 average recoveries ranged from 88 to 103% for m/z 334→107. The LOQ was 0.005 mg/kg for all tissues and eggs and 0.001 mg/kg for milk. The %RSDs ranged from 1.7 to 15. Linearity was good ($r^2 \geq 0.99$) for injections between 0.01 and 1.0 ng/mL.

Specificity. For the quantification of fenpropimorph, the transition 304 → 147 is used because of a higher response. The transition 304 → 132 is recommended for confirmatory purposes.

For the quantification of BF421-2, the transition 334 → 107 is used. The transition 334 → 91 is recommended for confirmatory purposes.

Extracts were stable when stored refrigerated; muscle (21 d), liver (21 d), fat (30 d) kidney (19 d) and milk (7d). Standard solutions were stable refrigerated and in the dark for at least 46 days.

Table 61 Recovery data for method 573/0 for the determination of fenpropimorph residues

			304→147			304→132		
Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range (%)	Mean recovery (%)	RSD (%)	Recovery range (%)
Muscle	0.005	5	81	2.7	79-85	82	3.0	79-84
	0.05	5	82	3.8	78-86	84	4.2	80-89
Liver	0.005	5	79	2.9	75-81	80	4.7	75-85
	0.05	5	81	7.6	72-87	80	7.4	71-86
Fat	0.005	5	89	6.2	80-93	91	4.8	85-95
	0.05	5	73	2.6	71-76	76	2.4	74-78
Kidney	0.005	4	75	5.3	69-78	74	3.8	71-78
	0.05	5	78	2.9	76-81	77	2.6	74-79
Milk	0.001	5	81	3.9	76-84	83	4.4	78-87
	0.01	5	84	2.5	81-86	84	4.0	80-89
Egg	0.005	5	81	13.5	71-98	78	11	71-92
	0.05	5	77	5.2	72-81	78	5.2	72-82

Table 62 Recovery data for method 573/0 for the determination of BF421-2 residues

Matrix	Fortification level (mg/kg)	N	334→107 Mean recovery (%)	334→107 RSD (%)	334→107 Recovery range (%)	334→91 Mean recovery (%)	334→91 RSD (%)	334→91 Recovery range (%)
Muscle	0.005	5	91	1.7	89-93	91	4.5	85-94
	0.05	5	92	3.4	88-95	95	4.5	90-100
Liver	0.005	5	88	15	65-97	89	15	66-101
	0.05	5	92	4.5	85-95	95	4.3	88-100
Fat	0.005	5	88	2.8	85-92	88	6.9	83-96
	0.05	5	89	3.5	87-94	90	2.5	87-92
Kidney	0.005	4	91	4.7	86-96	89	12	76-101
	0.05	5	87	4.5	82-91	91	5.9	85-100
Milk	0.001	5	103	2.0	100-106	104	6.4	96-112
	0.01	5	98	9.9	82-107	97	7.0	87-106
Egg	0.005	5	91	3.2	89-95	100	8.8	86-110
	0.05	5	91	4.9	87-99	99	7.9	86-106

An independent laboratory validation was conducted for method 573/0 by Schulz (2005a 1026505) (Table 63 and 64). For fenpropimorph mean recoveries ranged from 70 to 102% for m/z 304→147. The LOQ was 0.005 mg/kg for all tissues and eggs and 0.001 mg/kg for milk. The %RSDs ranged from 2.2 to 17. Linearity was good ($r^2 \geq 0.99$) for injections between 0.01 and 1.0 ng/mL.

For BF421-2 mean recoveries ranged from 77 to 102% for m/z 334→107. The LOQ was 0.005 mg/kg for all tissues and eggs and 0.001 mg/kg for milk. The %RSDs ranged from 3.2 to 17. Linearity was good ($r^2 \geq 0.99$) for injections between 0.01 and 1.0 ng/mL.

Table 63 Independent laboratory recovery data for analytical method 573/0 for the determination of fenpropimorph residues in animal matrices

Matrix	Fortification level (mg/kg)	N	304→147 Mean recovery (%)	304→147 RSD (%)	304→147 Recovery range (%)	304→132 Mean recovery (%)	304→132 RSD (%)	304→132 Recovery range (%)
Muscle	0.005	5	87	8.7	74-93	87	8.2	76-94
	0.05	5	95	14	77-114	92	19	68-117
Liver	0.005	5	79	8.9	69-88	79	8.9	67-80
	0.05	5	102	17	89-123	102	17	85-123
Fat	0.005	5	87	9.8	72-93	88	10	73-97
	0.05	5	92	2.2	90-94	90	2.6	87-93
Kidney	0.005	4	84	10	71-95	86	9.9	74-97
	0.05	5	83	13	65-90	84	14	64-94
Milk	0.001	5	102	10	91-114	94	16	69-107
	0.01	5	101	11	82-108	102	9.2	86-109
Egg	0.005	5	70	9.4	61-79	74	5.7	69-79
	0.05	5	74	11	66-86	78	10	70-90

Table 64 Independent laboratory recovery data for analytical method 573/0 for the determination of BF421-2 residues in animal matrices

Matrix	Fortification level (mg/kg)	N	334→107 Mean recovery (%)	334→107 RSD (%)	334→107 Recovery range (%)	334→91 Mean recovery (%)	334→91 RSD (%)	334→91 Recovery range (%)
Muscle	0.005	5	91	4.3	83-93	91	5.4	86-97
	0.05	5	96	11	87-110	96	12	83-109
Liver	0.005	5	83	7.6	73-88	91	7.2	79-95
	0.05	5	102	14	80-115	106	14	83-120
Fat	0.005	5	86	4.6	80-89	85	4.3	81-91
	0.05	5	89	3.2	86-92	88	3.3	86-93
Kidney	0.005	4	92	17	76-118	91	18	70-116
	0.05	5	77	7.1	71-84	77	11	68-88
Milk	0.001	5	102	11	93-119	106	8.8	97-114
	0.01	5	96	9.5	89-112	99	9.0	93-114

Matrix	Fortification level (mg/kg)	N	334→107 Mean recovery (%)	334→107 RSD (%)	334→107 Recovery range (%)	334→91 Mean recovery (%)	334→91 RSD (%)	334→91 Recovery range (%)
Egg	0.005	5	93	6.6	85-101	95	9.5	83-106
	0.05	5	89	11	75-100	91	11	76-101

Radiovalidation of the methanol buffer extraction step for BF421-2

Extractabilities with morpholine-labelled tissue samples were investigated using solvent extraction with methanol/buffer systems (methanol/buffer pH = 9, 4/1 v/v) as applied in the analytical methods 573/0 and REM 167.03. Samples of liver, kidney, fat and muscle were homogenised with methanol and buffer (0.017 M KH_2PO_4/ 0.043 M $Na_2B_4O_7$, pH 9), filtered and the residue washed with methanol/water (80:20 v/v). The filtrates were combined and processed for analysis by HPLC.

Samples of milk were mixed with acetonitrile and buffer, filtered, the filter and beaker washed with acetonitrile and the combined solutions reduced in volume at 40 °C to an oily residue. The oily residue was dissolved in methanol followed by the addition of water and buffer and partitioned with hexane. An aliquot of the aqueous phase was processed for analysis by HLPC.

Using the methanol/buffer system, the following extractabilities could be obtained for liver, kidney, fat, muscle and milk: 68% of the TRR, 77%, 71%, 70% and 17%, respectively. The HPLC analysis of the metabolism study extracts compared to the HPLC analysis of the extract obtained with the methanol/buffer systems, showed that the metabolite pattern, especially with respect to BF421-2, were qualitatively and quantitatively comparable. Extractabilities are shown in Table 65.

Table 65 Extractability of Tissues: Comparison of extraction procedures used for residue analysis versus metabolism (morpholine label) (Fabian and Knoell 2003a 1014911)

Matrix	TRR	Metabolism study methanol extract		Residue analysis methanol/buffer extract				Accountability
		BF421-2 (**A**)		Methanol/buffer extract		BF421-2 (**B**)		B/A
	mg/kg	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	%
Liver	0.777	0.442	56.8	0.527	67.8	0.306	39.4	69.4
Kidney	0.257	0.065	25.3	0.199	77.3	0.060	23.4	92.5
Fat	0.238	0.017	30.0	0.168	70.8	0.092	38.9	129.7
Muscle	0.055	0.080	33.5	0.039	70.4	0.014	25.3	75.5
Milk	0.160	0.005	2.9	0.027	16.8	0.012	7.8	[A]

[A] due to very low absolute amounts of BF421-2, quantitation not applicable

Multiresidue method DFG S19 for poultry matrices

Bacher (2001 1019510) investigated the applicability of an adapted multi-residue method DFG S19 for the determination of fenpropimorph residues in poultry matrices (eggs, muscle, liver, and fat). Residues of fenpropimorph are extracted from meat, liver, and egg specimens with water/acetone (1/2 v/v) and are partitioned into the organic phase by addition of ethyl acetate/cyclohexane (1/1 v/v). An aliquot of the raw extract is further purified by gel permeation chromatography (GPC) and a silica gel (SiO_2) fractionation. The final extract is analysed by GC-NPD. In case peak identity is doubtful, the LC-MS/MS method (as developed for the fat extracts) can be used for determination.

Fat specimens are extracted using acetonitrile/acetone (9/1 v/v) with addition of a Calflo E/Celite (2/1 w/w) mixture. An aliquot of the raw extract is transferred into methanol/water (1/1 v/v) and analysed by reversed phase LC-MS/MS.

Recovery and repeatability data for the determination of fenpropimorph residues in animal matrices using modified DFG S19 are presented in Table 66. Average recoveries ranged from 76 to 103%. The LOQ was 0.025 mg/kg for all tissues and eggs. The %RSDs ranged from 11 to 19. Linearity was good ($r^2 \geq 0.99$) for injections between 0.005 and 1.0 µg/mL for GC-NPD and between 0.001 and 0.5 µg/mL for LC-MS/MS.

Table 66 Recovery data for multiresidue method DFG S19 for the determination of fenpropimorph in poultry commodities

Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
Muscle	0.025	5	85	11	75-94
	0.25	5	90	15	72-107
Liver	0.025	5	87	19	64-105
	0.25	5	98	12	84-106
Eggs	0.025	5	100	11	88-116
	0.25	5	103	13	86-122
Fat	0.025	6	76	13	61-89
(m/z 116, 130, 147)	0.25	5	90	11	78-102

An independent laboratory validation of DFG S19 was conducted by Atkinson (2002 1007053). Recovery and repeatability data for the determination of fenpropimorph residues in animal matrices are presented in Table 67. Average recoveries ranged from 74.0 to 84%. The LOQ was 0.025 mg/kg for all muscle and eggs. The %RSDs ranged from 3.0 to 12.5. Linearity was good ($r^2 \geq 0.99$) for injections between 0.005 and 1.0 µg/mL for GC-NPD.

Table 67 Independent laboratory recovery data for multiresidue method DFG S19 adapted for the determination of fenpropimorph in animal commodities

Matrix	Fortification level (mg/kg)	N	Mean recovery (%)	RSD (%)	Recovery range
Muscle	0.025	5	75	6.3	69-82
	0.25	5	79	5.3	73-84
Eggs	0.025	5	74	12	58-82
	0.25	5	84	3.0	82-88

Radiovalidation of the extraction steps used in DFG S19 for poultry matrices

The residue analytical method for the determination of fenpropimorph in animal matrices is an adapted multi-residue method DFG S19 (2001/1019510), which uses acetone/acetonitrile (1:9) for the extraction of fat and water/acetone for the extraction of other matrices. The efficiency of these extraction solvents was investigated in the course of the metabolism study with samples of liver and fat of the phenyl label, since in these matrices fenpropimorph accounts for 28.8% (0.072 mg/kg) and 18.0% (0.061 mg/kg) of the extracted radioactivity, respectively.

The samples were extracted in duplicate as described in method DFG S19 and the radioactivity of the crude extracts was determined by LSC counting. The extracts were additionally analysed by HPLC. The extractabilities achieved with the residue method are compared with the results of the metabolism study in Table 68. The respective quantities of the extracted fenpropimorph are summarised in Table 69.

Table 68 Extractability of liver and fat with the residue analytical method for fenpropimorph

Matrix	TRR (mg eq/kg)	Methanol extract (metabolism study) (mg eq/kg)	Acetone/CH_3CN or water/acetone (residue method) (mg eq/kg) [A]
Liver, Phenyl label	0.250	0.139	0.105
Fat, Phenyl label	0.337	0.107	0.110

[A] Values represent the mean of two separate extractions

The solvent mixture acetone/acetonitrile used for fat yielded the same extractability as the methanol used in the metabolism study, whereas acetone/water (2:1) used with liver proved slightly less efficient, presumably because the polar liver metabolites are not as well extracted as with methanol.

However, the quantification of the extracted parent compound demonstrates that the extraction procedure of the residue analytical method DFG S19 is suitable for the quantitative

determination of fenpropimorph, since 94% and 115% of the amount of fenpropimorph of the metabolism study were found with the residue method.

Table 69 Comparison of the quantities of fenpropimorph determined in the metabolism study versus residue analytical method

Matrix	Fenpropimorph–metabolism study (mg/kg)	Fenpropimorph–residue method DFG S19 (mg/kg)[A]	Recovery (%)
Liver, phenyl label	0.072	0.068	94
Fat, phenyl label	0.061	0.070	115

[A] Values represent the mean of two separate extractions

Stability of pesticide residues in stored analytical samples

A number of studies of the stability fenpropimorph in plant commodities and fenpropimorph and BF421-2 in animal commodities following freezer storage of samples were made available to the Meeting.

Tilting (1993a 10781) investigated the deep freeze stability of fenpropimorph in cereals over a period of two years. Untreated wheat grain, wheat green plant and wheat straw samples were homogenised and fortified with 1.0 mg/kg fenpropimorph. The samples were stored under the usual storage conditions for field samples (polyethylene bottle, -20 °C). The samples were analysed with method no. 241.

The results of the analyses at the various sampling dates are given in Table 70.

Table 70 Storage stability of fenpropimorph in wheat commodities

Days storage	Wheat green plant		Wheat straw		Wheat grain	
	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)
0	0.94	96	1.08	109	0.92	95
30/32[A]	0.72	87	1.06	88	0.62	89
62/60[A]	0.83	88	0.95	82	0.55	50
118/119[A]	0.91	101	0.89	89	0.72	80
180	1.00	97	0.93	88	0.75	78
243/242[A]	0.71	83	1.02	103	0.46	52
362	0.84	87	1.00	101	0.59	59
742/735[A]	0.96	106	0.99	109	1.04	112

[A] sampling dates for straw

Fenpropimorph is stable on frozen storage of wheat green plant, straw and grain for at least 735 days.

The frozen storage stability of fenpropimorph in plant matrices was also investigated by Benz and Mackenroth (2003a 1004669, 2003b 1001287). Samples were spiked with the fenpropimorph at a concentration level of 0.5 mg/kg. The spiked samples were stored under the usual storage conditions for field samples (polyethylene containers, -20 °C). At different intervals, samples were analysed with method no. 456/0. Procedural recoveries for fenpropimorph averaged at about 82% in banana pulp, at about 86% in banana peel, at about 91% in sunflower seed, at about 80% in sugar beet leaves and at about 85% in sugar beet root (Table 71).

Table 71 Storage stability of fenpropimorph in various plant matrices

Days	Banana pulp		Banana peel		Sunflower seed		Sugar beet leaves w. tops		Sugar beet root	
storage	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)
0	0.48 [A]	83	0.47 [A]	93	0.50 [A]	92	0.41 [A]	89	0.48 [A]	86
30	0.42	82	0.40	90	0.40	98	0.44	88	0.47	90

Days storage	Banana pulp		Banana peel		Sunflower seed		Sugar beet leaves w. tops		Sugar beet root	
	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)
90	0.38	91	0.39	94	0.40	97	0.32	66	0.41	98
182	0.32	64	0.31	65	0.30	84	0.29	79	0.33	74
366	0.41	90	0.39	88	0.37	93	0.31	86	0.43	90
546	0.32	78	0.34	87	0.39	82	0.25	73	0.40	71
643	0.40	83	0.41	70	0.39	94	0.23	72	0.37	84
723	0.40	77	0.36	79	0.38	89	0.26	79	0.38	78

[A] Mean of five replicates day 0, two replicates for other times

The storage stability results obtained after 723 days show that the percent remaining (uncorrected for procedural recoveries) is 83% in banana pulp, 76% in peel, 76% in sunflower seed, 64% in sugar beet leaves with tops and 78% in roots. If corrected for procedural recoveries the percentage remaining after 723 days frozen storage is calculated to be 72% for sugar beet leaves and tops.

Thiaener (2009 1050905) studied the deep freeze stability of fenpropimorph and its metabolite BF421-2 in muscle, fat, liver, kidney, milk, skimmed milk and cream from the cow. The study was conducted over a period of about two years and four months. Muscle, liver, fat and kidney samples were spiked with the test items at a concentration level of 0.5 mg/kg. Milk, skimmed milk and cream samples were spiked with the test items at a concentration level of 0.1 mg/kg. The spiked samples were stored under the usual storage conditions for samples (plastic containers, -20 °C). At different intervals samples were analysed with method no. 573/0. The LOQ of the method for fenpropimorph and its metabolite BF421-2 is 0.005 mg/kg in tissues and 0.001 mg/kg in milk. Procedural recoveries for fenpropimorph averaged between 84 and 105%, for BF421-2 between 94 and 104%. The results are shown in tables 72 and 73 for fenpropimorph and Tables 74 and 75 for BF421-2.

Table 72 Summary of the stability data of fenpropimorph

Days storage	Muscle		Fat		Liver		Kidney	
	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)
0	0.48	99	0.47	99	0.42	97	0.45	91
30	0.38	87	0.62	122	0.42	83	0.44	103
90	0.30	70	0.46	89	0.30	69	0.33	74
180	0.31	85	0.44	91	0.28	76	0.33	78
270	0.36	83	0.46	89	0.34	79	0.31	77
365	0.34	82	0.47	99	0.36	87	0.27	77
540	0.43	109	0.38	86	0.47	95	0.38	106
660	0.41	109	0.44	98	0.29	87	0.28	87
760	0.41	106	0.53	97	0.38	89	0.27	84
880	0.30	70	0.46	103	0.34	85	0.43	98

Table 73 Summary of the stability data of fenpropimorph

Days storage	Milk		Skimmed Milk		Cream	
	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)
0	0.11	111	0.09	86	0.11	114
30	0.07	78	0.12	105	0.10	91
90	0.09	92	0.08	68	0.10	94
180	0.10	99	0.08	90	0.10	112
270	0.10	98	0.09	81	0.11	110
365	0.09	83	0.10	93	0.10	97
540	0.12	86	0.11	108	0.11	107
660	0.11	114	0.10	103	0.13	116
760	0.10	105	0.09	94	0.11	122

Days storage	Milk		Skimmed Milk		Cream	
	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)
880	0.10	109	0.08	72	0.09	96

The storage stability results obtained after 880 days show that the percent fenpropimorph remaining (uncorrected for procedural recoveries) is 62% in muscle (88% if corrected for procedural recoveries), 98% in fat, 81% in liver, 96% in kidney, 91% in milk, 89% in skimmed milk and 82% in cream. For muscle, the %remaining at 760 days was 85%. Overall, the data suggest residues of fenpropimorph are stable in animal commodities for at least 880 days.

Table 74 Summary of the stability data of the metabolite BF421-2

Days storage	Muscle		Fat		Liver		Kidney	
	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)
0	0.50	101	0.44	93	0.48	97	0.49	98
30	0.61	113	0.56	114	0.58	114	0.62	125
90	0.50	97	0.44	99	0.46	97	0.48	97
180	0.45	90	0.38	85	0.46	102	0.46	97
270	0.50	94	0.42	91	0.48	98	0.48	100
365	0.48	97	0.44	95	0.46	100	0.48	101
540	0.52	107	0.38	84	0.46	100	0.52	106
660	0.54	111	0.37	89	0.42	107	0.53	105
760	0.55	113	0.50	101	0.44	101	0.36	96
880	0.40	81	0.44	93	0.30	85	0.42	91

Table 75 Summary of the stability data of metabolite BF421-2

Days storage	Milk		Skimmed Milk		Cream	
	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)	Residue (mg/kg)	Procedural recovery (%)
0	0.11	110	0.10	99	0.11	106
30	0.13	120	0.13	128	0.13	125
90	0.10	104	0.10	101	0.10	102
180	0.10	104	0.10	88	0.11	111
270	0.10	99	0.10	107	0.11	106
365	0.11	106	0.11	110	0.12	113
540	0.10	109	0.11	105	0.11	106
660	0.10	113	0.10	90	0.08	91
760	0.08	94	0.11	105	0.07	62
880	0.08	78	0.10	101	0.09	78

The storage stability results obtained after 880 days show that the percent BF421-2 remaining (uncorrected for procedural recoveries) is 80% in muscle, 100% in fat, 62% in liver (71% if corrected for procedural recoveries), 86% in kidney, 73% in milk, 100% in skimmed milk and 82% in cream. For liver the percentage remaining at 760 days was 92%. Overall, the data suggest residues of BF421-2 are stable in animal commodities for at least 880 days.

Investigations on the stability of residues were also performed in parallel with the cow feeding studies (Hafemann and Mackenroth 2005 1025538). Muscle, fat, liver, kidney, milk, skimmed milk and cream from untreated animals were fortified with known amounts of BF421-2 and stored frozen at -20 °C in plastic containers. The storage period covered the time range between sampling and analysis. The samples were analysed with method No. 987. Procedural recoveries for BF421-2 averaged at about 92% in muscle, 89% in fat, 91% in liver, 76% in kidney, 82% in milk, 83% in skimmed milk and 75% in cream.

The results are presented in Table 76. The residues of BF421-2 were shown to be stable in all animal matrices and for the time intervals tested.

Table 76 Summary of the stability data of BF421-2

Days storage	Muscle Residue (mg/kg)	Procedural recovery (%)	Fat Residue (mg/kg)	Procedural recovery (%)	Liver Residue (mg/kg)	Procedural recovery (%)	Kidney Residue (mg/kg)	Procedural recovery (%)
0	0.20	94	0.16	90	1.73	84	0.32	76
7	0.17	90	0.19	88	1.83	93	0.29	74
14	0.20	83	0.15	74	1.73	94	0.37	76
28	0.19	94	0.17	90	1.59	84	0.29	76
60	0.20	89	0.18	89	1.75	96	0.32	88
90	0.21	115	0.18	104	1.60	96	0.30	78
120	0.18	95	0.18	87	1.67	92	0.25	74
180	0.18	76	0.17	92	1.50	88	0.29	69
225					1.58	100	0.28	74
270					1.61	94		
330					1.55	80		

Table 77 Summary of the stability data of metabolite BF421-2

Days storage	Milk Residue (mg/kg)	Procedural recovery (%)	Skimmed Milk Residue (mg/kg)	Procedural recovery (%)	Cream Residue (mg/kg)	Procedural recovery (%)
0	0.057	89	0.058	97	0.055	92
7	0.058	87	0.050	83	0.037	72
14	0.049	83	0.051	83	0.042	64
28	0.055	89	0.056	97	0.043	92
60	0.050	70	0.049	75	0.051	57
90	0.045	71	0.049	81	0.051	69
120	0.052	88	0.046	80	0.040	65
180	0.047	75	0.044	76	0.049	86
225	0.053	85	0.043	72	0.048	74
270	0.043	82				

USE PATTERNS

Fenpropimorph is a systemic fungicide belonging to the morpholine group of compounds. It inhibits the formation of *appressoria* and *haustoria* and controls mycelial growth and sporulation. Fenpropimorph is used to control a range of fungal diseases in cereals, sugar beet and banana.

Table 78 Selected registered uses of fenpropimorph on banana and plantain (foliar)

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
Banana	Belize	OL	All stages of development, add emulsifier	0.44–0.88	-	1-8	7–14	0
Banana	Bolivia	OL		0.62 – 0.7	20-30	2-6	15-21	0
Banana	Cameroon	EC	All stages of development	0.262–0.375	14✈-20	-	-	0
Banana	Cameroon	OL	Add emulsifier	0.440	15–25 10–15✈	ns	18 – 21	0
Banana	Colombia	EC	Add emulsifier	0.375	18-23	3	11–16	0
Banana	Colombia	OL	Add emulsifier	0.44–0.6	18-23		-	0
Banana	Costa Rica	OL	All stages of development, add emulsifier	0.44–0.88	-	1-8	7–14	0
Banana	Cuba	OL	Add emulsifier	0.44–0.88	-	1- 8	10–42	0
Banana	Dominica Rep.	EC	Recommended bagged	0.3 – 0.45	45-65 18–23✈	3	10–16	0
Banana	Dominica Rep.	OL	All stages of development, add emulsifier	0.44–0.88	-	1-8	10-42	0

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
Banana	Ecuador	EC		0.375		3	12-15	0
Banana	Ecuador	OL	Add emulsifier	0.616 – 0.88			10-28	0
Banana	Ghana	OL	Add emulsifier if using oil/water emulsion	0.440	15–25 oil 10–15✈ oil	-	14–42	0
Banana	Guatemala	EC		0.3 – 0.45	45-65 18–23✈	3	10–16	0
Banana	Guatemala	OL	All stages of development, add emulsifier	0.44–0.88	-	1-8	7–14	0
Banana	Honduras	OL	All stages of development, add emulsifier	0.44–0.88	-	1-8	7–14	0
Banana	Ivory Coast	EC		0.263– 0.375	20 14✈	2	21–30	0
Banana	Ivory Coast	OL		0.440	15–25 10–15✈	-	10-21	0
Banana	Mexico	OL		0.616	200-400			0
Banana	Nicaragua	OL	All stages of development, add emulsifier	0.44–0.88	-	1-8	7–14	0
Banana	Panama	OL	All stages of development, add emulsifier	0.44–0.88	-	1-8	7–14	0
Banana	Venezuela	EC		0.525	10-20	4	21	0
Plantain	Belize	OL	All stages of development, add emulsifier	0.44–0.88	-	1-8	7–14	0
Plantain	Cameroon	EC	All stages of development	0.262–0.375	14✈-20	-	-	0
Plantain	Cameroon	OL	Add emulsifier	0.440	15–25 10–15✈	ns	10 – 14	0
Plantain	Costa Rica	OL	All stages of development, add emulsifier	0.44–0.88	-	1-8	7–14	0
Plantain	Cuba	OL	Add emulsifier	0.44–0.88		Max 8	10 –42	0
Plantain	Dominica Rep.	EC	Recommended bagged	0.3 – 0.45	45 -65 18–23✈	3	10–16	0
Plantain	Dominica Rep.	OL	All stages of development, add emulsifier	0.44–0.88	-	1-8	10-42	0
Plantain	Ghana	OL	Add emulsifier if using oil/water emulsion	0.440	15–25 oil 10–15✈ oil	-	14–42	0
Plantain	Guatemala	EC		0.3 – 0.45	45-65 18–23✈	3	10–16	0
Plantain	Guatemala	OL	All stages of development, add emulsifier	0.44–0.88	-	1-8	7–14	0
Plantain	Honduras	OL	All stages of development, add emulsifier	0.44–0.88	-	1-8	7–14	0
Plantain	Ivory Coast	EC		0.263– 0.375	20 14✈	2	21–30	0
Plantain	Ivory Coast	OL		0.440	15–25 10–15✈	-	10-21	0
Plantain	Nicaragua	OL	All stages of development, add emulsifier	0.44–0.88	-	1-8	7–14	0
Plantain	Panama	OL	All stages of development, add emulsifier	0.44–0.88	-	1-8	7–14	0

✈ application using aircraft

Table 79 Selected registered uses of fenpropimorph on sugar beet (foliar)

Crop	Country		GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
Sugar beet	Belgium	SE	From 1st appearance of symptoms	0.175	-	-	-	45
Sugar beet	Chile	SE		0.1125	200–400	1–2	20–30	35
Sugar beet	Croatia	SE		0.250-0.300	200-600	1-2		35
Sugar beet	Czech Rep.			0.250	200–400	1–2	21	42
Sugar beet	Greece	EC		100-150 mL/1000mq 0.75-1.125	40/1000mq 400	-	-	7
Sugar beet	Greece	EC		100mL/1000mq 0.25	40/100mq 4000	1-2	10-14	45
Sugar beet	Hungary	SE		0.250-0.300	400–600	1–2	14–28	35
Sugar beet	Ireland	EC		0.750	250	-	-	28
Sugar beet	Italy	EC		0.750		1–3	15-20	21
Sugar beet	Luxembourg	SE		0.175				45
Sugar beet	Netherlands	SE		0.25		1–2	21	46[a]
Sugar beet	Poland	SE	BBCH39-49	0.25	100–400	1		35[b]
Sugar beet	Romania	SE		0.250	200–300	1–2	-	35
Sugar beet	Serbia	SE		0.175	200–400	1–2		35
Sugar beet	Switzerland	SE		0.3	200 – 400	1–2	21	-
Sugar beet	Turkey	SE		0.1875	-	2	15–20	28

[a] The foliage and heads of sugar beets treated with fenpropimorph must not be used for animal feed.

[b] Period between the last application of the product on plants destined for animal feed and the day when animals can be fed the plants (withdrawal period for feed): sugar beet leaves – 21 days

Table 80 Selected registered uses of fenpropimorph on barley

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
Barley	Austria	SE	1st application spring, 2nd up to start flowering BBCH 61	0.375	200–400	1–2	-	-
Barley	Belarus	SE		0.25-0.312	200- 400	1	-	30
Barley	Belgium	SE	1st button to last leaf with last application at last leaf	0.5	200–400	1–2	-	35
Barley	Belgium	EC	1st button (BBCH 30/31) with 2nd application at last leaf (BBCH 37)	0.562 – 0.75	200–400	1–2		28
Barley	Belgium	SE	button (BBCH 31) until last leaf (BBCH 39)	0.4	200–400	1–2	-	35
Barley	Belgium	SE	button (BBCH 31) until last leaf (BBCH 39)	0.375	200–400	1–2	28	-
Barley	Belgium	SE	1st at 1st button to last leaf (BBCH 37), 2nd in last leaf-beards stage	0.375	200–300	1	-	-

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
			(BBCH 45)					
Barley	Bulgaria	SE	Tillering (BBCH 25) to end of flowering (BBCH 69)	0.2 – 0.25	200–400	1–2	21	50
Barley	Bulgaria	SE	During tillering to beginning of elongation	0.2	200–490	1–2	21	35
Barley	Chile	SE	End of tillering or start of stalking to ear newly exposed	0.12–0.15	150 – 200 30 – 50✈	1-2	-	35
Barley	Chile	SE	Start of stalking to ear phase up to 0.429, ear emerged up to 0.321	0.214-0.429	150 – 200 30-50✈	1-2	-	35
Barley	Croatia	SE		0.250-0.300	200-600	1-2		42
Barley	Czech Rep.			0.75	100–400	1–2	21	35
Barley	Czech Rep.	SE		0.25	200–400	1–2	21	35
Barley	Czech Rep.	SE		0.15		1 -2		35
Barley	Czech Rep.	SE	Leaf/spikelet disease BBCH 25-61	0.4		1–2		35
Barley	Estonia	SE	Sprouting until opening calyx (BBCH 30-49)	0.23-0.46 max 0.46/season	200-400	1-2		35
Barley	Estonia	SE	AS30-69	0.21-0.42, max 0.46/season	100 – 400	1-2		35
Barley	Estonia	EC		0.375	100–400	1–2	21	35
Barley	Estonia	SE		0.188-0.375	100–400	1–2	21	35
Barley	France	SE		0.375	1	1	1	35
Barley	France	SE		0.4		1		35
Barley	Germany	EC	Until beginning flowering (BBCH 61)	0.750	200–400	1–2	-	-
Barley	Germany	SE	1st from mid-tillering (BBCH 25) last up to start flowering (BBCH 61)	0.375	200–400	1–2	-	-
Barley	Germany	SE	BBCH 25-61	0.15	200–400	1–2	-	-
Barley	Germany	SE	BBCH 25-61	0.375	200–400	1–2	-	-
Barley	Germany	SE		0.4	200–400	1–2		35
Barley	Greece	EC		0.750	400–600	1–2	-	35
Barley	Hungary	SE		0.2–0.3	250 – 350 60-80✈	1–2	-	35
Barley	Hungary	SE	1st during tillering, 2nd before blooming	0.38 – 0.476	50 – 70✈	1–2	-	35
Barley	Hungary	SE		0.2–0.32	250–300	1–2	21	35
Barley	Ireland	SE	Before GS59	0.4	>200	1–2	-	-
Barley	Ireland	EC		0.750	220-450	1–2	-	35
Barley	Ireland	SE	Before GS59	0.15	>200	1–2	-	-
Barley	Ireland	SE	Up to and including GS 59	0.375/spray, max 0.75/crop	>200	1–2	-	-
Barley	Ireland	SE	Up to and including GS 59	0.375/spray, max 0.75/crop	>200	1–2	-	-
Barley	Ireland	EC	Up to and	0.75	>200	1–2	-	-

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
			including GS 59					
Barley	Italy	EC		0.750		1–2		35
Barley	Latvia	SE	BBCH30-61 if 2 1st BBBCH30-37, 2nd BBCH37-51	1@0.23-0.307 2@0.23	200-400	1–2	-	35
Barley	Latvia	SE	BBCH30-69	0.2-0.4	200–400	1–2	21	35
Barley	Latvia	EC		0.375-0.75	100–400	1	-	35
Barley	Latvia	SE	BBCH30-69	0.188-0.375	100–400	1–2	21	35
Barley	Lithuania	SE	BBCH30-69	0.4	200–400	1	-	35
Barley	Lithuania	EC		0.375	200–400	1–2	-	35
Barley	Lithuania	SE		1×0.188-375 or 2×0.188	200–400	1–2		35
Barley	Luxembourg	SE		0.5	200–400	1–2	-	35
Barley	Luxembourg	EC		0.5625-0.75	200-400	1–2	-	28
Barley	Luxembourg	SE	BBCH31 BBCH45	0.375	200–300	1–2	-	-
Barley	Netherlands	EC		0.3975		1		35
Barley	Netherlands	SE		0.25-0.375		1–2	21	35[a]
Barley	New Zealand	SE	Up to end flowering (Zadocs GS 69)	0.375	> 150	1–2	21–35	42[b]
Barley	Norway	EC	Z30-72	0.056-0.075				
Barley	Norway	EC		0.075				
Barley	Poland	SE	BBCH29-55	0.28-0.4		1–2	21	35
Barley	Poland	EC	BBCH30-69	0.75	100–400	1–2	21	35
Barley	Poland	SE	BBCH29-51	0.25	100–400	1-2	21	35[c]
Barley	Poland	SE	End tillering to beginning earing	0.38-0.476	200–400	1	-	35[c]
Barley	Romania	SE	Until ear formation	0.2-0.3	150-300	-	-	-
Barley	Romania	SE	Until emergence of ear	0.250	200–300	1–2	-	35
Barley	Serbia	SE	BBCH30-65	0.250	200–400	1–2	-	42
Barley	Slovakia	SE	BBCH25-61	0.15	200–400	1–2	21	-
Barley	Slovakia	SE		0.2-0.3		1–2		35
Barley	Slovenia	SE		0.25-0.375	200–400	1	-	35
Barley	Sweden	EC	BBCH31-69	0.188-0.375	100–400	1–2	21	35
Barley	Switzerland	SE	BBCH30-61	0.4	200–400	1	-	-
Barley	Switzerland	SE	BBCH31-51	0.375	200 – 400	1–2	-	-
Barley	Turkey	SE		0.3	-	1–2	30	56
Barley	Turkey	SE		0.25		1–2	21-28	35
Barley	UK	SE	Up to and including emergence of ear just complete (GS 59)	0.15	>200	1–2	-	-
Barley	UK	SE	Up to and including emergence of ear just complete (GS 59	0.4	>200	1–2	-	-
Barley	UK	SE		0.562-0.75, max 2.25/winter crop with 1 spray in autumn, max 1.5/spring crop	220-450	1 – 4 winter crops 1-2 spring crops	-	35

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
Barley	UK	SE	Up to and including emergence of ear just complete (GS 59	0.375	>200	1–2	-	-
Barley	UK	SE	Up to and including emergence of ear just complete (GS 59)	0.375	>200	1–2	-	-
Barley	UK	EC	Up to and including emergence of ear just complete (GS 59	0.75	>200	1–2	-	-
Barley	UK	SE	Up to and including emergence of ear just complete (GS 59)	0.375	>200	1–2	-	-
Barley	Ukraine	SE		0.2 – 0.3	200–400	1–2	-	30

✈ application using aircraft

[a] Do NOT harvest grain for human or animal consumption within 42 days of application. Do NOT feed or allow stock to graze treated straw or stubble within 42 days of application. Stock must not feed or be allowed to graze treated green-feed or silage.

[b] Straw of grains treated with fenpropimorph must not be used for animal feed

[c] Period between the last application of the product on plants destined for animal feed and the day when animals can be fed the plants (withdrawal period for feed): cereal grains – 35 days leaves of cereals – 21 days

Table 81 Selected registered uses of fenpropimorph on oats (foliar)

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
Oat	Belgium	EC		0.562 – 0.75	-	1–2	-	28
Oat	Chile	SE	End of tillering or start of stalking to ear newly exposed	0.12–0.15	150 – 200 30 – 50✈	1-2	-	35
Oat	Chile	SE	Start of stalking to ear phase up to 0.429, ear emerged up to 0.321	0.214-0.429	150 – 200 30-50✈	1-2	-	35
Oat	Estonia	SE	Start tillering to start blooming (AS 37-51)	0.23-0.46 max 0.46/season	200-400	1-2		35
Oats	Estonia	SE	AS30-69	0.21-0.42, max 0.46/season	100 – 400	1-2		35
Oat	Estonia	EC		0.375	100–400	1–2	21	35
Oat	Estonia	SE		0.188-0.375	100–400	1–2	21	35
Oat	France	SE		0.375		1		35
Oat	France	SE		0.4		1		35
Oat	Germany	SE	BBCH 32-61	0.15	200–400	1	-	-
Oat	Hungary	SE		0.2–0.3	250 – 350 60-80✈	1–2	-	35
Oat	Hungary	SE		0.2–0.32	250–300	1–2	21	35

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
Oat	Ireland	SE	Before GS59	0.4	>200	1–2	-	-
Oat	Ireland	EC		0.750	220-450	1–2	-	35
Oat	Ireland	SE	Before GS59	0.15	>200	1–2	-	-
Oat	Ireland	SE	Up to and including GS 59	0.375/spray, max 0.75/crop	>200	1–2	-	-
Oat	Ireland	EC	Up to and including GS 59	0.75	>200	1–2	-	-
Oat	Ireland	SE	Up to and including GS 59	0.375/spray, max 0.75/crop	>200	1–2	-	-
Oat	Italy	EC		0.750		1–2		35
Oat	Latvia	SE	BBCH30-61	1×0.23-0.307 2×0.23	200-400	1–2	-	35
Oat	Latvia	SE	BBCH25-59	0.2-0.4	200–400	1–2	21	35
Oat	Latvia	EC		0.375-0.75	100–400	1	-	35
Oat	Latvia	SE	BBCH30-69	0.188-0.375	100–400	1–2	21	35
Oat	Lithuania	SE	BBCH30-69	0.4	200–400	1	-	35
Oat	Lithuania	EC		0.375	200–400	1–2	-	35
Oat	Lithuania	SE		1×0.188-375 or 2×0.188	200–400	1–2		35
Oat	Luxembourg	EC		0.5625-0.75	200-400	1–2	-	28
Oat	Netherlands	EC		0.3975		1		35
Oat	Netherlands	SE		0.375		1–2	21	35[a]
Oat	Norway	EC	Z30-72	0.056-0.075				
Oat	Norway	EC		0.075				
Oat	Slovenia	SE		0.25-0.375	200–400	1	-	35
Oat	Sweden	EC	BBCH31-69	0.188-0.375	100–400	1–2	21	35
Oat	UK	SE	Up to and including emergence of ear just complete (GS 59)	0.15	>200	1–2	-	-
Oat	UK	SE	Up to and including emergence of ear just complete (GS 59	0.4	>200	1–2	-	-
Oat	UK	EC		0.562-0.75, max 2.25/winter crop with 1 spray in autumn, max 1.5/spring crop	220-450	1 – 4 winter crops 1-2 spring crops	-	35
Oat	UK	SE	Up to and including emergence of ear just complete (GS 59)	0.375	>200	1–2	-	-
Oat	UK	SE	Up to and including emergence of ear just complete (GS 59)	0.375	>200	1–2	-	-
Oat	UK	EC	Up to and including emergence of	0.75	>200	1–2	-	-

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
			ear just complete (GS 59					
Oat	UK	SE	Up to and including emergence of ear just complete (GS 59)	0.375	>200	1–2	-	-

[a] Straw of grains treated with fenpropimorph must not be used for animal feed

Table 82 Selected registered uses of fenpropimorph on rye (foliar)

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
Rye	Austria	SE	1st application spring, 2nd up to start flowering BBCH 61	0.375	200–400	1–2	-	-
Rye	Belarus	SE		0.312	200- 400	1	-	30
Rye	Belgium	SE	1st at 1st button (BBCH 31) until last leaf (BBCH 39), second application all ears out (BBCH 59)	0.375	200–400	1-2	28	-
Rye	Belgium	SE	1st last leaf (BBCH 37), to all ears out (BBCH 50)	0.375	200–300	1	-	-
Rye	Belgium	SE	1st at 1st button (BBCH 31) until last leaf (BBCH 39), second application end ear formation (BBCH 58)	0.375	200–400	1–2	-	35
Rye	Belgium	SE	1st button to last leaf with last application at all ears out	0.5	200–400	1–2	-	35
Rye	Chile	SE	End of tillering or start of stalking to ear newly exposed	0.12–0.15	150 – 200 30 – 50✈	1-2	-	35
Rye	Czech Rep.			0.75	100–400	1–2	21	35
Rye	Czech Rep.	SE	Leaf/spikelet disease BBCH 25-61	0.4		1–2		35
Rye	Estonia	SE	Start tillering to mid stem elongation (AS 30-55)	0.23-0.46 max 0.46/season	200-400	1-2		35
Rye	Estonia	SE	AS30-69	0.21-0.42, max 0.46/season	100 – 400	1-2		35
Rye	Estonia	EC		0.375	100–400	1–2	21	35
Rye	Estonia	SE		0.188-0.375	100–400	1–2	21	35
Rye	France	SE		0.375		1		35
Rye	France	SE		0.4		1		35
Rye	Germany	EC	Until beginning	0.750	200–400	1–2	-	-

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
			flowering (BBCH 61)					
Rye	Germany	SE	1st from mid-tillering (BBCH 25) last up to start flowering (BBCH 61)	0.375	200–400	1–2	-	-
Rye	Germany	SE	BBCH 25-61	0.15	200–400	1–2	-	-
Rye	Germany	SE	BBCH 25-61	0.375	200–400	1–2	-	-
Rye	Germany	SE		0.4	200–400	1–2		35
Rye	Hungary	SE		0.2–0.3	250 – 350 60-80✈	1–2	-	35
Rye	Hungary	SE		0.2–0.32	250–300	1–2	21	35
Rye	Ireland	EC		0.750	220-450	1–2	-	35
Rye	Ireland	SE	Before GS65	0.15	>200	1–2	-	-
Rye	Ireland	SE	Up to and including GS 69	0.75	>200	1–2	-	-
Rye	Ireland	SE	Before GS61	0.4	>200	1–2	-	-
Rye	Ireland	SE	Up to and including GS 61	0.375/spray, max 0.75/crop	>200	1–2	-	-
Rye	Italy	EC		0.750		1–2		35
Rye	Latvia	SE	BBCH30-55 if 2 1st BBBCH30-37, 2nd BBCH39-55	1×0.23-0.307 2×0.23	200-400	1–2	-	35
Rye	Latvia	SE	BBCH30-61	0.2-0.4	200–400	1–2	21	35
Rye	Latvia	EC		0.375-0.75	100–400	1	-	35
Rye	Latvia	SE	BBCH30-69	0.188-0.375	100–400	1–2	21	35
Rye	Lithuania	SE	BBCH30-61	1×0.238-478 or 2×0.238	150–400	1–2	21	35
Rye	Lithuania	SE	BBCH30-69	1×0.2-0.4 or 2×0.2	200–400	1	-	35
Rye	Lithuania	EC		0.375	200–400	1–2	-	35
Rye	Lithuania	SE		1×0.188-375 or 2×0.188	200–400	1–2		35
Rye	Luxembourg	SE		0.5	200–400	1–2	-	35
Rye	Luxembourg	SE	BBCH37 BBCH50	0.375	200–300	1–2	-	-
Rye	Netherlands	EC		0.3975		1		35
Rye	Netherlands	SE		0.25-0.375		1–2	21	35[a]
Rye	Norway	SE	Z30-72	0.056-0.075				
Rye	Poland	EC	BBCH30-69	0.75	100–400	1–2	21	35
Rye	Poland	SE	BBCH30-51	0.25	100–400	1-2	21	35[b]
Rye	Poland	SE	Beginning shooting to beginning earing	0.38-0.476	200–400	1	-	35[b]
Rye	Slovakia	SE	BBCH25-61	0.15	200–400	1–2	21	-
Rye	Slovakia	SE		0.2-0.3		1–2		35
Rye	Slovenia	SE		0.25-0.375	200–400	1	-	35
Rye	Sweden	EC	BBCH31-69	0.188-0.375	100–400	1–2	21	35
Rye	Switzerland	SE	BBCH30-61	0.4	200–400	1	-	-
Rye	Switzerland	SE	BBCH31-61	0.188-0.375	200 – 400	1–2	-	-
Rye	UK	SE	Up to and including flowering half way (GS 65)	0.15	>200	1–2	-	-
Rye	UK	SE	Up to and including flowering half way (GS 65)	0.4	>200	1–2	-	-
Rye	UK	EC		0.562-0.75,	220-450	1 – 4	-	35

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
				max 2.25/winter crop with 1 spray in autumn		winter crops		
Rye	UK	EC	Up to and including flowering just complete (GS 69)	0.375	>200	1–2	-	-
Rye	UK	SE	Up to and including flowering just complete (GS 69	0.375	>200	1–2	-	-

[a] Straw of grains treated with fenpropimorph must not be used for animal feed

[b]Period between the last application of the product on plants destined for animal feed and the day when animals can be fed the plants (withdrawal period for feed): cereal grains – 35 days leaves of cereals – 21 days

Table 83 Selected registered uses of fenpropimorph on triticale

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
Triticale	Austria	SE	1st application spring, 2nd up to start flowering BBCH 61	0.375	200–400	1–2	-	-
Triticale	Belarus	SE		0.2-0.3	200–400	1	-	30
Triticale	Belarus	SE		0.25-0.312	200- 400	1	-	30
Triticale	Belgium	EC	1st at 1st button (BBCH 31) until last leaf (BBCH 39), second application end ear formation (BBCH 58)	0.562–0.75	200–400	1–2	-	28
Triticale	Belgium	SE	Between last leaf (BBCH 37) and all ears out (BBCH 50)	0.4	200–400	1	-	35
Triticale	Belgium	SE	1st at 1st button (BBCH 31) until last leaf (BBCH 39), second application all ears out (BBCH 59)	0.375	200–400	1-2	28	-
Triticale	Belgium	SE	1st button to last leaf with last application at all ears out	0.5	200–400	1–2	-	35
Triticale	Belgium	SE	1st last leaf (BBCH 37), to all ears out (BBCH 50)	0.375	200–300	1	-	-
Triticale	Chile	SE	End of tillering or start of stalking to ear newly exposed	0.12–0.15	150 – 200 30 – 50✈	1-2	-	35

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
Triticale	Czech Rep.	SE	Leaf/spikelet disease BBCH 25-61	0.4		1–2		35
Triticale	Estonia	SE	Start tillering to mid stem elongation (AS 30-55)	0.23-0.46 max 0.46/season	200-400	1-2		35
Triticale	Estonia	SE	AS30-69	0.21-0.42, max 0.46/season	100 – 400	1-2		35
Triticale	Estonia	SE		0.188-0.375	100–400	1–2	21	35
Triticale	France	SE		0.4		1		35
Triticale	Germany	SE	1st from mid-tillering (BBCH 25) last up to start flowering (BBCH 61)	0.375	200–400	1–2	-	-
Triticale	Germany	SE	BBCH 25-61	0.15	200–400	1–2	-	-
Triticale	Germany	SE	BBCH 25-61	0.375	200–400	1–2	-	-
Triticale	Germany	SE		0.4	200–400	1–2		35
Triticale	Hungary	SE		0.2–0.32	250–300	1–2	21	35
Triticale	Hungary	SE		0.2–0.3	250 – 350 60-80✈	1–2	-	35
Triticale	Ireland	SE	Up to and including GS 61	0.375/spray, max 0.75/crop	>200	1–2	-	-
Triticale	Ireland	EC		0.750	220-450	1–2	-	35
Triticale	Ireland	SE	Before GS65	0.15	>200	1–2	-	-
Triticale	Ireland	SE	Up to and including GS 69	0.75	>200	1–2	-	-
Triticale	Ireland	SE	Before GS61	0.4	>200	1–2	-	-
Triticale	Latvia	SE	BBCH30-55 if 2 1st BBBCH30-37, 2nd BBCH39-55	1×0.23-0.307 2×0.23	200-400	1–2	-	35
Triticale	Latvia	SE	BBCH30-69	0.2-0.4	200–400	1–2	21	35
Triticale	Latvia	EC		0.375-0.75	100–400	1	-	35
Triticale	Latvia	SE	BBCH30-69	0.188-0.375	100–400	1–2	21	35
Triticale	Lithuania	SE	BBCH30-61	1×0.238-478 or 2×0.238	150–400	1–2	21	35
Triticale	Lithuania	SE	BBCH30-69	0.4	200–400	1	-	35
Triticale	Lithuania	EC		0.375	200–400	1–2	-	35
Triticale	Lithuania	SE		1×0.188-375 or 2×0.188	200–400	1–2		35
Triticale	Luxembourg	SE		0.5	200–400	1–2	-	35
Triticale	Luxembourg	EC		0.5625-0.75	200-400	1–2	-	28
Triticale	Luxembourg	SE	BBCH37 BBCH50	0.375	200–300	1–2	-	-
Triticale	Netherlands	EC		0.3975		1		35
Triticale	Netherlands	SE		0.25-0.375		1–2	21	35[a]
Triticale	Norway	SE	Z30-72	0.056-0.075				
Triticale	Poland	SE	BBCH30-59	0.28-0.4		1–2	21	35
Triticale	Poland	EC	BBCH30-69	0.75	100–400	1–2	21	35
Triticale	Poland	SE	BBCH30-59	0.25	100–400	1-2	21	35[b]
Triticale	Poland	SE	Beginning shooting to beginning earing	0.38-0.476	200–400	1	-	35[b]
Triticale	Slovakia	SE	BBCH25-61	0.15	150–400	1	-	-

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
Triticale	Slovakia	SE		0.2-0.3		1–2		35
Triticale	Slovenia	SE		0.25-0.375	200–400	1	-	35
Triticale	Sweden	EC	BBCH31-69	0.188-0.375	100–400	1–2	21	35
Triticale	Switzerland	SE	BBCH30-61	0.4	200–400	1	-	-
Triticale	UK	SE	Up to and including flowering half way (GS 65)	0.15	>200	1–2	-	-
Triticale	UK	SE	Up to and including flowering half way (GS 65)	0.4	>200	1–2	-	-
Triticale	UK	SE		0.562-0.75, max 2.25/winter crop with 1 spray in autumn	220-450	1 – 4 winter crops	-	35
Triticale	UK	SE	Up to and including flowering just complete (GS 69)	0.375	>200	1–2	-	-
Triticale	UK	SE	Up to and including flowering just complete (GS 69	0.375	>200	1–2	-	-

[a] Straw of grains treated with fenpropimorph must not be used for animal feed

[b] Period between the last application of the product on plants destined for animal feed and the day when animals can be fed the plants (withdrawal period for feed): cereal grains – 35 days, leaves of cereals – 21 days

Table 84 Selected registered uses of fenpropimorph on wheat

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
Wheat	Austria	SE	1st application spring, 2nd up to start flowering BBCH 61	0.375	200–400	1–2	-	-
Wheat	Belarus	SE		0.2-0.3	200–400	1	-	30
Wheat	Belarus	SE		0.25-0.312	200- 400	1	-	30
Wheat	Belgium	EC	1st at 1st button (BBCH 31) until last leaf (BBCH 39), second application end ear formation (BBCH 58)	0.562–0.75	200–400	1–2	-	28
Wheat	Belgium	SE	1st at 1st button (BBCH 31) until last leaf (BBCH 39), second application end ear formation (BBCH 58)	0.4	200–400	1–2	-	35
Wheat	Belgium	SE	1st at 1st button (BBCH 31) until last leaf (BBCH 39), second application all ears out (BBCH	0.375	200–400	1-2	28	-

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
			59)					
Wheat	Belgium	SE	1st at 1st button (BBCH 31) until last leaf (BBCH 37), second application all ears out (BBCH 59)	0.375	200–300	1-2	-	-
Wheat	Belgium	SE	1st button to last leaf with last application at all ears out	0.5	200–400	1–2	-	35
Wheat	Bulgaria	SE	Tillering (BBCH 25) to end of flowering (BBCH 69)	0.2 – 0.25	200 – 400 50-70✈	1–2	21	50
Wheat	Bulgaria	SE	During tillering to beginning of flowering	0.2	200–490	1–2	21	35
Wheat	Brazil	EC		0.56 – 0.75	200–300	1-2	-	35
Wheat	Chile	SE	End of tillering or start of stalking to ear newly exposed	0.12–0.15	150 – 200 30 – 50✈	1-2	-	35
Wheat	Chile	SE	Start of stalking to ear phase up to 0.429, ear emerged up to 0.321	0.214-0.429	150 – 200 30-50✈	1-2	-	35
Wheat	Croatia	SE		0.250-0.300	200-600	1-2		42
Wheat	Czech Rep.			0.75	100–400	1–2	21	35
Wheat	Czech Rep.	SE	8 Feekes to early flowering	0.25	200–400	1–2	21	35
Wheat	Czech Rep.	SE		0.15		1 -2		35
Wheat	Czech Rep.	SE	Leaf/spikelet disease BBCH 25-61 Wheat stem break BBCH 29-32	0.28-0.4		1–2		35
Wheat	Estonia	SE	Start tillering to start blooming (AS30-55)	0.23-0.46, max 0.46/season	100 – 400	1-2	21	35
Wheat	Estonia	SE	Start tillering to 1st flag, ear elongation (AS30-37) to end blooming (AS49-61)	0.21-0.42, max 0.46/season	100 – 400	1-2		35
Wheat	Estonia	EC		0.375	100–400	1–2	21	35
Wheat	Estonia	SE		0.188-0.375	100–400	1–2	21	35
Wheat	France	SE		0.375		1		35
Wheat	France	SE		0.4		1		35
Wheat	Germany	EC	Until beginning flowering (BBCH 61)	0.750	200–400	1–2	-	-
Wheat	Germany	SE	1st from mid-tillering (BBCH 25) last up to start flowering (BBCH 61)	0.375	200–400	1–2	-	-
Wheat	Germany	SE	BBCH 25-61	0.15	200–400	1–2	-	-
Wheat	Germany	SE	BBCH 25-61	0.375	200–400	1–2	-	-

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
Wheat	Germany	SE		0.4	200–400	1–2		35
Wheat	Greece	EC		0.750	400–600	1–2	-	35
Wheat	Hungary	SE		0.2–0.3	250 – 350 60-80✈	1–2	-	35
Wheat	Hungary	SE	1st during tillering, 2nd before blooming	0.38 – 0.476	50 – 70✈	1–2	-	35
Wheat	Hungary	SE		0.2–0.32	250–300	1–2	21	35
Wheat	Ireland	SE	Before GS61	0.4	>200	1–2	-	-
Wheat	Ireland	EC		0.750	220-450	1–2	-	35
Wheat	Ireland	SE	Before GS65	0.15	>200	1–2	-	-
Wheat	Ireland	SE	Up to and including GS 69	0.375/spray, max 0.75/crop	>200	1–2	-	-
Wheat	Ireland	SE	Up to and including GS 69	0.75	>200	1–2	-	-
Wheat	Ireland	SE	Up to and including GS 61	0.375/spray, max 0.75/crop	>200	1–2	-	-
Wheat	Italy	EC		0.750		1–2		35
Wheat	Latvia	SE	BBCH30-55 if 2 1st BBBCH30-37, 2nd BBCH39-55	1×0.23-0.307 2×0.23	200-400	1–2	-	35
Wheat	Latvia	SE	BBCH30-37 BBCH49-69	0.2-0.4	200–400	1–2	21	35
Wheat	Latvia	EC		0.375-0.75	100–400	1	-	35
Wheat	Latvia	SE	BBCH30-69	0.188-0.375	100–400	1–2	21	35
Wheat	Lithuania	SE	BBCH30-61	1×0.238-478 or 2×0.238	150–400	1–2	21	35
Wheat	Lithuania	SE	BBCH30-69	1×0.2-0.4 or 2×0.2	200–400	1	-	35
Wheat	Lithuania	EC		0.375	200–400	1–2	-	35
Wheat	Lithuania	SE		1×0.188-375 or 2×0.188	200–400	1–2		35
Wheat	Luxembourg	SE		0.5	200–400	1–2	-	35
Wheat	Luxembourg	EC		0.5625-0.75	200-400	1–2	-	28
Wheat	Luxembourg	SE	BBCH31-37 BBCH59	0.375	200–300	1–2	-	-
Wheat	Morocco	SE	Up to ear emergence	0.24				
Wheat	Netherlands	EC		0.3975		1		35
Wheat	Netherlands	SE		0.25-0.375		1–2	21	35[a]
Wheat	New Zealand	SE	Up to end flowering (Zadocs GS 69)	0.375	> 150	1–2	21–35	42[b]
Wheat	Norway	SE	Z30-72	0.056-0.075				
Wheat	Norway	EC		0.075				
Wheat	Poland	SE	BBCH30-59	0.28-0.4		1–2	21	35
Wheat	Poland	EC	BBCH30-69	0.75	100–400	1–2	21	35
Wheat	Poland	SE	BBCH30-59	0.25	100–400	1-2	21	35[c]
Wheat	Poland	SE	Beginning shooting to beginning earing	0.38-0.476	200–400	1	-	35[c]
Wheat	Romania	SE	Until end blooming	0.2-0.3	150-300	-	-	-
Wheat	Romania	SE	Until emergence of ear	0.250	200–300	1–2	-	35
Wheat	Serbia	SE	BBCH30-65	0.250	200–400	1–2	-	42
Wheat	Slovakia	SE	BBCH25-61	0.15	200–400	1–2	21	-
Wheat	Slovakia	SE		0.2-0.3		1–2		35
Wheat	Slovakia	SE	BBCH32-59	0.25	200–400	1–2	21	35

Crop	Country	Form	GS	Rate (kg ai/ha)	Water (L/ha)	No.	Interval (days)	PHI (days)
Wheat	Slovenia	SE	Last at beginning flowering	0.25-0.375	200–400	1-2	na	35
Wheat	Sweden	EC	BBCH31-69	0.188-0.375	100–400	1–2	21	35
Wheat	Switzerland	SE	BBCH30-61	0.4	200–400	1	-	-
Wheat	Switzerland	SE	BBCH37-61	0.375	200 – 400	1–2	-	-
Wheat	Tunisia	SE		0.3	200–400	1–2	-	-
Wheat	Turkey	SE		0.3	-	1–2	30	56
Wheat	Turkey	SE		0.25-0.313		1–2	21-28	35
Wheat	UK	SE	Up to and including flowering half way (GS 65)	0.15	>200	1–2	-	-
Wheat	UK	SE	Up to and including flowering half way (GS 65)	0.4	>200	1–2	-	-
Wheat	UK	EC		0.562-0.75, max 2.25/winter crop with 1 spray in autumn, max 1.5/spring crop	220-450	1 – 4 winter crops 1-2 spring crops	-	35
Wheat	UK	EC	Up to and including flowering just complete (GS 69)	0.375	>200	1–2	-	-
Wheat	UK	SE	Up to and including flowering just complete (GS 69)	0.375	>200	1–2	-	-
Wheat	UK	SE	Up to and including flowering just complete (GS 69	0.75	>200	1–2	-	-
Wheat	UK	SE	Up to and including flowering just complete (GS 69	0.375	>200	1–2	-	-
Wheat	Ukraine	SE		0.2 – 0.3	200–400	1–2	-	30

✈Aerial application

[a] Do NOT harvest grain for human or animal consumption within 42 days of application. Do NOT feed or allow stock to graze treated straw or stubble within 42 days of application. Stock must not feed or be allowed to graze treated green-feed or silage.

[b] Straw of grains treated with fenpropimorph must not be used for animal feed

[c] Period between the last application of the product on plants destined for animal feed and the day when animals can be fed the plants (withdrawal period for feed): cereal grains – 35 days, leaves of cereals – 21 days

RESULTS OF RESIDUE TRIALS ON CROPS

The Meeting received information on supervised field trials for fenpropimorph on the following crops or crop groups:

Crop	Table No.
Banana	85
Sugar beet	86-87
Barley	88-89
Oats	90
Rye	91
Wheat	92-93

Trials were generally well documented with laboratory and field reports. Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Control samples are indicated in the summary tables with a "c". Unless stated otherwise, residue data are recorded unadjusted for recovery.

Residues and application rates have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. Those results included in the evaluation are underlined.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Trial designs used non-replicated plots. Field reports provided data on the sprayers used, plot size, field sample size and sampling date.

Where duplicate field samples from an un-replicated plot were taken at each sampling time and were analysed separately, the mean of the two analytical results was taken as the best estimate of the residues in the plot and only the means are recorded in the tables. Similarly, where samples were collected from replicate plots the mean result is reported (see general consideration JMPR 2010).

Houbraken *et al.* (2015) studied the effect of different adjuvants on the volatilisation of fenpropimorph thus giving some insight into the effect of formulation type, adjuvants and spray tank additives on potential residues. Without adjuvants, volatilisation of pure fenpropimorph was 90.3%, with methylated seed oils 53.1%, polymeric surfactant 72.8%, ethoxylated alcohol 59.9% and anionic surfactant 30.5%. Volatilisation of the commercial EC formulation was 87.1%, i.e. not very different to pure fenpropimorph. While adjuvants may also modify the rate of uptake when applied to leaf surfaces, the data do suggest addition of additives to spray tank mixtures might influence observed residues in trials.

Banana

Table 85 Residues of fenpropimorph in banana (Wofford and Artz 1997a/5004, Tilting and Mackenroth 1995/10747, Bissetti Costa 2012a 2012/3005401, Blaschke 1995a 1995/10739)

Location, year, variety BANANA	N (int)	Rate g ai/ha	Vol L/ha	GS application	DALA	Sample	Residues (mg/kg)
Pueblo Nuevo, Costa Rica, 1996, Gran Enano OL last 20/5	4 (13 43 12)	439 621 493 552	16 19 18 20	Raceme form Raceme dev Raceme dev Mature mats	0 0 5 5	whole fruit pulp whole fruit pulp	<u>1.20</u> 0.30 0.72 0.17
					0 0 5 5	whole fruit bgd pulp bgd whole fruit bgd pulp bgd	0.13 0.081 0.16 < 0.05

Location, year, variety BANANA	N (int)	Rate g ai/ha	Vol L/ha	GS application	DALA	Sample	Residues (mg/kg)
Pueblo Nuevo, Costa Rica, 1996 Gran Enano OL last 21/5	4 (12 42 13)	608 574 568 520✈	22 21 21 19	Raceme form Raceme dev Raceme dev Mature mats	0 0 5 5	whole fruit pulp whole fruit pulp	0.11 < 0.05 0.09 < 0.05
					0 0 5 5	whole fruit bgd pulp bgd whole fruit bgd pulp bgd	< 0.05 < 0.05 < 0.05 < 0.05
Santa Maria, Costa Rica, 1996 Gran Enano OL last 17/6	4 (13 44 12)	612 565 505 552	22 21 18 20	Raceme form Raceme dev Raceme dev Mature mats	0 0 5 5 10 10 15 15 25 25	whole fruit pulp whole fruit pulp whole fruit pulp whole fruit pulp whole fruit pulp	0.75 0.22 0.38 0.18 0.50 0.29 0.32 0.13 < 0.05 < 0.05
					0 0 5 5 10 10 15 15 25 25	whole fruit bgd pulp bgd whole fruit bgd pulp bgd whole fruit bgd pulp bgd whole fruit bgd pulp bgd whole fruit bgd pulp bgd	0.37 0.20 0.38 0.19 0.40 0.19 0.31 0.11 < 0.05 < 0.05
Santa Maria, Costa Rica, 1996 Gran Enano OL last 4/6	4 (12 44 12)	545 517 539 556	20 19 20 20	Raceme form Raceme dev Raceme dev Mature mats	0 0 5 5	whole fruit pulp whole fruit pulp	1.40 0.29 0.66 0.43
					0 0 5 5	whole fruit bgd pulp bgd whole fruit bgd pulp bgd	0.13 < 0.05 0.33 0.074
Boliche, Ecuador, 1996 Cavendish OL last 8/5	4 (12 45 12)	527 567 620 558	20 21 23 20	Raceme form Raceme dev Raceme dev Mature mats	0 0 5 5	whole fruit pulp whole fruit pulp	0.26 0.058 0.20 0.06
					0 0 5 5	whole fruit bgd pulp bgd whole fruit bgd pulp bgd	< 0.05 < 0.05 < 0.05 < 0.05
Boliche, Ecuador, 1996 Cavendish OL last 10/5	4 (11 46 10)	524 545 545 599✈	20 20 20 22	Flowering Flowering Flowering Export grade	0 0 5 5	whole fruit pulp whole fruit pulp	< 0.05 < 0.05 < 0.05 < 0.05
					0 0 5 5	whole fruit bgd pulp bgd whole fruit bgd pulp bgd	< 0.05 < 0.05 < 0.05 < 0.05
Milagros, Ecuador, 1996 Cavendish OL last 11/5	4 (12 45 13)	493 556 636 557	19 20 23 20	Raceme form Raceme dev Raceme dev Mature mats	0 0 5 5	whole fruit pulp whole fruit pulp	0.21 < 0.05 0.36 < 0.05
					0 0 5 5	whole fruit bgd pulp bgd whole fruit bgd pulp bgd	< 0.05 < 0.05 < 0.05 < 0.05
Marcelino Maridueño, Ecuador, 1996 Cavendish OL last 12/5	4 (12 43 12)	514 526 604 519	19 19 22 19	Raceme form Raceme dev Raceme dev Mature mats	0 0 5 5	whole fruit pulp whole fruit pulp	0.10 < 0.05 0.06 < 0.05

Location, year, variety BANANA	N (int)	Rate g ai/ha	Vol L/ha	GS application	DALA	Sample	Residues (mg/kg)
					0	whole fruit bgd	0.17
					0	pulp bgd	0.07
					5	whole fruit bgd	0.17
					5	pulp bgd	0.06
Cienaga, Columbia, 1996 Cavendish OL last 20/6	4 (12 44 12)	518 518 540 512	19 17 20 19	Raceme form Raceme dev Raceme dev Mature mats	0	whole fruit	0.16
					0	pulp	< 0.05
					5	whole fruit	0.08
					5	pulp	< 0.05
					10	whole fruit	0.09
					10	pulp	< 0.05
					15	whole fruit	0.11
					15	pulp	< 0.05
					25	whole fruit	< 0.05
					25	pulp	< 0.05
					0	whole fruit bgd	< 0.05
					0	pulp bgd	< 0.05
					5	whole fruit bgd	< 0.05
					5	pulp bgd	< 0.05
					10	whole fruit bgd	< 0.05
					10	pulp bgd	< 0.05
					15	whole fruit bgd	0.13
					15	pulp bgd	< 0.05
					25	whole fruit bgd	< 0.05
					25	pulp bgd	< 0.05
Cienaga, Colombia, 1996 Cavendish OL last 5/6	4 (12 44 12)	554 566 577 537✈	20 21 21 20	Raceme form Raceme dev Raceme dev Mature mats	0	whole fruit	< 0.05
					0	pulp	< 0.05
					5	whole fruit	< 0.05
					5	pulp	< 0.05
					0	whole fruit bgd	< 0.05
					0	pulp bgd	< 0.05
					5	whole fruit bgd	< 0.05
					5	pulp bgd	< 0.05
La Aguja, Columbia, 1996 Cavendish OL last 22/6	4 (12 44 12)	562 517 569 527	20 19 21 19	Raceme form Raceme dev Raceme dev Mature mats	0	whole fruit	0.12
					0	pulp	< 0.05
					5	whole fruit	0.07
					5	pulp	< 0.05
					0	whole fruit bgd	< 0.05
					0	pulp bgd	< 0.05
					5	whole fruit bgd	< 0.05
					5	pulp bgd	< 0.05
Batan, Honduras, 1996 Cavendish OL last 29/3	4 (13 45 12)	533 499 502 538	20 18 18 20	Raceme form Raceme dev Raceme dev Mature mats	0	whole fruit	0.47
					0	pulp	0.19
					5	whole fruit	0.65
					5	pulp	0.28
					0	whole fruit bgd	< 0.05
					0	pulp bgd	< 0.05
					5	whole fruit bgd	< 0.05
					5	pulp bgd	< 0.05
Cortes, Honduras, 1996 Cavendish OL last 31/3	4(12 43 13)	545 524 535 482	20 19 20 18	Raceme form Raceme dev Raceme dev Mature mats	0	whole fruit	0.30
					0	pulp	0.14
					5	whole fruit	0.43
					5	pulp	0.12
					0	whole fruit bgd	< 0.05
					0	pulp bgd	< 0.05
					5	whole fruit bgd	< 0.05
					5	pulp bgd	< 0.05
San Marcos, Guatemala, 1996 Grand Naine OL last 15/4	4 (12 44 11)	622 534 574 566	45 20 21 21	Raceme form Raceme dev Raceme dev Mature mats	0	whole fruit	0.70
					0	pulp	0.18
					5	whole fruit	0.45
					5	pulp	0.12
					0	whole fruit bgd	< 0.05
					0	pulp bgd	< 0.05
					5	whole fruit bgd	< 0.05
					5	pulp bgd	< 0.05

Location, year, variety BANANA	N (int)	Rate g ai/ha	Vol L/ha	GS application	DALA	Sample	Residues (mg/kg)
Chiapas, Mexico, 1996 Enana Gigante OL last 19/5	4 (12 44 11)	578 573 523 494	21 21 19 18	Raceme form Raceme dev Raceme dev Mature mats	0 0 5 5	whole fruit pulp whole fruit pulp	0.32 0.07 0.18 0.08
					0 0 5 5	whole fruit bgd pulp bgd whole fruit bgd pulp bgd	< 0.05 < 0.05 < 0.05 < 0.05
Larentry, Lamentin, Martinique, 1995, Eloise No.1 (dry season) Grande Naine EC	4 (28 28 27)	525 525 525 525	20 20 20 20	Developing fruit	0 3 7 14	Whole fruit Whole fruit Whole fruit Whole fruit	< 0.05 < 0.05 < 0.05 < 0.05
Petit Morne, Lamentin, Martinique, 1995, Barriére No.2 (dry season) Grande Naine EC	4 (28 28 27)	525 525 525 525	20 20 20 20	Developing fruit	0 3 7 14	Whole fruit Whole fruit Whole fruit Whole fruit	0.07 < 0.05 < 0.05 < 0.05
Riviere Lézarde, St. Joseph, Martinique, 1995, Chemin de Fer No.3 (dry season) Grande Naine EC	4 (28 28 27)	525 525 525 525	20 20 20 20	Developing fruit	0 3 7 14	Whole fruit Whole fruit Whole fruit Whole fruit	< 0.05 < 0.05 < 0.05 < 0.05
Riviere Lézarde, St. Joseph, Martinique, 1995, Savane Bas No.4 (dry season) Grande Naine EC	4 (28 28 27)	525 525 525 525	20 20 20 20	Developing fruit	0 3 7 14	Whole fruit Whole fruit Whole fruit Whole fruit	< 0.05 < 0.05 < 0.05 < 0.05
Riviere Lézarde, St. Joseph, Martinique, 1995, Morne Vent No.5 (wet season) Grande Naine EC	4 (28 28 28)	525 525 525 525	20 20 20 20	Developing fruit	0 3 7 14	Whole fruit Whole fruit Whole fruit Whole fruit	< 0.05 < 0.05 < 0.05 < 0.05
Riviere Lézarde, St. Joseph, Martinique, 1995, Laurencine No.6 (wet season) Grande Naine EC	4 (28 28 28)	525 525 525 525	20 20 20 20	Developing fruit	0 3 7 14	Whole fruit Whole fruit Whole fruit Whole fruit	0.13 < 0.05 < 0.05 < 0.05
Bochet Lamentin, Martinique, 1995, Parc á Mullet No.7 (wet season) Grande Naine EC	4 (28 28 28)	525 525 525 525	20 20 20 20	Developing fruit	0 3 7 14	Whole fruit Whole fruit Whole fruit Whole fruit	< 0.05 < 0.05 < 0.05 < 0.05
Bochet Lamentin, Martinique, 1995, Boise de l'Homme No.8 (wet season) Grande Naine EC	4 (28 28 28)	525 525 525 525	20 20 20 20	Developing fruit	0 3 7 14	Whole fruit Whole fruit Whole fruit Whole fruit	< 0.05 < 0.05 < 0.05 < 0.05
Registro (SP) Brazil, 2011 Nanicão EC + Agral S 24°33'24.07" W47°54'04.18"	6 (14 14 14 14 14)	750 750 750 750 750 750	25 25 25 25 25 25	61 62 63 64 65 67	1	Whole fruit	0.37
Guaramirim (SC) Brazil, 2011 Prata Mineira EC + Agral S 26°29'05.03" W48°58'29.89"	6 (14 14 14 14 14)	750 750 750 750 750 750	25 25 25 25 25 25	61 62 63 64 65 67	1	Whole fruit	0.10
Iracemápolis (SP) Brazil, 2011 Nanicão EC + Agral S 22°36'39.0" W47°30'12.02"	6 (14 14 14 14 14)	750 750 750 750 750 750	25 25 25 25 25 25	66 66 67 68 69 70	0 1 7	Whole fruit Whole fruit Whole fruit	0.47 0.80 0.24

Location, year, variety BANANA	N (int)	Rate g ai/ha	Vol L/ha	GS application	DALA	Sample	Residues (mg/kg)
Limeira (SP) Brazil, 2011 Prata Mineira EC + Agral S 22°31'21.09" W47°17'43.0"	6 (14 14 14 14 14)	750 750 750 750 750 750	25 25 25 25 25 25	65 66 67 68 69 70	0 1 7	Whole fruit Whole fruit Whole fruit	0.7 0.31 0.24
Teapa, Tabasco, Mexico 1992 Grand Nain EC	5 (29 27 15 14)	453 447 458 481 462	24 24 25 26 25	7 DAS 36 DAS 63 DAS 77 DAS 92 DAS	0 0 0 0 5 5 5 5 5	Pulp bgd Pulp Peel bgd Peel Pulp bgd Pulp Peel bgd Peel Peel bgd	< 0.05 < 0.05 < 0.05 0.13 < 0.05 < 0.05 < 0.05 0.10 < 0.05
	5 (29 27 15 14)	905 894 917 961 920	24 24 25 26 25	7 DAS 36 DAS 63 DAS 77 DAS 92 DAS	0 0 0 0 5 5 5 5	Pulp bgd Pulp Peel bgd Peel Pulp bgd Pulp Peel bgd Peel	< 0.05 < 0.05 < 0.05 < 0.05 0.06 0.21 < 0.05 0.23

DAS = days after shooting

Sugar beet

Table 86 Residues of fenpropimorph in sugar beet (Linder 2014a 2013/1396627, Oxspring 2009a 2009/1012810, Schulz H., 2009 a 2009/1075091, Perny 2004 1024749, Lantos 2000 1013434)

Location, year, variety SUGAR BEET	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Wunstorf, Lower Saxony Germany 2007 Modus EC	1	0.75	200	49	0 22 29 35 42	roots roots roots roots roots	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01
Grantham, Leicestershire UK 2007 Octa EC	1	0.75	200	45-48	0 21 31 35 42	roots roots roots roots roots	0.088 0.011 0.03 0.059 0.06
Mauchenheim Germany 2008 Tiziana EC	1	0.78	208	39	0 21 28 36 41	roots roots roots roots roots	0.02 < 0.01 < 0.01 < 0.01 < 0.01
Loivre France (north) 2008 Julietta EC	1	0.74	198	39	0 20 29 35 41	roots roots roots roots roots	0.02 < 0.01 < 0.01 < 0.01 < 0.01
Mauchenheim Germany 2013 Kleist EC	1	0.75	200	39	0 22 28 36	roots roots roots roots	< 0.01 < 0.01 < 0.01 < 0.01
Witry Les Reims France (north) 2013 Vienna EC	1	0.72	200	39	0 20 29 35	roots roots roots roots	0.048 < 0.01 < 0.01 < 0.01

Location, year, variety SUGAR BEET	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Ottersum Netherlands 2013 Isabella EC	1	0.80	200	39	0 21 28 36	roots roots roots roots	< 0.01 < 0.01 ≤ 0.01 < 0.01
Lawford Essex UK 2013 Pasteur EC	1	0.80	200	47	0 21 27 35	roots roots roots roots	0.017 < 0.01 ≤ 0.01 < 0.01
Finhan France (south) 2007 Python EC	1	0.75	200	47	0 3 7 14	roots roots roots roots	0.07 < 0.01 < 0.01 < 0.01
Via Olmo, Bologna Italy 2007 Zanzibar EC	1	0.75	200	47	0 3 8 14	roots roots roots roots	< 0.01 0.025 < 0.01[A] 0.027
Nerja Spain 2008 Diamante EC	1	0.74	197	49	0 3 7 14	roots roots roots roots	0.13 0.01 0.03 0.01
Pella, North Macedonia, Greece 2008 Areti EC	1	0.74	196	47	0 3 7 14	roots roots roots roots	0.03 < 0.01 < 0.01 < 0.01
Szabolcs-Szatmár- Bereg County, Nagyhalász Hungary 1999 Marika SE	2 (14)	0.438 0.438	300 300	End of vegetation	35	Root	< 0.025 0.04 < 0.025
Salteras Sevilla Spain 2003 Lucia SE	2 (14)	0.25 0.25	300 300	37 39	0 28 42	Roots Roots Roots	< 0.05 < 0.05 < 0.05
Guillena Sevilla Spain 2003 Napoli SE	2 (14)	0.25 0.25	300 300	39 39	0 28 43	Roots Roots Roots	< 0.05 < 0.05 < 0.05
Nisi Alexandria Greece 2003 23-39 Rizor SE	2 (15)	0.25 0.25	300 300	38 38	0 27 41	Roots Roots Roots	< 0.05 < 0.05 < 0.05
Drepano Kozani Greece 2003 Asso SE	2 (14)	0.25 0.25	300 300	38 38	0 28 42	Roots Roots Roots	< 0.05 < 0.05 < 0.05

[A] Likely that the treated and untreated specimens were mislabelled during the study since corresponding untreated specimens gave residue levels of 1.53 mg/kg and 0.020 mg/kg for 'leaves with tops' and 'roots', respectively

Table 87 Supporting data on residues of fenpropimorph in sugar beet from trial reports available only in summary form

Location, year, variety SUGAR BEET	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Stättahra Halland Sweden 1985 Primahill① EC	3	0.75 0.75 0.75	250 250 250		65	Roots	< 0.05
Villers la Ville Belgium 1985② EC	1	0.75	600		46	roots	< 0.02 < 0.02 < 0.02
Capalbio Italy 1991 Buramo ③ EC	1	0.75	400		0 20 30	roots	< 0.05 < 0.05 < 0.05
Auberive, France 1983 Monosvalof ④ EC	1	0.75		maturity	72	Roots	< 0.05
Beaumont sur Vesle France 1983 Régina ⑤ EC	1	0.75		Maturity	82	roots	< 0.05

Location, year, variety SUGAR BEET	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Jodoigne Belgium 1985⑥ EC	1	0.75	600		72	Roots	< 0.02 0.04 < 0.02
Ambresin Belgium 1985⑦ EC	1	0.75	600		62	Roots	< 0.02 < 0.02
Assevillers France 1983 Monostar⑧ EC	2 (14)	0.75 0.75			68	Roots	< 0.05
Bressana Boharone Italy 1987 Dima⑨ EC	3 (22 19)	0.75 0.75 0.75	400 400 400	49	20	Roots	< 0.05
Cervesina Italy 1991 Perla⑩ EC	3 (14 15)	0.75 0.75 0.75	500 500 500	Crop cover complete	0 20 32	Roots Roots Roots	< 0.05 < 0.05 < 0.05
Abinia Italy 1989 Monova❶ EC	2 (15)	0.56 0.56	500 500		3	Roots	< 0.05
Marsigliana Italy 1989 Maribo Unica❷ EC	2 (13)	0.56 0.56	500 500	maturity	5	Roots	< 0.05
Due Pavoni Italy 1989 Maribo Monova❸ EC	2 (17)	0.56 0.56	500 500	49	16 31	Roots Roots	< 0.05 < 0.05
Corana Italy 1989 Novaghemo❹ EC	3 (12 10)	0.56 0.56 0.56	400 400 400	49	1 7 13 22	Roots Roots Roots Roots	< 0.05 < 0.05 < 0.05 < 0.05
Ceruesina Italy 1988 Rizo❺ EC	3 (16 18)	0.56 0.56 0.56	500 500 500	49	2 8 15 30	Roots Roots Roots Roots	< 0.05 < 0.05 < 0.05 < 0.05

① 1985 10359, ② 1986 10161, ③ 1991 11786, ④ 1983 10351, ⑤ 1983 10353, ⑥ 1986 10160, ⑦ 1986 10162, ⑧ 1983 10352, ⑨ 1987 10463, ⑩1991 11785, ❶1989 10504, ❷1989 10505, ❸1989 10768, ❹1989 10770, ❺1988 10823

Cereal grains

Table 88 Residues of fenpropimorph in barley (2001 1009068, 2001 1009081, 2004 1010542, 2005 7004267, 2007 1050094, 2012 3003426)

Location, year, variety BARLEY	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Puerto S. María, Cádiz, Andalucia Spain 2000 Almudena 23/3 18/4 EC	2 (26)	0.76 0.72	305 290	47-49 75	35 42	Grain Grain	< 0.05 < 0.05
Algodonales, Cádiz, Andalucia Spain 2000 Irene 17/4 28/4 EC	2 (11)	0.75 0.75	302 301	47-49 67	35 42	Grain Grain	< 0.05 < 0.05
Settala, Milano, Lombardia Italy 2000 Baraka 21/4 23/5 EC	2 (32)	0.74 0.73	297 296	47 69	35 42	Grain Grain	< 0.05 0.07
Liscate, Milano, Lombardia Italy 2000 Astrix 26/4 17/5 EC	2 (21)	0.77 0.76	309 305	49 65-67	35 42	Grain Grain	< 0.05 0.07
Lund Sweden 2000 Barke 5/6 29/6 EC	2 (24)	0.72 0.76	294 310	37 59	42 49	Grain Grain	< 0.05 < 0.05
Salmonby UK 2000 Pearl 13/5 30/5 EC	2 (17)	0.77 0.76	312 306	39 61	56 64	Grain Grain	< 0.05 < 0.05
Taissy France (north) 2000 Esterel 9/5 26/5 EC	2 (17)	0.74 0.74	301 302	39 59	34 42	Grain Grain	< 0.05 < 0.05
Brandenburg Germany 2003 Candesse 26/5 EC	1	0.4	300	69	35 42	Grain Grain	0.02 0.02
	1	0.75	300	69	35 42	Grain Grain	0.04 0.02
Seebach Alsace France 2003 Astoria 12/6 EC	1	0.4	300	73	36 42	Grain Grain	0.02 0.02
	1	0.75	300	73	36 42	Grain Grain	0.04 0.05

Location, year, variety BARLEY	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Rhône-Aples France 2003 Orelie 15/5 EC	1	0.4	300	83	35 42	Grain Grain	0.02 0.01
	1	0.75	300	83	35 42	Grain Grain	0.03 0.03
Piemonte Italy 2003 Prosa 26/5 EC	1	0.4	300	55	35 42	Grain Grain	< 0.01 0.01
	1	0.75	300	55	35 42	Grain Grain	0.02 0.01
Rheinland-Pfalz Germany 2005 Scarlett 25/5 9/6 EC	2 (15)	0.4 0.4	200 200	69	29 36 42	Grain Grain Grain	< 0.01 0.01 < 0.01
	2 (15)	0.75 0.75	200 200	69	29 36 42	Grain Grain Grain	< 0.01 < 0.01 0.02
Rhône-Alpes France 2005 Orelie 26/4 18/5 EC	2 (22)	0.4 0.4	200 200	69	35 41	Grain Grain	0.01 0.01
	2 (22)	0.75 0.75	200 200	69	35 41	Grain Grain	0.01 0.02
Malmoe Sweden 2005 Prestige 2/6 14/7 EC	2 (42)	0.4 0.4	200 200	69	36 43	Grain Grain	0.06 0.05
	2 (42)	0.75 0.75	200 200	69	36 43	Grain Grain	0.09 0.06
Oxfordshire UK 2005 Pearl 5/5 20/6 EC	2 (46)	0.4 0.4	200 200	73	28 35 42	Grain Grain Grain	0.03 0.03 0.03
	2 (46)	0.75 0.75	200 200	73	28 35 42	Grain Grain Grain	0.08 0.09 0.11
Duras Aquitaine France 2007 Nikel 5/4 10/5 EC	2 (35)	0.75 0.75	200 200	69	41	Grain	0.06
Rhône Alpes France 2007 Menhir 6/4 30/4 EC	2 (24)	0.75 0.75	200 200	69	35 42 49	Grain Grain Grain	0.01 0.01 0.01
North-Rhine Westphalia/Kleve Germany 2007 Franziska 16/4 18/5 EC	2 (32)	0.75 0.75	200 200	69	35 42	Grain Grain	0.02 0.02
Brunne Brandenburg Germany 2007 Campanile 6/4 13/5 EC	2 (37)	0.75 0.75	200 200	69	43	Grain	0.01
Sorocaba SP Brazil 2011 BRS 195 13/9 20/9 EC S23°25'20.19 W47°23'03.95	2 (7)	0.75 0.75	100 100	69 77	35	Grain	< 0.05
Cambará PR Brazil 2011 Câue 20/9 27/9 EC S23°00'13 W50°02'17	2 (7)	0.75 0.75	100 100	78 81	35	Grain	0.07
Iracemápolis SP Brazil 2011 BRS 195 6/10 13/10 EC S22°39'33 W47°31'17	2 (7)	0.75 0.75	100 100	76 79	25	Grain	< 0.05
26/9 3/10	2 (7)	0.75 0.75	100 100	73 75	35	Grain	< 0.05
16/9 23/9	2 (7)	0.75 0.75	100 100	67 72	45	Grain	< 0.05
Santa Mariana PR Brazil 2011 Câue 16/9 9/11 EC S23°08'41 W50°29'28 6/10 13/10	2 (7)	0.75 0.75	100 100	81 82	25	Grain	0.11
26/9 3/10	2 (7)	0.75 0.75	100 100	80 81	35	Grain	0.07

Location, year, variety BARLEY	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
16/9 23/9	2 (7)	0.75 0.75	100 100	75 78	45	Grain	0.08

Table 89 Supporting information on residues of fenpropimorph in barley from trial reports available only in summary form

Location, year, variety BARLEY	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Hohenschulen Germany 1980 Georgie 12/6 15/7 EC❾	2 (33)	0.75	600	75	35 42	Grain Grain	0.13 0.07
Böhl Germany 1980 Gerda 27/5 18/6❸ EC	2 (22)	0.75	600	75	28 35 44	Grain Grain Grain	0.17 0.06 < 0.05
Hergarten Germany 1980 Aura 2/6 14/7❹ EC	2 (42)	0.75	600	77	28 35	Grain Grain	0.13 0.13
Hohenschulen Germany 1980 Georgie 12/6 22/7❺ EC	2 (40)	0.75	600	75-85	28 35	Grain Grain	0.25 0.10
Hergarten Germany 1980 Aura 2/6 7/7❻ EC	2 (35)	0.75	600	73-75	35 42	Grain Grain	0.19 0.22
Böhl Germany 1980 Carina 27/5 11/6❼ EC	2 (15)	0.75	600	69	35 42 51	Grain Grain Grain	0.24 0.08 0.05
Good Easter Essex UK 1979❶ EC	1	1.1		10.5.4-11.1	55	grain	0.07 0.14 (0.10)
Good Easter Essex UK 1979❶ EC	1	0.75		10.5.4-11.1	55	grain	0.05 0.12 (0.08)
Brentwood Essex UK 1979❶ EC	1	1.1		10.5.4	35	grain	0.17 0.22 (0.20)
Brentwood Essex UK 1979❶ EC	1	0.75		10.5.4	35	grain	0.10 0.10 (0.10)
Fyfield Essex UK 1979 Ark Royal❶ EC	1	1.1	211	8-9 Feekes	71	grain	< 0.05 < 0.05 (< 0.05)
Fyfield Essex UK 1979 Ark Royal❶ EC	1	1.1	211	11.1-11.2 Feekes	29	grain	0.21 0.30 (0.26)
Fyfield Essex UK 1979 Ark Royal❶ EC	2	1.1	211	8-9 Feekes 11.1-11.2 Feekes	29	grain	0.25 0.21 (0.23)
Fyfield Essex UK 1979 Ark Royal❶ EC	2	0.75	211	8-9 Feekes 11.1-11.2 Feekes	29	grain	0.17 0.15 (0.16)
Ongar Essex UK 1979 Armelle❶ EC	1	1.1	211	7-9 Feekes	32	grain	0.11 0.10 (0.10)
Ongar Essex UK 1979 Armelle❶ EC	1	1.1	211	11.2-11.2 Feekes	74	Grain	< 0.05 < 0.05 (< 0.05)
Ongar Essex UK 1979 Armelle❶ EC	2	1.1	211	7-9 11.1-11.2	32	Grain	0.11 0.09 (0.10)
Ongar Essex UK 1979 Armelle❶ EC	2	0.75	211	7-9 11.1-11.2	32	Grain	0.08 0.09 (0.08)
Grimsby Lincolnshire UK 1979❿	2	0.75		6-7	77	Grain	< 0.05
Chiseldon Wiltshire UK 1979 ❿	2	0.75		10	48	Grain	< 0.05
Pakenham Suffolk UK 1979 ❿	1	0.75		8	75	Grain	< 0.05
Bosomworth Yorkshire UK 1979 ❿	1	1.1		10	63	Grain	< 0.05
May & Baker UK 1979 spring barley❽ EC	1	1.1		11.1	29	grain	0.13
	1	1.1		9	71	Grain	0.05
	2	0.75		9 11.1	29	Grain	0.13

Location, year, variety BARLEY	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
	2	1.1		9 11.1	29	grain	0.11
May & Baker UK 1979 spring barley❽ EC	1	1.1		11.1	32	grain	0.08
	1	1.1		9	71	Grain	0.05
	2	1.1		9 11.1	29	Grain	0.08
	2	0.75		9 11.1	29	grain	0.10
Loch Winnoch Renfrewshire UK 1980❷ EC	2	1.5		60-65	35	grain	0.11
Grayton Norfolk UK 1980❷ EC	2	0.75		57 66	59 83	Grain grain	< 0.05 < 0.05
Kineton Warwickshire UK 1980❷ EC	1	0.75 (ground)		32	81	grain	< 0.05
Kineton Warwickshire UK 1980❷ EC	1	0.75 (air)		32	81	grain	< 0.05
Dodington Lincolnshire UK 1980❷ EC	1	0.75		49	62	grain	< 0.05
Ilchester Somerset UK 1979 EC❿	1	1.1		10.5	37 48	Grain Grain	0.07 < 0.05
May & Baker UK 1979 winter barley❽ EC	1	1.1		10.5	35	Grain	0.09
	1	0.75		10.5	35	Grain	0.05
May & Baker UK 1979 winter barley❽ EC	1	1.1		10.5	55	Grain	< 0.05
	1	0.75		10.5	55	Grain	0.05
Ross on Wye Herefordshire UK 1979❷ EC	1	0.75		32	88	Grain	< 0.05
Wimborne Dorset UK 1979 ❷	2	0.75		32	108	grain	< 0.05
Dorchester UK 1979❷ EC	2	0.75		39-45	87	Grain	< 0.05
Swaton Lincolnshire UK 1979❷ EC	2	0.75		58	39	grain	0.27
Blyton Lincolnshire UK 1979❷ EC	3	0.75		69	35	grain	< 0.05

❶1980 10189, ❷1981-10737, ❸1980 10358, ❹1980 10359, ❺1980 10360, ❻1980 10795, ❼1980 10797❽1981 10736, ❾1980 10357, ❿1980 10822

Table 90 Supporting data for residues of fenpropimorph in oats from trial reports available only in summary form

Location, year, variety OATS	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
West Yonderton, Paisley UK 1980❷ EC	2	0.75		22	35 81	Grain Grain	0.36 < 0.05
Heckington Lincolnshire UK 1980❷	2	0.75		32-33	83	Grain	< 0.05
Stetten Baden-Württemberg Germany 1992 Klaus❸	3 (15 24)	0.76 0.76 0.74	304 304 298	37 49 75	35 41	Grain Grain	0.1 0.09

❷1981 10737, ❸1993 10693

Table 91 Supporting data for residues of fenpropimorph in rye from trial reports available only in summary form

Location, year, variety RYE	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Limburgerhof Rheinland-Pflaz Germany 1992 Luchs❶	3 (7 34)	0.77 0.77 0.77	310 310 308	39 49 75	35 42	Grain Grain	0.08 0.11
Dunsville Yorkshire UK 1983❷	2	0.75			49	Grain	< 0.05
Standon St John Oxfordshire UK 1993 Animo❷	2	0.75			45	Grain	< 0.05

❶1993 10693, ❷1984 1000101

Table 92 Residues of fenpropimorph in wheat (2001 1009068, 2004 1010542, 2005 7004267, 2007 1050094, 2007 1050096)

Location, year, variety WHEAT	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Middlefart, Denmark 2000 Kris 18/5 29/6	2 (42)	0.73 0.76	296 309	39 69-71	43 50	Grain Grain	< 0.05 < 0.05
Halton Holegate UK 2000 Consort 22/5 5/7	2 (44)	0.75 0.73	303 298	37-39 69	42 49	Grain Grain	< 0.05 < 0.05
Middlefart Fuenen Denmark 2003 Triso EC 29/7	1	0.4	300	77	35 41	Grain Grain	< 0.01 < 0.01
	1	0.75	300	77	35 41	Grain Grain	0.02 0.01
Sevilla Andalucia Spain 2003 Vitromax 21/4	1	0.4	300	69	35 42	Grain Grain	< 0.01 < 0.01
	1	0.75	300	69	35 42	Grain Grain	< 0.01 < 0.01
Aussonne Midi-Pyrenes France (south) 2003 Nefer 28/5	1	0.4	300	73	35 42	Grain Grain	0.02 0.01
	1	0.75	300	73	35 42	Grain Grain	0.02 0.01
Bicester Oxfordshire UK 2003 Malacca 2/7	1	0.4	300	83	34 41	Grain Grain	0.01 0.01
	1	0.75	300	83	34 41	Grain Grain	0.02 0.02
Fuenen Denmark 2005 Kris 24/5 6/7 EC	2 (43)	0.4 0.4	200 200	69	35 42	Grain Grain	< 0.01 < 0.01
	2 (43)	0.75 0.75	200 200	69	35 42	Grain Grain	0.01 0.01
Baden-Württemberg Germany 2005 Isengrain 25/5 30/6 EC	2 (36)	0.4 0.4	200 200	69	27 34 41	Grain Grain Grain	< 0.01 < 0.01 < 0.01
	2 (36)	0.75 0.75	200 200	69	27 34 41	Grain Grain Grain	< 0.01 < 0.01 < 0.01
Rhône-Alpes France 2005 Caphorn 9/5 30/5 EC	2 (21)	0.4 0.4	200 200	69	28 35 42	Grain Grain Grain	0.01 < 0.01 < 0.01
	2 (21)	0.75 0.75	200 200	69	28 35 42	Grain Grain Grain	< 0.01 0.02 0.01
Pays de le Loire France 2005 Royssac 27/4 31/5 EC	2 (34)	0.4 0.4	200 200	69	35 42	Grain Grain	< 0.01 < 0.01
	2 (34)	0.75 0.75	200 200	69	35 42	Grain Grain	< 0.01 < 0.01

Location, year, variety WHEAT	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
North-Rhine Westphalia Germany 2007 Biscay 19/4 29/5 EC	2 (40)	0.75 0.75	200 200	69	36 42	Grain Grain	≤ 0.01 < 0.01
Brunne Brandenburg Germany 2007 Brilliant 6/4 31/5 EC	2 (55)	0.75 0.75	200 200	69	34 41 49	Grain Grain Grain	≤ 0.01 < 0.01 < 0.01
Midi Pyrenes France 2007 Kalango 3/4 9/5	2 (36)	0.75 0.75	200 200	69	49	Grain	< 0.01
Rhône-Alpes France 2007 Epidoc 6/4 23/5 EC	2 (47)	0.75 0.75	200 200	69	35 42 47	Grain Grain Grain	0.04 0.03 0.03

Table 93 Supporting information on residues of fenpropimorph in wheat from trial reports available only in summary form

Location, year, variety WHEAT	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Stommeln Köln Germany 1977 Kolibri 10/6 1978-1000062① EC	1	0.75	600		56 63	Grain Grain	< 0.05 < 0.05
Altheim Germany 1977 Kolibri 7/6① EC	1	0.75	600		49 56 63	Grain Grain Grain	< 0.05 0.05 < 0.05
Kiel Germany 1977 Kolibri 20/6① EC	1	0.75	600		56 63 70	Grain Grain Grain	< 0.05 < 0.05 < 0.05
Speyer Germany 1977 Kolibri 26/5① EC	1	0.75	600		63	Grain	< 0.05
Mechtersheim Germany 1977 Kolibri 26/5 14/6① EC	2 (18)	0.75	600		42 49 56	Grain Grain Grain	< 0.05 < 0.05 < 0.05
Keil Germany 1977 Kolibri 20/6 27/6① EC	2 (7)	0.75	600		49 56	Grain Grain	< 0.05 < 0.05
Altheim Germany 1977 Kolibri 7/6 and 22/6 1978 1000062① EC	2 (15)	0.75	600		35 42 49 56	Grain Grain Grain Grain	0.07 < 0.05 < 0.05 < 0.05
Stommeln Köln Germany 1977 Kolibri 24/6① EC	1	0.75	600		42 49 56	Grain Grain Grain	< 0.05 < 0.05 < 0.05
Böhl Germany 1979 Schirokko 30/5 4/7② EC	2	0.75	600		29 36	Grain Grain	< 0.05 < 0.05
Hohenschulen Germany 1979 Selpek 7/6 24/7③ EC	2	0.75	600		28 35 42	Grain Grain Grain	< 0.05 < 0.05 < 0.05
Stommeln Germany 1979 Schirocko 8/6 23/7④ EC	2	0.75	600		28 35	Grain Grain	0.08 0.08
Ketsch Germany 1979 Kolibri 6/6 10/7⑤ EC	2	0.75	600		28 35	Grain Grain	0.08 0.06
Stommeln Germany 1979 Schirocko 8/6 19/7⑥ EC	2	0.75	600		32 39	Grain Grain	< 0.05 < 0.05
Holzen Germany 1979 Quintus 30/5 11/7⑦ EC	2	0.75	600		35	Grain	0.08
Ketsch Germany 1979 Kolibri 6/6 29/6⑧ EC	2	0.75	600		28 35 42	Grain Grain Grain	< 0.05 0.05 < 0.05
Böhl Germany 1979 Schirokko 5/6 28/6⑨ EC	2	0.75	600		28 35 42	Grain Grain Grain	0.07 0.09 0.07

Location, year, variety WHEAT	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Hohenschulen Germany 1979 Selpek 7/6 17/7⑩ EC	2	0.75	600		35 42 49	Grain Grain Grain	< 0.05 < 0.05 < 0.05
Ongar Essex UK 1979 Maris Huntsman❶ EC	1	1.1	211	8-9 Feekes	84	Grain	< 0.05 < 0.05
Ongar Essex UK 1979 Maris Huntsman❶ EC	1	1.1	211	11.1 Feekes	34	Grain	< 0.05 < 0.05
Ongar Essex UK 1979 Maris Huntsman❶ EC	2	1.1	211	8-9 Feekes 11.1 Feekes	34	Grain	< 0.05 0.05
Ongar Essex UK 1979 Maris Huntsman❶ EC	2	0.75	211	8-9 Feekes 11.1 Feekes	34	Grain	< 0.05 < 0.05
Great Dunmow Essex UK 1979 Maris Hobbit❶ EC	1	1.1	211	8 Feekes, 37 Zadocks	88	Grain	< 0.05 < 0.05
Great Dunmow Essex UK 1979 Maris Hobbit❶ EC	1	1.1	211	11.1 Fek 73 Zad	41	Grain	< 0.05 < 0.05
Great Dunmow Essex UK 1979 Maris Hobbit❶ EC	2	1.1	211	8 Fe 37 Z 11.1 F 73 Z	41	Grain	< 0.05 < 0.05
Great Dunmow Essex UK 1979 Maris Hobbit❶ EC	2	0.75	211	8 Fe 37 Z 11.1 F 73 Z	41	Grain	< 0.05 < 0.05
Holbeach Lincolnshire UK 1980❷ EC	2	0.75		60-55	72	Grain	0.056
Rutland Leicestershire UK 1980❷ EC	2	0.75		57-60	72	Grain	< 0.05
Swaton Lincolnshire UK 1980 ❷ EC	3	0.75		70-75	55 74	Grain Grain	< 0.05 < 0.05
Gloucestershire UK 1980❷ EC	2	0.75		50	35 47	Grain Grain	< 0.05 < 0.05
Sebstead Kent UK 1980❷ EC	1	0.75		69	42	Grain	0.06
Spalding UK 1980❷ EC	2	0.75		56-58	50	Grain	< 0.05
Harris Norfolk UK 1980❷ EC	1	0.75air		60	35 82	Grain Grain	0.34 < 0.05
	1	0.75grd		60	35 82	Grain Grain	0.11 < 0.05
Yorkshire UK 1980❷ EC	1	0.75		60-68	39	Grain	0.08
Usk Gwent UK 1980❷ EC	1	0.75		45	35 69	Grain Grain	- < 0.05
Shimpling Suffolk UK 1980❷ EC	1	0.75		32	145	Grain	< 0.05
Shepherdswell Kent UK 1980❷ EC	1	0.75		59	62	Grain	< 0.05
Canterbury Kent UK 1980❷ EC	1	0.75		71	75	Grain	0.20
Preston Deanery Northantshire 1979 EC❸	1	1.5		10.1	25 46	Grain Grain	0.07 < 0.05
Bridgewater Sommerset UK 1979 EC❸	1	1.1		10.5	27 62	Grain Grain	0.27 < 0.05
Elmsett Suffolk UK 1979❸	1	1.1		7	102	Grain	< 0.05
Bosomworth Yorkshire UK 1979 EC❸	1	1.1		8	98	Grain	< 0.05
Ongar Essex UK 1979 EC❸	1	1.1		9	84	Grain	< 0.05

Location, year, variety WHEAT	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Ongar Essex UK 1979 EC❸	1	1.1		11.1	34	Grain	< 0.05
Ongar Essex UK 1979 EC❸	2	0.75		11.1	34	Grain	< 0.05
Ongar Essex UK 1979 EC❸	2	1.1		11.1	34	Grain	< 0.05
Dunmow Essex UK 1979 EC❸	2	0.75		11.1	41	Grain	< 0.05
Dunmow Essex UK 1979 EC❸	2	1.1		11.1	41	Grain	< 0.05
Dunmow Essex UK 1979 EC❸	1	1.1		9	91	Grain	< 0.05
Dunmow Essex UK 1979 EC❸	1	1.1		11.1	41	Grain	< 0.05

①1978-1000062, ②1979 10261, ③1979 10262, ④1979 10263, ⑤1979 10330, ⑥1979 10331, ⑦1979 10332, ⑧1979 10333, ⑨1979 10334, ⑩1979 10335, ❶1980 10189, ❷1981-10737, ❸1980 10822

PRIMARY FEED COMMODITIES OF PLANT ORIGIN

Table 94 Residues of fenpropimorph in sugar beet tops (Linder 2014a 2013/1396627, Oxspring 2009a 2009/1012810, Schulz H., 2009 a 2009/1075091, Perny 2004 1024749, Lantos 2000 1013434)

Location, year, variety SUGAR BEET TOPS	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Wunstorf, Lower Saxony Germany 2007 Modus	1	0.75	200	49	0 22 29 35 42	leaves with tops leaves with tops leaves with tops leaves with tops leaves with tops	6.5 0.20 0.14 0.15 0.09
Grantham, Leicestershire UK 2007 Octa	1	0.75	200	45-48	0 21 31 35 42	leaves with tops leaves with tops leaves with tops leaves with tops leaves with tops	6.5 0.47 0.24 0.44 0.44
Mauchenheim Germany 2008 Tiziana	1	0.78	208	39	0 21 28 36 41	leaves with tops leaves with tops leaves with tops leaves with tops leaves with tops	6.8 0.19 0.11 0.09 0.06
Loivre France (north) 2008 Julietta	1	0.74	198	39	0 20 29 35 41	leaves with tops leaves with tops leaves with tops leaves with tops leaves with tops	6.4 0.48 0.33 0.20 0.22
Mauchenheim Germany 2013 Kleist	1	0.75	200	39	0 22 28 36	Tops Tops Tops tops	13 0.34 0.17 0.093
Witry Les Reims France (north) 2013 Vienna	1	0.72	200	39	0 20 29 35	Tops Tops Tops Tops	8.0 0.31 0.21 0.13
Ottersum Netherlands 2013 Isabella	1	0.80	200	39	0 21 28 36	Tops Tops Tops Tops	14 0.29 0.22 0.15
Lawford Essex UK 2013 Pasteur	1	0.80	200	47	0 21 27 35	Tops Tops Tops Tops	29 0.30 0.20 0.15

Location, year, variety SUGAR BEET TOPS	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Finhan France (south) 2007 Python	1	0.75	200	47	0 3 7 14	leaves with tops leaves with tops leaves with tops leaves with tops	15 2.3 1.1 0.29
Via Olmo, Bologna Italy 2007 Zanzibar	1	0.75	200	47	0 3 8 14	leaves with tops leaves with tops leaves with tops leaves with tops	8.5 2.3 < 0.01* 1.2
Nerja Spain 2008 Diamante	1	0.74	197	49	0 3 7 14	leaves with tops leaves with tops leaves with tops leaves with tops	12 0.25 0.22 0.22
Pella, North Macedonia, Greece 2008 Areti	1	0.74	196	47	0 3 7 14	leaves with tops leaves with tops leaves with tops leaves with tops	4.8 1.4 0.79 0.21
Szabolcs-Szatmár- Bereg County, Nagyhalász Hungary 1999 Marika	2 (14)	0.44 0.44	300 300	End of vegetation	35	Leaves with tops	0.06 < 0.05 0.09
Salleras Sevilla Spain 2003 Lucia	2 (14)	0.25 0.25	300 300	37 39	0 28 42	Leaves Leaves Leaves	2.2 0.06 < 0.05
Gullena Spain 2003 Napoli	2 (14)	0.25 0.25	300 300	39 39	0 28 43	Leaves Leaves Leaves	1.4 < 0.05 < 0.05
Nisi Alexandria Greece 2003 23-39 Rizor	2 (15)	0.25 0.25	300 300	38 38	0 27 41	Leaves Leaves Leaves	2.7 < 0.05 < 0.05 < 0.05
Drepano Greece 2003 Asso	2 (14)	0.25 0.25	300 300	38 38	0 28 42	Leaves Leaves Leaves	2.6 < 0.05 < 0.05 < 0.05

* Likely that the treated and untreated specimens were mislabelled during the study since corresponding untreated specimens gave residue levels of 1.53 mg/kg and 0.020 mg/kg for 'leaves with tops' and 'roots', respectively

Table 95 Supporting data for residues of fenpropimorph in sugar beet tops from trial reports available only in summary form

Location, year, variety SUGAR BEET TOPS	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Stättahra Halland Sweden 1985 Primahill①	3 (16 11)	0.75 0.75 0.75	250 250 250		65	Leaves	0.22
Villers la Ville Belgium 1985②	1	0.75	600		46	Leaves	0.09 0.08 0.09
Capalbio Italy 1991 Buramo③	1	0.75	400		0 20 30		6.0 0.17 -
Auberive, France (north) 1983 Monosvalof④	1	0.75		maturity	72	leaves	0.2
Beaumont sur Vesle France 1983 Régina⑤	1	0.75		Maturity	82	Leaves	0.09
Jodoigne Belgium 1985⑥	1	0.75	600		72	Leaves	0.07
Ambresin Belgium 1985⑦	1	0.75	600		62	Leaves	0.04 < 0.02
Assevillers France (north) 1983 Monostar⑧	2 (14)	0.75 0.75			68	Leaves	0.28
Bressana Boharone Italy 1987 Dima⑨	3 (22 19)	0.75 0.75 0.75	400 400 400		20	Leaves	< 0.05

Location, year, variety SUGAR BEET TOPS	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Cervesina Italy 1991 Perla⑩	3 (14 15)	0.75 0.75 0.75	500 500 500		0 20 32	Leaves Leaves Leaves	6.5 0.64 0.44
Abinia Italy 1989 Monova❶	2 (15)	0.56 0.56	500 500		3	Leaves	0.8
Marsigliana Italy 1989 Maribo Unica❷	2 (13)	0.56 0.56	500 500		5	Leaves	1.6
Due Pavoni Italy 1989 Maribo Monova❸	2 (17)	0.56 0.56	500 500		16 31	Leaves Leaves	0.23 0.06
Corana Italy 1989 Novaghemo❹	3 (12 10)	0.56 0.56 0.56	400 400 400	49	1 7 13 22	Leaves Leaves Leaves Leaves	0.20 0.93 0.33 0.14
Ceruesina Italy 1988 Rizo❺	3 (16 18)	0.56 0.56 0.56	500 500 500	49	2 8 15 30	Leaves Leaves Leaves Leaves	0.48 0.82 0.33 1.0

① 1985 10359, ② 1986 10161, ③ 1991 11786, ④ 1983 10351, ⑤ 1983 10353, ⑥ 1986 10160, ⑦ 1986 10162, ⑧ 1983 10352, ⑨ 1987 10463, ⑩1991 11785, ❶1989 10504, ❷1989 10505, ❸1989 10768, ❹1989 10770, ❺1988 10823

Table 96 Residues of fenpropimorph in barley plants and straw (2001 1009068, 2001 1009081, 2004 1010542, 2005 7004267, 2007 1050094, 2012 3003426)

Location, year, variety BARLEY STRAW	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Puerto S. María, Cádiz, Andalucia Spain 2000 Almudena 23/3 18/4	2 (26)	0.76 0.72	305 290	47-49 75	0 21 21 35 42	pl. w/o roots ears haulms straw straw	5.9 < 0.05 < 0.05 < 0.05 < 0.05
Algodonales, Cádiz, Andalucia Spain 2000 Irene 17/4 28/4	2 (11)	0.75 0.75	302 301	47-49 67	0 21 21 35 42	pl. w/o roots ears haulms straw straw	9.7 < 0.05 0.1 < 0.05 < 0.05
Settala, Milano, Lombardia Italy 2000 Baraka 21/4 23/5	2 (32)	0.74 0.73	297 296	47 69	0 21 21 35 42	pl. w/o roots ears haulms straw straw	1.3 < 0.05 0.05 0.16 0.05
Liscate, Milano, Lombardia Italy 2000 Astrix 26/4 17/5	2 (21)	0.77 0.76	309 305	49 65-67	0 21 21 35 42	pl. w/o roots ears haulms straw straw	6.6 0.21 0.28 0.39 0.36
Lund Sweden 2000 Barke 5/6 29/6	2 (24)	0.72 0.76	294 310	37 59	0 28 28 35 35 42 49	Shoots Ears Stalks Ears Stalks Straw Straw	13 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 0.07

Location, year, variety BARLEY STRAW	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Salmonby UK 2000 Pearl 13/5 30/5	2 (17)	0.77 0.76	312 306	39 61	0 28 28 35 35 42 42 49 49 56 64	Shoots Ears Stalks Ears Stalks Ears Stalks Ears Stalks Straw Straw	9.8 < 0.05 0.09 < 0.05 0.12 < 0.05 0.06 < 0.05 0.05 0.09 0.12
Taissy France (north) 2000 Esterel 9/5 26/5	2 (17)	0.74 0.74	301 302	39 59	0 27 27 34 42	pl. w/o root ears haulms straw straw	5.6 0.08 < 0.05 0.10 0.17
Brandenburg Germany 2003 Candesse 26/5	1	0.4	300	69	0 28 28 35 42	Plant w/o root Ears Culm Straw Straw	5.7 < 0.05 0.10 0.07 < 0.05
	1	0.75	300	69	0 28 28 35 42	Plant w/o root Ears Culm Straw Straw	11 0.09 0.12 0.14 0.06
Seebach Alsace France 2003 Astoria 12/6	1	0.4	300	73	0 28 28 36 42	Plant w/o root Ears Culm Straw Straw	4.0 < 0.05 < 0.05 < 0.05 < 0.05
	1	0.75	300	73	0 28 28 36 42	Plant w/o root Ears Culm Straw Straw	8.2 0.08 0.08 0.07 < 0.05
Rhône-Aples France 2003 Orelie 15/5	1	0.4	300	83	0 29 29 35 42	Plant w/o root Ears Culm Straw Straw	2.6 < 0.05 0.09 < 0.05 < 0.05
	1	0.75	300	83	0 29 29 35 42	Plant w/o root Ears Culm Straw Straw	5.8 0.06 0.12 0.06 0.08
Piemonte Italy 2003 Prosa 26/5	1	0.4	300	55	0 28 28 35 42	Plant w/o root Ears Culm Straw Straw	10 < 0.05 0.12 0.08 0.06
	1	0.75	300	55	0 28 28 35 42	Plant w/o root Ears Culm Straw Straw	14 < 0.05 0.22 0.15 0.08
Rheinland-Pfalz Germany 2005 Scarlett 25/5 9/6	2 (15)	0.4 0.4	200 200	69	0 0 29 36 42	Whole plant Ear, panicle Straw Straw Straw	5.0 - 0.05 0.07 0.05

Location, year, variety BARLEY STRAW	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
	2 (15)	0.75 0.75	200 200	69	0 0 29 36 42	Whole plant Ear, panicle Straw Straw Straw	7.4 - < 0.01 0.09 0.13
Rhône-Alpes France 2005 Orelie 26/4 18/5	2 (22)	0.4 0.4	200 200	69	0 0 29 29 35 41	Whole plant Ear, panicle Whole plant Ear, panicle Straw Straw	5.6 5.1 0.13 < 0.01 0.15 0.03
	2 (22)	0.75 0.75	200 200	69	0 0 29 29 35 41	Whole plant Ear, panicle Whole plant Ear, panicle Straw Straw	7.8 9.9 0.26 0.02 0.1 0.05
Malmoe Sweden 2005 Prestige 2/6 14/7	2 (42)	0.4 0.4	200 200	69	0 0 29 29 36 43	Whole plant Ear, panicle Whole plant Ear, panicle Straw Straw	1.9 1.9 0.07 0.04 0.07 0.04
	2 (42)	0.75 0.75	200 200	69	0 0 29 29 36 43	Whole plant Ear, panicle Whole plant Ear, panicle Straw Straw	7.9 3.0 0.11 0.08 0.08 0.05
Oxfordshire UK 2005 Pearl 5/5 20/6	2 (46)	0.4 0.4	200 200	73	0 0 28 35 42	Whole plant Ear, panicle Straw Straw Straw	3.0 2.1 0.06 0.12 0.11
	2 (46)	0.75 0.75	200 200	73	0 0 28 35 42	Whole plant Ear, panicle Straw Straw Straw	2.2 1.9 0.13 0.11 0.14
Duras Aquitaine France 2007 Nikel 5/4 10/5	2 (35)	0.75 0.75	200 200	69	0 12 15 0 35 35 41	Plant w/o roots Plant w/o roots Plant w/o roots Plant w/o roots Ears Rest plant w/o rt Straw	3.5 0.12 0.09 3.2 0.05 < 0.01 0.03
Rhône Alpes France 2007 Menhir 6/4 30/4	2 (24)	0.75 0.75	200 200	69	0 6 10 0 35 42 49	Plant w/o roots Plant w/o roots Plant w/o roots Plant w/o roots Straw straw Straw	8.4 1.4 0.64 11 0.04 0.03 0.02
North-Rhine Westphalia/Kleve Germany 2007 Franziska 16/4 18/5	2 (32)	0.75 0.75	200 200	69	0 9 11 0 35 42	Plant w/o roots Plant w/o roots Plant w/o roots Plant w/o roots Straw straw	17 1.2 0.60 9.2 0.04 0.03

Location, year, variety BARLEY STRAW	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Brunne Brandenburg Germany 2007 Campanile 6/4 13/5	2 (37) 0.75	0.75 0.75	200 200	69	0 13 21 0 36 36 43	Plant w/o roots Plant w/o roots Plant w/o roots Plant w/o roots Ears Rest plant w/o rt Straw	13 0.09 < 0.01 4.1 0.02 0.01 0.01

Table 97 Supporting data for fenpropimorph residues in barley straw from trial reports available only in summary form

Location, year, variety BARLEY STRAW	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Hohenschulen Germany 1980 Georgie 12/6 15/7 ❾	2 (33)	0.75	600	75	0 21 28 35 42	whole plant whole plant whole plant straw straw	200 0.36 0.25 0.69 1.3
Böhl Germany 1980 Gerda 27/5 18/6❸	2 (22)	0.75	600	75	0 14 21 28 35 44	whole plant whole plant whole plant straw straw straw	7.6 0.71 0.71 0.54 0.76 0.74
Hergarten Germany 1980 Aura 2/6 14/7❹	2 (42)	0.75	600	77	0 14 21 28 35	whole plant whole plant whole plant straw straw	27 0.76 0.59 0.86 1.34
Hohenschulen Germany 1980 Georgie 12/6 22/7❺	2 (40)	0.75	600	75-85	0 14 21 28 35	whole plant whole plant whole plant straw straw	11 0.58 0.40 1.0 2.5
Hergarten Germany 1980 Aura 2/6 7/7❻	2 (35)	0.75	600	73-75	0 21 28 35 42	whole plant whole plant whole plant straw straw	16 0.93 0.63 1.7 1.0
Böhl Germany 1980 Carina 27/5 11/6❼	2 (15)	0.75	600	69	0 21 28 35 42 51	whole plant whole plant whole plant straw straw straw	27 0.79 0.97 0.61 1.2 0.65
Good Easter Essex UK 1979 4/7❶	1	1.1		10.5.4-11.1	55	straw	0.20 0.12
Good Easter Essex UK 1979 4/7❶	1	0.75		10.5.4-11.1	55	straw	0.06 0.16
Brentwood Essex UK 1979 2/7❶	1	1.1		10.5.4	35	straw	0.86 0.86
Brentwood Essex UK 1979 2/7❶	1	0.75		10.5.4	35	straw	0.64 1.2
Fyfield Essex UK 1979 Ark Royal 18/6❶	1	1.1	211	8-9 Feekes	71	straw	0.48 0.63
Fyfield Essex UK 1979 Ark Royal 30/7❶	1	1.1	211	11.1-11.2 Feekes	29	straw	0.63 0.94
Fyfield Essex UK 1979 Ark Royal 18/6 30/7❶	2	1.1	211	8-9 Feekes 11.1-11.2 Feekes	29	straw	1.1 1.2
Fyfield Essex UK 1979 Ark Royal 18/6 30/7❶	2	0.75	211	8-9 Feekes 11.1-11.2 Feekes	29	straw	0.75 0.56
Ongar Essex UK 1979 Armelle 30/7❶	1	1.1	211	7-9 Feekes	32	straw	0.14 0.22

Location, year, variety BARLEY STRAW	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Ongar Essex UK 1979 Armelle 18/6❶	1	1.1	211	11.2-11.2 Feekes	74	straw	0.10 0.18
Ongar Essex UK 1979 Armelle 18/6 30/7❶	2	1.1	211	7-9 11.1-11.2	32	straw	0.23 0.69
Ongar Essex UK 1979 Armelle 18/6 30/7❶	2	0.75	211	7-9 11.1-11.2	32	straw	0.30 0.75
Grimsby Lincolnshire UK 1979❿	2	0.75		6-7	2877	plant straw	1.8 1.2
Chiseldon Wiltshire UK 1979 ❿	2	0.75		10	28 48	Plant straw	1.1 2.2
Pakenham Suffolk UK 1979 ❿	1	0.75		8	0 7 14 21 75	plant plant plant plant straw	27 7.3 2.6 2.6 1.2
Bosomworth Yorkshire UK 1979 ❿	1	1.1		10	63	straw	1.5
May & Baker UK 1979❽	1	1.1		11.1	29	straw	2.5
	1	1.1		9	71	Straw	2.1
	2	0.75		9 11.1	29	Straw	3.2
	2	1.1		9 11.1	29	Straw	4.0
May & Baker UK 1979❽	1	1.1		11.1	32	Straw	1.3
	1	1.1		9	74	Straw	0.34
	2	1.1		9 11.1	32	Straw	1.2
	2	0.75		9 11.1	32	Straw	1.7
Loch Winnoch Renfrewshire UK 1980❷	2	1.5		60-65	35	Straw	1.3
Grayton Norfolk UK 1980❷	2	0.75		57 66	59 83	straw straw	0.10 1.7
Kineton Warwickshire UK 1980❷	1	0.75 (ground)		32	35 81	whole plant straw	0.76 0.28
Kineton Warwickshire UK 1980❷	1	0.75 (air)		32	35 81	whole plant straw	0.95 0.28
Dodington Lincolnshire UK 1980❷	1	0.75		49	35 62	whole plant straw	0.58 0.82
Ilchester Somerset UK 1979 ❿	1	1.1		10.5	37 48	Straw straw	1.8 1.6
May & Baker UK 1979❽	1	1.1		10.5	35	straw	1.1
	1	0.75		10.5	35	straw	2.0
May & Baker UK 1979❽	1	1.1		10.5	55	Straw	1.0
	1	0.75		10.5	55	straw	0.9
Dorchester UK 1979❷	2	0.75		39-45	87	Straw	0.35
Swaton Lincolnshire UK 1979❷	2	0.75		58	39	Straw	3.9
Blyton Lincolnshire UK 1979❷	3	0.75		69	35	Straw	0.10

❶1980 10189, ❷1981 10737, ❸1980 10358, ❹1980 10359, ❺1980 10360, ❻1980 10795, ❼1980 10797, ❽1981 10736, ❾1980 10357, ❿1980 10822

Table 98 Supporting data for residues of fenpropimorph in oat straw from trial reports available only in summary form

Location, year, variety OATS	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
West Yonderton, Paisley UK 1980❷	2	0.75		22	35	Straw	0.77
Heckington Lincolnshire UK 1980❷	2	0.75		32-33	38 49 83	Plant Plant straw	1.1 0.47 1.2
Stetten Baden-Württemberg Germany 1992 Klaus❸	3 (15 24)	0.76 0.76 0.74	304 304 298	37 49 75	0 10 10 35 41	Plant Ear Haulm Straw Straw	4.0 0.28 0.84 2.1 1.4

❷1981 10737, ❸1993 10693

Table 99 Supporting data for residues of fenpropimorph in rye from trial reports available only in summary form

Location, year, variety RYE	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Limburgerhof Rheinland-Pflaz Germany 1992 Luchs❶	3 (7 34)	0.77 0.77 0.77	310 310 308	39 49 75	0 11 11 35 42	Plant Ear Haulm Straw Straw	2.1 0.94 3.2 2.5 2.5
Dunsville Yorkshire UK 1983❷	2	0.75			49	Straw	1.3
Standon St John Oxfordshire UK 1993 Animo❷	2	0.75			45	Straw	0.95

❶1993 10693, ❷1984 1000101

Table 100 Residues of fenpropimorph in wheat plants and straw wheat (2001 1009068, 2004 1010542, 2005 7004267, 2007 1050094, 2007 1050096)

Location, year, variety WHEAT STRAW	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Middlefart, Denmark 2000 Kris 18/5 29/6 2001 1009068	2 (42)	0.73 0.76	296 309	39 69-71	0 29 29 36 36 43 50	pl. w/o root ears haulms ears haulms straw straw	5.5 0.17 0.51 0.12 0.47 0.33 0.32
Halton Holegate UK 2000 Consort 22/5 5/7 2001 1009068	2 (44)	0.75 0.73	303 298	37-39 69	0 28 28 35 35 42 49	pl. w/o root ears haulms ears haulms straw straw	8.2 0.20 0.34 0.22 0.25 0.30 0.23
Middlefart Fuenen Denmark 2003 Triso EC 29/7	1	0.4	300	77	0 28 28 35 41	Plant w/o root Ears Culm Straw Straw	3.0 0.07 0.32 0.25 0.13
	1	0.75	300	77	0 28 28 35 41	Plant w/o root Ears Culm Straw Straw	7.3 0.09 0.58 0.51 0.27

Location, year, variety WHEAT STRAW	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Sevilla Andalucia Spain 2003 Vitromax 21/4	1	0.4	300	69	0 29 29 35 42	Plant w/o root Ears Culm Straw Straw	6.8 < 0.05 0.12 0.11 0.11
	1	0.75	300	69	0 29 29 35 42	Plant w/o root Ears Culm Straw Straw	15 0.05 0.24 0.24 0.18
Aussonne Midi-Pyrenes France (south) 2003 Nefer 28/5	1	0.4	300	73	0 28 28 35 42	Plant w/o root Ears Culm Straw Straw	5.2 c1.0 0.08 0.47 c0.13 0.32 c0.12 0.36 c0.12
	1	0.75	300	73	0 28 28 35 42	Plant w/o root Ears Culm Straw Straw	16 c1.0 0.27 0.49 c0.13 0.46 c0.12 0.32 c0.12
Bicester Oxfordshire UK 2003 Malacca 2/7	1	0.4	300	83	0 27 27 34 41	Plant w/o root Ears Culm Straw Straw	4.2 < 0.05 0.20 0.17 0.19
	1	0.75	300	83	0 27 27 34 41	Plant w/o root Ears Culm Straw Straw	5.8 0.06 0.28 0.24 0.26
Fuenen Denmark 2005 Kris 24/5 6/7	2 (43)	0.4 0.4	200 200	69	0 0 28 28 35 42	Whole plant Ears, panicle Whole plant Ears, panicle Straw Straw	2.2 1.3 0.08 0.07 0.10 0.06
	2 (43)	0.75 0.75	200 200	69	0 0 28 28 35 42	Whole plant Ears, panicle Whole plant Ears, panicle Straw Straw	4.2 0.33 0.03 0.09 <u>0.07</u> 0.05
Baden-Württemberg Germany 2005 Isengrain 25/5 30/6	2 (36)	0.4 0.4	200 200	69	0 0 27 34 41	Whole plant Ears, panicle Straw Straw Straw	3.7 2.1 0.17 0.13 0.05
	2 (36)	0.75 0.75	200 200	69	0 0 27 34 41	Whole plant Ears, panicle Straw Straw Straw	6.2 4.4 0.21 <u>0.14</u> 0.11
Rhône-Alpes France 2005 Caphorn 9/5 30/5	2 (21)	0.4 0.4	200 200	69	0 0 28 35 42	Whole plant Ears, panicle Straw Straw Straw	3.2 3.2 0.30 0.14 0.10
	2 (21)	0.75 0.75	200 200	69	0 0 28 35 42	Whole plant Ears, panicle Straw Straw Straw	7.2 3.8 <u>0.34</u> 0.25 0.16

Location, year, variety WHEAT STRAW	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Pays de le Loire France 2005 Royssac 27/4 31/5	2 (34)	0.4 0.4	200 200	69	0 0 28 28 35 42	Whole plant Ears, panicle Whole plant Ears, panicle Straw Straw	2.3 2.8 0.08 0.05 0.08 0.09
	2 (34)	0.75 0.75	200 200	69	0 0 28 28 35 42	Whole plant Ears, panicle Whole plant Ears, panicle Straw Straw	9.5 12 0.05 0.07 0.21 0.14
North-Rhine Westphalia Germany 2007 Biscay 19/4 29/5	2 (40)	0.75 0.75	200 200	69	0 15 22 0 36 42	Plant w/o roots Plant w/o roots Plant w/o roots Plant w/o roots Straw Straw	23 0.56 0.11 8.4 0.07 0.06
Brunne Brandenburg Germany 2007 Brilliant 6/4 31/5	2 (55)	0.75 0.75	200 200	69	0 30 37 0 34 41 49	Plant w/o roots Plant w/o roots Plant w/o roots Plant w/o roots Straw Straw straw	25 0.06 < 0.01 10 0.11 0.07 0.05
Midi Pyrenes France Kalango 3/4 9/5	2 (36)	0.75 0.75	200 200	69	0 20 23 0 35 35 42 42 49	Plant w/o roots Plant w/o roots Plant w/o roots Plant w/o roots Ears Rest of plant Ears Rest of plant straw	38 0.24 0.15 < 0.01 0.05 0.07 0.07 0.02 0.07
Rhône-Alpes France 2007 Epidoc 6/4 23/5	2 (47)	0.75 0.75	200 200	69	0 10 20 0 35 42 47	Plant w/o roots Plant w/o roots Plant w/o roots Plant w/o roots Straw Straw straw	11 1.1 0.37 8.1 0.12 0.08 0.08

Table 101 Supporting data for residues of fenpropimorph in wheat straw from trial reports available only in summary form

Location, year, variety WHEAT STRAW	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Stommeln Köln Germany 1977 Kolibri 10/6 ①	1	0.75	600		0 28 35 42 49 56 63	whole plant whole plant whole plant whole plant whole plant straw straw	10 1.1 0.41 0.26 0.12 0.13 0.08
Altheim Germany 1977 Kolibri 7/6①	1	0.75	600		0 28 35 42 49 56 63	whole plant whole plant whole plant whole plant straw straw straw	14 0.14 0.11 0.05 0.20 < 0.05 < 0.05

Location, year, variety WHEAT STRAW	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Kiel Germany 1977 Kolibri 20/6①	1	0.75	600		0 28 35 42 49 56 63 70	whole plant whole plant whole plant whole plant whole plant straw straw straw	17 0.76 0.27 0.14 < 0.05 0.08 0.07 0.07
Speyer Germany 1977 Kolibri 26/5①	1	0.75	600		0 28 35 42 49 56 63	whole plant whole plant whole plant whole plant whole plant straw straw	8.6 0.20 0.17 0.08 0.11 0.09 0.07
Mechtersheim Germany 1977 Kolibri 26/5 14/6①	2 (18)	0.75	600		0 21 28 35 42 49 56	whole plant whole plant whole plant whole plant straw straw straw	7.3 0.54 0.36 0.26 0.29 0.20 0.17
Keil Germany 1977 Kolibri 20/6 27/6①	2 (7)	0.75	600		0 21 28 35 42 49 56	whole plant whole plant whole plant whole plant whole plant straw straw	17 1.4 0.43 0.31 0.21 0.10 0.11
Altheim Germany 1977 Kolibri 7/6 22/6①	2 (15)	0.75	600		0 21 28 35 42 49 56	whole plant whole plant whole plant straw straw straw straw	11 0.28 0.25 0.19 0.18 0.06 0.06
Köln Germany 1977 Kolibri 24/6①	1	0.75	600		0 21 28 35 42 49 56	whole plant whole plant whole plant whole plant straw straw straw	7.8 0.78 0.44 0.36 0.19 0.10 0.10
Böhl Germany 1979 Schirokko 30/5 4/7②	2	0.75	600		0 15 22 22 29 36 189	whole plant whole plant whole plant whole plant straw straw roll	9.1 1.8 3.3 0.06 2.6 2.9 < 0.05
Hohenschulen Germany 1979 Selpek 7/6 24/7③	2	0.75	600		0 14 21 28 35 42 169	whole plant whole plant whole plant straw straw straw roll	13 1.6 1.0 1.9 1.6 1.6 < 0.05
Stommeln Germany 1979 Schirocko 8/6 23/7④	2	0.75	600		0 14 21 28 35 170	whole plant whole plant whole plant straw straw roll	14 2.2 1.6 3.6 3.6 < 0.05

Location, year, variety WHEAT STRAW	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Ketsch Germany 1979 Kolibri 6/6 10/7⑤	2	0.75	600		0 14 21 28 35	whole plant whole plant whole plant straw straw	17 2.8 5.9 4.8 3.6
Stommeln Germany 1979 Schirocko 8/6 19/7⑥	2	0.75	600		0 18 25 32 39	whole plant whole plant whole plant straw straw	5.1 1.9 1.3 2.76 2.73
Holzen Germany 1979 Quintus 30/5 11/7⑦	2	0.75	600		0 21 28 35	whole plant whole plant whole plant straw	14 1.1 1.08 0.92
Ketsch Germany 1979 Kolibri⑧	2	0.75	600		0 21 28 35 42	whole plant whole plant straw straw straw	5.2 1.2 1.2 2.1 1.8
Böhl Germany 1979 Schirokko 5/6 28/6⑨	2	0.75	600		0 21 28 35 42	whole plant whole plant straw straw straw	14 3.9 4.4 4.2 4.3
Hohenschulen Germany 1979 Selpek 7/6 17/7⑩	2	0.75	600		0 21 28 35 42 49	whole plant whole plant whole plant straw straw straw	13 0.80 0.50 1.1 0.79 0.79
Ongar Essex UK 1979 Maris Huntsman 6/6 ❶	1	1.1	211	8-9 Feekes	84	straw	0.27 0.16
Ongar Essex UK 1979 Maris Huntsman 26/7 ❶	1	1.1	211	11.1 Feekes	34	straw	0.37 0.54
Ongar Essex UK 1979 Maris Huntsman 6/6 26/7 ❶	2	1.1	211	8-9 Feekes 11.1 Feekes	34	straw	0.48 0.72
Ongar Essex UK 1979 Maris Huntsman 6/6 26/7❶	2	0.75	211	8-9 Feekes 11.1 Feekes	34	straw	0.53 0.42
Great Dunmow Essex UK 1979 Maris Hobbit 9/6❶	1	1.1	211	8 Feekes, 37 Zadocks	88	straw	0.05 0.08
Great Dunmow Essex UK 1979 Maris Hobbit 26/7❶	1	1.1	211	11.1 Fek 73 Zad	41	Straw	0.17 0.10
Great Dunmow Essex UK 1979 Maris Hobbit 9/6 26/7❶	2	1.1	211	8 Fe 37 Z 11.1 F 73 Z	41	Straw	0.24 0.22
Great Dunmow Essex UK 1979 Maris Hobbit 9/6 26/7❶	2	0.75	211	8 Fe 37 Z 11.1 F 73 Z	41	Straw	0.19 0.15
Preston Deanery, Northhantshire UK 1979 ❸	1	1.5		10.1	25 46	straw straw	6.0 1.6
Bridgewater, Somerset UK 1979 ❸	1	1.1		10.5	27 46	straw straw	1.1 1.4
Mickfield Suffolk UK 1979 ❸	1	0.75		10.5.4	0 7 14 21	plant plant plant plant	6.7 1.3 1.1 0.57
Elmsett Suffolk UK 1979 ❸	1	1.1		7	102	straw	1.8

Location, year, variety WHEAT STRAW	N (int)	Rate kg ai/ha	L/ha	GS application	DALA	Sample	Residues (mg/kg)
Bosomworth Yorkshire UK 1979 ❸	1	1.1		8	98	straw	1.2
Ongar Essex UK 1979 ❸	1	1.1		9	84	straw	1.6
Ongar Essex UK 1979 ❸	1	1.1		11.1	34	straw	3.3
Ongar Essex UK 1979 ❸	2	0.75		11.1	34	straw	3.8
Ongar Essex UK 1979 ❸	2	1.1		11.1	34	straw	4.0
Dunmow Essex UK 1979 ❸	2	0.75		11.1	41	straw	3.3
Dunmow Essex UK 1979 ❸	2	1.1		11.1	41	straw	4.9
Dunmow Essex UK 1979 ❸	1	1.1		9	91	straw	1.8
Dunmow Essex UK 1979 ❸	1	1.1		11.1	41	straw	3.2

①1978 1000062, ②1979 10261, ③1979 10262, ④1979 10263, ⑤1979 10330, ⑥1979 10331, ⑦1979 10332, ⑧1979 10333, ⑨1979 10334, ⑩1979 10335, ❶1980 10189, ❷1981 10737, ❸1980 10822

FATE ON PROCESSING

Effects on the Nature of the Residues during Processing

Scharf (1999 a 1999/11092) studied the degradation behaviour of fenpropimorph during different processes (pasteurisation, baking, brewing, boiling and sterilisation) relevant to processing. Two tests were performed using [morpholine-2(6)-^{14}C]-fenpropimorph, whereas the third test was performed using [phenyl-U-^{14}C]-fenpropimorph. The test substance was dissolved in aqueous buffer solutions of different pH-values. For LSC- and HPLC-analysis, aliquots were taken right before starting a test and at the end of the test after cooling of the solution.

Table 102 Recovery data after the simulation of processing (mean values of three measurements)

Process	Test conditions	morpholine label % TAR* after test	phenyl label % TAR* after test
Pasteurisation	pH 4, 90 °C, 20 min	99.7	
Baking, brewing and boiling	pH 5, 100 °C, 60 min		95.9
Sterilisation	pH 6, 120 °C, 20 min	100.1	

* Total applied radioactivity

Fenpropimorph was not degraded during the simulation of pasteurization (pH 4, 90 °C) nor during the simulation of baking, boiling, brewing (pH 5, 100 °C) or during sterilization (pH 6, 120 °C).

The Meeting received trials on the processing of sugar beet, barley, oats and wheat.

Sugar beet

Plier 2013a (2013/1037958) conducted a study on the residue behaviour of fenpropimorph in sugar beets and their processed products. The sugar beets were obtained from three trials conducted in Germany at Motterwitz in Saxony, Schackenthal in Saxony-Anhalt and Häschendorf in Mecklenburg-West Pomerania. At each site an EC formulation of fenpropimorph was foliar applied once at a target rate 3.75 kg ai/ha (actual 3.45–3.92 kg ai/ha) with application 14 days before intended harvest using a spray volume of 200 L/ha. Sugar beets were sampled on the day of application and 14–15 days after.

Mature sugar beet roots were transported at ambient temperature to the processing facility where they were processed into refined sugar, molasses and dry pulp samples.

Sugar beets (255–282 kg batch per trial site) were cleaned prior to processing by washing by spraying beets with water in a cylindrical beet-washer. Cleaned beets were then sliced in a Königsfelder slicer and the slices (cossettes) were extracted using a DdS-extraction trough with a denaturation temperature 75.5–77.1 °C (water temperature 69.8–72.8 °C). The raw juice was purified using a two-stage liming followed by a two-stage carbonation process.

Pre-liming

Cold pre-liming was carried out at a temperature between 36.8 and 42.1 °C with lime milk (approx. 330 g $Ca(OH)_2$/L equivalent to approx. 250 g CaO/L) up to pH 11.2 (lime milk was added stepwise). After reaching the above-mentioned pH-value, the reaction time was 15–22 minutes.

Main-liming

After the reaction time of the pre-liming had expired, the total lime milk quantity (2% $Ca(OH)_2$ in the juice) was added and the juice was heated up to a temperature of 82.9–85.0 °C. After an interval of 15 minutes (reaction time), the first carbonation was started.

First Carbonation

During this step, carbon dioxide was added until the pH-value of pre-liming (pH 11.2) was reached. The carbonation temperature was at 83.4–85.1 °C. Frame filter presses were used for the subsequent filtration process.

Second Carbonation

The filtrate was heated up to 93.1–93.9 °C and carbon dioxide was added until the lime ($Ca(OH)_2$) content in the juice was reaching a minimum (optimum alkalinity, pH 9.1 to 9.2) Subsequently the filtration was carried out by means of frame filter presses.

At the end of the process thin juice was sampled

Evaporating Crystallisation

Evaporating Crystallisation was conducted under a reduced pressure of 0.25 bar. The thick juice was concentrated into the metastable supersaturation state. As soon as the desired supersaturation was reached, powdered sugar was injected as seed. In order to maintain a constant supersaturation, thick juice was soaked in continuously. Subsequently crystallisation was finished and the massecuite was brought into the cooling crystalliser.

Cooling Crystallisation

During this process the massecuite from the evaporating crystallisation was cooled down to 22.5–39.5 °C over a period of 2 to 20 hours.

Fifteen different fractions of sugar beet products or intermediates were collected for analysis, namely roots without tops, washed beets, wash water, cossettes, pressed pulp, press water, raw juice, thin juice, thick juice, molasses, raw sugar, affinated syrup, refined sugar, dried pulp and ensiled pulp.

Centrifugation was used to separate crystals and molasses to obtain molasses and raw sugar. After centrifuging, the raw sugar was air-dried. Molasses and raw sugar were sampled.

Decolouring

For the raw sugar purification, distilled water was added (approx. 10% of raw sugar weight). The raw sugar and added water were mixed for 35–50 min. After mixing, the intermediate product was centrifuged and affinated syrup and pure sugar were obtained from the separation. Affinated syrup was sampled. Pure sugar was air-dried and sampled as refined sugar.

Pulp

After extraction, the wet pulp was pressed and separated into press water and pressed pulp. Samples of both were taken. One part of the pressed pulp was dried in a drying chamber at 35 °C until a moisture content of < 10% was reached. Dried pulp was sampled. A further part of the pressed pulp was used for silage production. The extracted sliced beet was put in a special silage glass container (under pressure). Subsequently, the glass container was closed and stored at 19.6–20.3 °C for approx. 6 weeks. After this fermentation time, ensilage pulp was sampled.

The specimens were analysed for residues of fenpropimorph using BASF method No. L0076/01, which has a limit of quantitation (LOQ) of 0.01 mg/kg. The mean recovery results for fenpropimorph were 92.9% (RSD: 11%) at fortification levels from 0.01 to 100 mg/kg.

The mean transfer factors representing the different processing steps were below one for washed beets, wash water, cossettes, pressed pulp, press water, raw juice, thin juice, thick juice, molasses, raw sugar, affinated syrup and refined sugar. Therefore it can be concluded, that fenpropimorph is not being accumulated in these processed fractions. The mean transfer factor of dried pulp and ensiled pulp (> 1) indicates that fenpropimorph residues are concentrated due to water loss (in case of ensiled pulp also weight reduction due to loss of gases, e.g. CO_2).

Table 103 Residues of fenpropimorph in sugar beet processed fractions following processing of roots (Plier 2013a 2013/1037958)

Location, year, variety	No	kg ai/ha	DALA	Crop part	Residue (mg/kg)	TF
Motterwitz Germany 2012 Taifun **282 kg batch**	1	13.45	0	Leaves with tops	27	-
			0	Roots	0.62	-
			15	Leaves with tops	2.8	-
			15	Roots	0.30	-
			15	Roots prior to processing	0.24	1.00
				Washed beets	0.062	0.26
				Wash water	0.11	0.46
				Cossettes	0.053	0.22
				Pressed pulp	0.13	0.54
				Press water	< 0.01	< 0.04
				Raw juice	< 0.01	< 0.04
				Thin juice	< 0.01	< 0.04
				Thick juice	< 0.01	< 0.04
				Molasses	< 0.01	< 0.04
				Raw sugar	< 0.01	< 0.04
				Affinated syrup	< 0.01	< 0.04
				Refined sugar	< 0.01	< 0.04
				Dried pulp	0.94	3.92
				Ensiled pulp	0.24	1.00
Schackenthal Germany 2012 Benno **263 kg batch**	1	3.92	0	Leaves with tops	17	-
			0	Roots	0.019	-
			15	Leaves with tops	3.2	-
			15	Roots	0.015	-
			15	Roots prior to processing	< 0.01	1.00
				Washed beets	< 0.01	-
				Wash water	< 0.01	-
				Cossettes	< 0.01	-
				Pressed pulp	0.013	>1.30
				Press water	< 0.01	-
				Raw juice	< 0.01	-
				Thin juice	< 0.01	-
				Thick juice	< 0.01	-
				Molasses	< 0.01	-
				Raw sugar	< 0.01	-
				Affinated syrup	< 0.01	-
				Refined sugar	< 0.01	-
				Dried pulp	0.099	>9.90
				Ensiled pulp	0.029	>2.90

Location, year, variety	No	kg ai/ha	DALA	Crop part	Residue (mg/kg)	TF
Häschendorf Germany 2012 Emila **255 kg batch**	1	3.90	0	Leaves with tops	60	-
			0	Roots	0.34	-
			14	Leaves with tops	4.6	-
			14	Roots	0.22	-
			14	Roots prior to processing	0.16	1.00
				Washed beets	0.035	0.22
				Wash water	0.099	0.62
				Cossettes	0.030	0.19
				Pressed pulp	0.062	0.39
				Press water	< 0.01	< 0.06
				Raw juice	< 0.01	< 0.06
				Thin juice	< 0.01	< 0.06
				Thick juice	< 0.01	< 0.06
				Molasses	< 0.01	< 0.06
				Raw sugar	< 0.01	< 0.06
				Affinated syrup	< 0.01	< 0.06
				Refined sugar	< 0.01	< 0.06
				Dried pulp	0.47	2.94
				Ensiled pulp	0.13	0.81

Fenpropimorph was concentrated in dried pulp and in ensiled pulp. In all further matrices, the residues declined during processing.

Wheat

Schulz 2002a (2001/1009082) studied the transfer of fenpropimorph during the processing of wheat. Four field trials were conducted in different representative wheat growing areas in Germany where two applications of fenpropimorph, as an EC-formulation, were made at 1.5 kg ai/ha. The growth stages for application were BBCH 47–49 and 75–77 targeting harvest about 35 days after the final application. Grain samples could only be collected from one out of four trials due to continued bad weather. In those trials where the wheat was not ripe enough for threshing, ears were taken instead of grain. After 50–60 DALA, grain for both analysis and processing was sampled from all trials. The samples were analysed for residues of fenpropimorph using BASF method No. 241/3 (LOQ 0.05 mg/kg for all sample matrices). Concurrent recoveries averaged at 74% (SD: 5.5, CV: 7.5%, n=29).

At zero DALA, the residues of fenpropimorph in the whole aerial part of plant were between 15.43 and 38.30 mg/kg. Because of continued bad weather, ear samples were taken from three trials at 35–36 DALA. The residues thereof were between 0.076 and 0.130 mg/kg. From the fourth trial, the grain was taken at 35 DALA as scheduled. The fenpropimorph residues were 0.064 mg/kg. The fenpropimorph residues of the other three trials (taken at 50–60 DALA) were below the LOQ.

Mature grain was transported at ambient temperature to the processing facility where they were processed into cleaned grain which is the wheat raw agricultural commodity, fine bran (middlings), coarse bran, total bran, straight flour, low grade meal, scoured bran, flour (type 550), wholemeal flour and wholemeal bread using pilot scale processing equipment.

Grain (16.3–26.6 kg) was cleaned in batches using a dockage tester and a laboratory grain cleaner o remove straw, ear particles, dust and dirt by wind sifting, sieves and the trieur. Cleaned grain was conditioned to a moisture content of 15–17% by addition of water and 12 kg used for processing to flour (type 550) and 4–5 kg for processing to wholemeal flour.

Milling of flour (type 550)

Conditioned grain was milled and sieved in a Bühler Mahlautomat in which the grain passed three breaking rolls followed by three resolution rolls. After passing the breaking rolls, three portions of break flour and one fraction of coarse bran were obtained. After the resolution rolls, three portions of resolution flour and one fraction of fine bran (middlings) were obtained. The six flour fractions were

combined to obtain straight flour. The coarse and fine bran fractions were combined and scoured using a laboratory bran duster to give low grade meat (adhered flour to bran) and scoured bran. Straight flour and low grade meal were blended to obtain flour (type 550) with an ash content of 0.6%.

Milling of wholemeal flour

Conditioned grain was milled and sieved in a Bühler Mahlautomat as for milling of flour type 550 however, the resulting whole bran and straight flour were combined.

Wholemeal flour was subsequently used to prepare sour-dough wholemeal bread.

Table 104 Residues of fenpropimorph in wheat processed fractions following processing of grain (Schulz 2002a 2001/1009082)

Location, year, variety	No	kg ai/ha	DALA	Crop part	Residue (mg/kg)	TF
Aarbergen-Panrod Germany 2000 Pegassos 26.5 kg batch	2 (42)	1.5 1.6	0	Plant w/o roots	15.73	
			36	Ears	0.13	
			60	Grain	< 0.05	
				Cleaned grain (RAC)	< 0.05	
				Fine bran (middlings)	0.055	>1.1
				Coarse bran	0.072	>1.4
				Total bran	0.068	>1.4
				Straight flour	< 0.05	
				Low grade meal	< 0.05	
				Scoured bran	0.118	>2.4
				Flour (type 550)	< 0.05	
				Wholemeal flour	< 0.05	
				Wholemeal bread	< 0.05	
Grabau Germany 2000 Rialto 20.6 kg batch	2 (37)	1.5 1.5	0	Plant w/o roots	15.43	
			35	Ears	0.092	
			51	Grain	< 0.05	
				Cleaned grain (RAC)	< 0.05	
				Fine bran (middlings)	< 0.05	
				Coarse bran	0.05	
				Total bran	0.05	>1
				Straight flour	< 0.05	
				Low grade meal	< 0.05	
				Scoured bran	0.072	>1.4
				Flour (type 550)	< 0.05	
				Wholemeal flour	< 0.05	
				Wholemeal bread	< 0.05	
Sonneborn Germany 2000 Aristos 16.3 kg batch	2 (40)	1.5 1.5	0	Plant w/o roots	15.65	
			35	Ears	0.076	
			50	Grain	< 0.05	
				Cleaned grain (RAC)	< 0.05	
				Fine bran (middlings)	< 0.05	
				Coarse bran	< 0.05	
				Total bran	< 0.05	
				Straight flour	< 0.05	
				Low grade meal	< 0.05	
				Scoured bran	< 0.05	
				Flour (type 550)	< 0.05	
				Wholemeal flour	< 0.05	
				Wholemeal bread	< 0.05	
Uttenweiler-Ahlen Germany 2000 Bussard 21.4 kg batch	2 (43)	1.5 1.5	0	Plant w/o roots	38.3	
			-	Ears	-	

Location, year, variety	No	kg ai/ha	DALA	Crop part	Residue (mg/kg)	TF
			35	Grain	0.064	
				Cleaned grain (RAC)	0.05	
				Fine bran (middlings)	0.137	2.7
				Coarse bran	0.149	3.0
				Total bran	0.146	2.9
				Straight flour	< 0.05	<1
				Low grade meal	0083	1.7
				Scoured bran	0.161	3.2
				Flour (type 550)	< 0.05	<1
				Wholemeal flour	0.063	1.3
				Wholemeal bread	0.057	1.1

Although the application rate used was the twofold of the normal practice only fenpropimorph residues at or below the limit of quantitation were found in the cleaned grain, which was the starting material for the processing. From the residue values found in the process fractions, it can be concluded that in the different bran fractions especially in the scoured bran some concentrations of fenpropimorph took place. However, in the important final products flour, wholemeal flour and wholemeal bread, no or very slight concentrations were observed.

In a separate study Kniep (2006a 2006/1025930) also studied the transfer of fenpropimorph during the processing of wheat. Two field trials were conducted in different representative wheat growing areas in Germany where two applications of fenpropimorph, as an EC-formulation, were made at 3.75 kg ai/ha. The applications were made 69 days and 41 days as well as 61 days and 33 days before harvest using a spray volume of 300 L/ha. The specimens were analysed according to BASF method no. 535/0 (LOQ 0.01 mg/kg for all sample matrices). Concurrent recoveries averaged at 92% (CV: 17%, n=16). Apart from the cleaned grain, which is the wheat raw agricultural commodity, the following fractions were prepared and analysed: fine bran (middlings), coarse bran, total bran, straight flour, low-grade meal, scoured bran, flour (type 550), wholemeal flour and wholemeal bread.

Mature grain was transported at ambient temperature to the processing facility where they were processed into cleaned grain which is the wheat raw agricultural commodity, fine bran (middlings), coarse bran, total bran, straight flour, low grade meal, scoured bran, flour (type 550), wholemeal flour and wholemeal bread using pilot scale processing equipment.

Grain (16.3–26.6 kg) was cleaned in batches using a laboratory grain cleaner. Where necessary, cleaned grain was conditioned to a moisture content of 15–16% by addition of water and 12 kg used for processing to flour (type 550) and 4-5 kg for processing to wholemeal flour.

Milling of flour (type 550)

Conditioned grain was milled and sieved in a Bühler Mahlautomat to produce coarse bran, fine bran (middlings) and straight flour. In a further processing step the low-grade meal (toppings) were separated from coarse bran and middlings by a centrifuge/scouring machine to obtain total bran and toppings. Straight flour and low grade meal (toppings) were blended to obtain flour (type 550) with an ash content of 0.6%.

Milling of wholemeal flour

Conditioned grain was milled and sieved in a Bühler Mahlautomat as for milling of flour type 550 resulting in coarse bran, middlings and straight flour. In a further processing step the low-grade flour (toppings) was separated from coarse bran and middlings by a centrifuge/scouring machine. The total bran (coarse and fine bran fractions) was cracked with an impact mill to smaller pieces and all milling products were used for wholemeal flour (straight flour, total bran and toppings).

Wholemeal flour was subsequently used to prepare sour-dough wholemeal bread.

Wheat germ

To produce wheat germ grain was bruised in a roller mill (0.5 mm gap) and the 400–1000 µm faction collected. The fraction up to 1000 µm was broken on a 0.3 mm gap roller mill and the milling/sieving process repeated three times. The roller gap for the final milling was set at 0.2 mm. The fractions < 400 and > 1000 µm were discarded and the 400-10000 µm fraction (combined bran, semolina, germ) separated in a Leichtgewichtsauslesser). The semolina and germ mixture was milled to flour, small germ discs with a small amount of bran with a smooth roller and the bran sieved.

No residues of fenpropimorph above the LOQ were found in the untreated specimens, with exception of plant without roots (0.03 mg/kg), middlings (0.03 mg/kg) and wheat germs (0.02 mg/kg).

Table 105 Residues of fenpropimorph in wheat processed fractions following processing of grain (Kniep 2006a 2006/1025930)

Location, year, variety	No	kg ai/ha	DALA	Crop part	Residue (mg/kg)	TF
Neustadt Dosse Brandenburg Germany 2005 Winnetou 23.3 kg batch	2 (28)	3.75 3.68	0	Plant w/o roots	46	
			41	Grain at harvest (RAC)	0.32	
				Fine bran (middlings)	0.77	2.41
				Coarse bran	0.05	0.16
				Total bran	0.06	0.19
				Straight flour	0.09	0.28
				Toppings	0.16	0.50
				Flour (type 550)	0.09	0.28
				Wholemeal flour	0.49	1.53
				Wholemeal bread	0.45	1.41
				Wheat germ	0.76	2.37
Kleinrheinfeld Bavaria Germany 2005 Enorm 20.7 kg batch	2 (28)	3.94 3.72	0	Plant w/o roots	43	
			33	Grain at harvest (RAC)	0.17	
				Fine bran (middlings)	1.0	5.88
				Coarse bran	1.1	6.47
				Total bran	1.1	6.47
				Straight flour	0.05	0.29
				Toppings	0.39	2.29
				Flour (type 550)	0.06	0.35
				Wholemeal flour	0.24	1.41
				Wholemeal bread	0.55	3.24
				Wheat germ	0.71	4.18

In the final products flour and wholemeal flour only in the wholemeal flour, a slight concentration was observed. In the wholemeal bread, a concentration of fenpropimorph of a factor 1.5 to 3 was determined.

Barley

Schulz 2002b (2001/1009083) studied the transfer of fenpropimorph during the processing of barley. Four field trials were conducted in different representative barley growing areas in Germany where two applications of fenpropimorph, as an EC-formulation, were made at 1.5 kg ai/ha. The growth stages for application were BBCH 47–49 and 69–75 targeting a harvest about 35 days after the last application. The samples were analysed for residues of fenpropimorph using BASF method No. 241/3 (LOQ 0.05 mg/kg for all sample matrices). Concurrent recoveries averaged at 81% (CV: 11.8%, n=28).

At zero DALA, the residues of fenpropimorph in the whole aerial part of plant were between 15.7 and 41.5 mg/kg. Because of continued bad weather, grain samples could only be collected from one out of four trials. In the other trials where the barley was not ripe, enough for threshing, ears were taken instead of grain. After 51–54 DALA, grain for both analysis and processing was sampled from all trials.

Mature grain was transported at ambient temperature to the processing facility. In the processing part, the production of the two consumer products beer and pot barley were simulated. The malting and brewing process led to the fractions spent grain (drip dry), condensed water, trub (flocs), beer yeast, beer, beer (frozen). In the milling process, pot barley and pearling dust were obtained.

Grain was cleaned in batches using a laboratory grain cleaner to particles < 2.5 mm and > 6.5 mm as well as other impurities and stones. Cleaned grain was conditioned to a moisture content of 13–15% by drying where required.

De-hulling

Barley grain was pearled using a Schule-Vertikal-Schälmaschine with a target abrasion of 7-14% determined as the ratio of pearling dust to cleaned grain.

Processing to beer

Cleaned grain was steeped in water at a nominal temperature of 16 °C for 44 h (cycles contact with water and aeration) prior to being placed in a germination box at 13–15 °C for 4 days Following germination the kiln dried for about 15 hours with an initial temperature of 55–56 °C and a final temperature of 80–81 °C. The resulting malt is freed from germ by rubbing over a sieve and the germ and malt collected. The was ground by two passes through a two roll mill and the ground malt and grist mashed into decarbonised water heated to 52 °C with stirring and then to 76 °C. The mash was drained off into a heated lauter tub, the wort separated from the spent grain by filtration, and the spent grain washed with sparging water. The wort and sparging liquid were boiled and hop extract added. After additional heating, the material coagulated and the trub was separated from the wort. Yeast was added the wort and the mixture fermented at 9.5–24.5 °C for 6–9 days after which fermentation was complete. The green beer was drained cold filtered and bottled.

Table 106 Residues of fenpropimorph in barley processed fractions following processing of grain (Schulz 2002 b 2001/1009083)

Location, year, variety	No	kg ai/ha	DALA	Crop part	Residue (mg/kg)	TF
Hünstetten-Ketternschwalbach Germany 2000 Scarlett Batch 7 kg malting 1.9 kg milling	2 (14)	1.5 1.5	0	Plant w/o roots	41.5	
			35	Ears	0.107	
			54	Grain (field)	< 0.05	
				Grain prior to processing	< 0.05	
				Brewing malt	< 0.05	
				Malt germ	< 0.05	
				Spent grain	0.063	>1.3
				Condensed water	< 0.05	
				Trub (flocs)	< 0.05	
				Beer yeast	< 0.05	
				Beer (frozen)	< 0.05	
				Cleaned grain	< 0.05	
				Pot barley	< 0.05	
				Pearling dust	0.085	>1.7
Hünstetten-Leusel Germany 2000 Scarlett Batch 7 kg malting 1.7 kg milling	2 (37)	1.5 1.5	0	Plant w/o roots	15.74	
			35	Ears	0.051	
			51	Grain (field)	< 0.05	
				Grain prior to beer processing	< 0.05	
				Brewing malt	< 0.05	
				Malt germ	< 0.05	
				Spent grain	< 0.05	
				Condensed water	< 0.05	
				Trub (flocs)	< 0.05	

Location, year, variety	No	kg ai/ha	DALA	Crop part	Residue (mg/kg)	TF
				Beer yeast	< 0.05	
				Beer (frozen)	< 0.05	
				Cleaned grain	< 0.05	
				Pot barley	< 0.05	
				Pearling dust	< 0.05	
Brüheim Germany 2000 Barke Batch 7 kg malting 1.9 kg milling	2 (40)	1.5 1.5	0	Plant w/o roots	20.8	
			35	Ears	0.143	
			52	Grain (field)	< 0.05	
				Grain prior to beer processing	0.096	-
				Brewing malt	0.08	0.8
				Malt germ	0.051	0.5
				Spent grain	0.151	1.6
				Condensed water	< 0.05	< 0.5
				Trub (flocs)	< 0.05	< 0.5
				Beer yeast	< 0.05	< 0.5
				Beer (frozen)	< 0.05	< 0.5
				Cleaned grain	0.082	-
				Pot barley	0.063	0.8
				Pearling dust	0.198	2.4
Biberach Germany 2000 Scarlett Batch 7 kg malting 2.2 kg milling	2 (43)	1.5 1.5	0	Plant w/o roots	22.1	
			35	Grain (field)	0.207	
				Grain prior to beer processing	0.142	-
				Brewing malt	0.203	1.4
				Malt germ	0.07	0.5
				Spent grain	0.316	2.2
				Condensed water	< 0.05	< 0.4
				Trub (flocs)	< 0.05	< 0.4
				Beer yeast	< 0.05	< 0.4
				Beer (frozen)	< 0.05	< 0.4
				Cleaned grain	0.129	
				Pot barley	0.205	1.6
				Pearling dust	0.424	3.3

In beer, which is the final product of the malting and brewing process, no fenpropimorph residues above the limit of quantitation and therefore no concentration of residues could be observed. In pot barley, the final product of the milling process, in one out of four samples, a slight concentration (transfer factor 1.6) was observed.

Harant (2006a 2006/1025931) also studied the transfer of fenpropimorph on processing barley. Two field trials were conducted in representative spring barley growing areas in Germany in order to generate specimens for processing and determining residues in brewing malt, malt culms, spent grain, spent hops (flocs), spent yeast, beer, pot barley, pearling dust/bran and flour. An EC formulation of fenpropimorph was foliar applied twice at an exaggerated target rate of 3.75 kg ai/ha. The applications were made 63 (± 1) days and 35 (± 1) days before the planned harvest date, using a spray volume of 300 L/ha. Plants without roots were collected at the day of the last application and 35 (± 1) days later for analysis of the raw agricultural commodity and for processing. The specimens were analysed according to BASF method no. 535/0 (LOQ 0.01 mg/kg for all sample matrices). Concurrent recovery values averaged at 103% (CV 15%, n=22).

Before malting grain was sieved, cleaned, and graded to a minimum grain size (2.5 mm sieve).

Processing to beer

A combined wet/dry steeping process was used. The steeping temperature was 13.5-14.5 °C and the final water content achieved was 44.5–44.7%.

Steeped grain was placed in a germination box at 13.5–14.5 °C and 80-100% relative humidity for 5 days following germination the germinated grain was kiln dried from a starting water content of 44.6–46.3% to a final water content of 4.3–4.4%. The resulting malt is freed from germ by rubbing over a sieve and the germ and malt collected. The was ground by two passes through a two roll mill and the ground malt and grist mashed into tap water heated to 47–48 °C with stirring and then to 76 °C. The mash was drained off into a heated lauter tub, the wort separated from the spent grain by filtration, and the spent grain washed with sparging water. The wort and sparging liquid were boiled and hop extract added. After additional heating, the material coagulated and the trub was separated from the wort. Yeast was added the wort and the mixture fermented for 8–9 days after which fermentation was complete. The green beer was drained and filtered.

De-hulling

Barley grain was conditioned (dampened) to a moisture content of 15–15.2% prior to milling. Grain was pearled using a Schule-Vertikal-Schälmaschine with a target abrasion of 20–25% determined as the proportion of pot barley to the total portion of purified grain used for hulling.

No residues of fenpropimorph above the LOD were found in the untreated specimens of barley grain and its processed fractions, except of the specimen of plant without roots, where 0.007 mg/kg, the specimen of spent hops, where 0.01 mg/kg and the specimen flour, where 0.0042 mg/kg were found.

The residue levels detected in the treated specimens and its processed fractions as well as the calculated transfer factor are presented in the following table.

Table 107 Residues of fenpropimorph in barley processed fractions following processing of grain (Harant 2006a 2006/1025931)

Location, year, variety	No	kg ai/ha	DALA	Crop part	Residue (mg/kg)	TF
Motterwitz Saxony Germany 2005 Barke Batch 30 kg malting 5 kg milling	2 (28)	3.5 3.4	0	Plant without roots	54	
			35	Grain at harvest [A]	0.64	
				Brewing malt	0.74	1.16
				Malt culms	0.22	0.34
				Spent grain	0.71	0.96
				Spent hops (flocs)	0.08	0.11
				Spent yeast	< 0.003	< 0.004
				Beer	< 0.003	< 0.004
				Grain, moistened [B]	0.63	
				Pot barley	0.65	1.03
				Pearling dust/bran	1.1	1.75
				Flour	1.6	2.54
Kleinrheinfeld Bavaria Germany 2005 Margret Batch 30 kg malting 5 kg milling	2 (28)	3.9 3.8	0	Plant without roots	51	
			34	Grain at harvest [A]	0.94	
				Brewing malt	0.67	0.71
				Malt culms	0.33	0.35
				Spent grain	0.97	1.45
				Spent hops (flocs)	0.11	0.16
				Spent yeast	< 0.003	< 0.004
				Beer	< 0.003	< 0.004
				Grain, moistened [B]	0.93	
				Pot barley	0.52	0.56
				Pearling dust/bran	1.2	1.29
				Flour	2.3	2.47

[A] set cleaned grain to same as grain at harvest

[B] calculated

In beer, which is the final product of the malting and brewing process, no fenpropimorph residues above the limit of quantitation and therefore no concentration of residues could be observed. In pot barley, the final product of the milling process small residues but no concentration of Fenpropimorph was observed.

Oats

Four field trials were conducted in different representative oat growing areas in Germany to determine the residue levels of fenpropimorph in oat (plants without roots, grain) and process fractions (husk and dust, flakes) Renner (2005a 2005/1014084). Fenpropimorph as an EC-formulation was applied twice to oat plants at growth stages BBCH 31–32 and 55–59 at application rates of 3.75 kg ai/ha. For the analysis, whole plants without roots were taken at day 0 after the last application. Grain samples were taken at harvest (44–64 days after last application) and stored at -18 °C until ready for processing. The samples were analysed for residues of fenpropimorph using BASF method No. 456/0 that is based on LC-MS/MS quantitation after methanol/water/hydrochloric acid extraction (LOQ of the method was 0.01 mg/kg for grain, flakes, husk and dust).

Grain was cleaned using a laboratory grain cleaner (Labofix). Cleaned grain with moisture content 9.6–11.3% was kiln-dried at 120 °C for 40 minutes to a moisture content of 7.0–8.1%.

Husking

Grain was husked using a MIAG-Fliehkraftschäler and the oat grains passed to a crash ring. The glumes, husked and non-husked grains were separated (MIAG-Separator) and the remaining material passed to a Schule-Tischausleser, which separated oat nuclei from non-husked grain, and remaining glumes. Glume removal of about 30–40% was achieved. The oat nuclei were steamed (3 minutes) and the steamed nuclei rolled out (flaked) using a Schule-Flockenstuhl.

No residues at or above the limit of quantitation were detected in untreated oat grain samples or in the respective processed fractions.

Directly after the last application, the residues of fenpropimorph in plants without root were between 44 and 122 mg/kg. In oat grain harvested 44 to 64 days after the last application only low residues of fenpropimorph (< 0.01–0.03 mg/kg) were found. The process product oat flakes showed residue concentrations in the same order of magnitude as harvested grain or slightly below that. In the fractions, husks and dust, slightly higher fenpropimorph levels were found. A summary of the results including processing factors is given in Table 108.

Table 108 Residues of fenpropimorph in oat processed fractions following processing of grain (Renner 2005a 2005/1014084)

Location, year, variety	No	kg ai/ha	DALA	Crop part	Residue (mg/kg)	TF
Koppelow Germany 2003 Alfred **10 kg batch**	2 (15)	3.6 3.7	0	Plant w/o roots	121.81	
			51	Grain	0.012	
				Husks and dust	0.017	1.42
				Oat flakes	0.010	0.83
Neugattersleben Germany 2003 Flämings Profi **10 kg batch**	2 (16)	3.7 3.6	0	Plant w/o roots	43.99	
			44	Grain	0.030	
				Husks and dust	0.056	1.87
				Oat flakes	0.015	0.5
Lizendorf Germany 2003 Jumbo **10 kg batch**	2 (26)	3.4 3.6	0	Plant w/o roots	46.8	
			57	Grain	0.010	

Location, year, variety	No	kg ai/ha	DALA	Crop part	Residue (mg/kg)	TF
				Husks and dust	0.016	1.6
				Oat flakes	0.010	1
Motterwitz Germany 2003 Jumbo **10 kg batch**	2 (24)	3.5 3.3	0	Plant w/o roots	98.76	
			64	Grain	0.012	
				Husks and dust	0.012	1
				Oat flakes	0.012	1

A summary of relevant fenpropimorph processing factors is provided below.

Table 109 Summary of fenpropimorph processing factors

	Processed Fraction	Processing Factor	Best estimate PF
Sugar beet	Molasses	< 0.04 < 0.06	0.05
	Refined sugar	< 0.04 < 0.06	0.05
	Dried pulp	2.9 3.9	3.4
	Ensiled pulp	0.8 1	0.9
Wheat	Bran	0.19 2.9 6.5	2.9
	Flour	0.28 0.35 <1	0.35
	Wholemeal flour	1.3 1.4 1.5	1.4
	Wholemeal bread	1.1 1.4 3.2	1.4
	Germ	2.4 4.2	3.3
Barley	Brewing malt	0.7 0.8 1.2 1.4	1
	Spent grain	0.96 1.4 1.6 2.2	1.5
	Beer	< 0.004 < 0.004 < 0.4 < 0.5	0.004
	Pot barley	0.6 0.8 1.0 1.6	0.9
	Pearling dust	1.3 1.8 2.4 3.3	2.1
	Flour	2.5 2.5	2.5
Oats	Husks/dust	1 1.4 1.6 1.9	1.6
	Oat flakes	0.5 0.8 1 1	0.8

Livestock feeding studies

Dairy cow feeding studies

The transfer of fenpropimorph from feed to tissues and milk of dairy cows was studied by Tribolet (1999a 1999/10433). Three groups of three Simmental × Red Holstein cows (3-5 years old, 519–637 kg bw) were fed with silage maize (about 26 kg daily) and concentrate containing fenpropimorph for 28–29 days. The daily amounts of fenpropimorph were 136, 408 and 1360 mg/day provided in two daily 500 g portions of concentrate. Mean daily milk yields for the dose groups during were 15.2 to 30.2 kg/cow/day. Based on mean daily feed consumption (26 kg maize + 1.9-2.5 kg concentrate), the exposure was equivalent to 5.2 (1×), 15.7 (5×) and 52.4 (10×) ppm in the feed or 0.23, 0.68 and 2.3 mg/kg bw. Milk was collected twice daily (am and pm sampling pooled in a ratio of 6.5:3.5 v/v) at 11 intervals through the 28 days of dosing. Muscle (tenderloin, round steak, and diaphragm), liver, kidney and fat (perirenal, omental) and blood samples were collected at sacrifice a day after the last dose. The maximum frozen storage interval for tissue and milk samples was 196 days. Analysis was carried out according to method REM 167/03 for the determination of BF421-2 (fenpropimorph acid). The LOQ of the methods was 0.002 mg/kg for milk; 0.01 mg/kg for tissues and 0.05 mg/kg for blood.

Table 110 Mean residues of BF421-2 (fenpropimorph acid) in milk

Day of dosing	5.2 ppm	15.7 ppm	52.4 ppm
- 4	< 0.002	< 0.002	< 0.002
- 3	< 0.002	< 0.002	< 0.002
1	< 0.002	0.002	0.017
4	0.007	0.009	0.063
7	0.010	0.015	0.052
9	0.011	0.015	0.058

Day of dosing	5.2 ppm	15.7 ppm	52.4 ppm
14	0.013	0.019	0.108
17	0.013	0.016	0.092
21	0.010	0.018	0.051
23	0.011	0.015	0.071
28	0.012	0.019	0.065

Table 111 Residues of BF421-2 (fenpropimorph acid) in tissues

Dose group	Tenderloin (mg/kg)	Round steak (mg/kg)	Diaphragm (mg/kg)	Liver (mg/kg)	Kidney (mg/kg)	Perirenal fat (mg/kg)	Omental fat (mg/kg)
5.2 ppm	0.02 0.03 0.04	< 0.01 0.03 0.01	0.03 0.04 0.04	0.56 0.75 0.94	0.08 0.11 0.08	0.02 0.02 0.02	0.02 0.02 002
Mean	0.03	0.02	0.04	0.75	0.09	0.02	0.02
15.7 ppm	0.02 0.02 0.04	0.03 0.02 0.02	0.03 0.02 0.03	0.45 0.51 0.74	0.10 0.08 0.12	0.02 0.02 0.03	0.02 0.01 0.03
Mean	0.03	0.02	0.03	0.57	0.10	0.02	0.02
52.4 ppm	0.20 0.10 0.31	0.20 0.08 0.25	0.25 0.10 0.26	7.8 3.55 5.5	0.92 0.34 0.92	0.18 0.08 0.22	0.12 0.07 0.22
Mean	0.20	0.18	0.20	5.6	0.73	0.16	0.14

In an additional study Hafemann and Gläßgen (2006a 2005/1021394) studied the transfer of the sum of fenpropimorph and fenpropimorph acid (BF421-2) expressed as fenpropimorph. Fourteen Holstein/Friesian dairy cows (ca 3-7 years on, 508-664 kg bw) were dosed twice daily by gelatine capsule for 28 days immediately after morning and afternoon milkings. The animals were fed a daily allowance of 2×4 kg of non-medicated protein concentrate consisting of cereal silage as well as oil fruits and molasses. Good quality hay and water were also made available to the animals *ad libitum*. The average feed intake was 21 kg/day. Average doses were 43.5, 131 and 433 mg/animal/day for the three dose groups. The doses correspond to 0.07, 0.2 and 0.7 mg/kg bw and are the equivalent of in-feed concentrations of 2.0, 6.0 and 20 ppm based on intakes of 21 kg feed/days. Average milk yields 17.5, 18.4 and 19.9 kg/day for the three dose groups. Cows were milked twice daily. Milk samples were collected on study days -1, 1, 3, 5, 7, 10, 14, 17, 21, 24 and 28 from all cows (except for one cow which was terminated on study day 18) and on study days 29 and 30, 32, 34 and 35 from remaining depuration group cows. Milk from afternoon and next morning milk of each cow was pooled and two aliquots were stored in a freezer. On day 21, additional sub-samples of milk were taken and cream and skim milk were separated by centrifugation. Tissue samples were taken immediately after sacrifice of the animals (22–24 hours after the last dose except for one cow on study day 18, which was 6.5 hours, and the depuration animals). They were chopped into small pieces, weighed, placed in storage bags, and frozen immediately. The frozen samples were homogenized and stored deep-frozen. Maximum storage intervals (days) between sampling and analysis using two analytical methods and for the different samples are shown below (Table 112).

Table 112 Storage interval (days) between sampling and analysis for tissues and milk

Method	Milk	Skim milk	Cream	Muscle	Liver	Kidney	Fat
987	229	188	189	168	300	185	170
573/0	879	756	756	623	656	621	651

Samples were analysed with analytical method 987 (REM 167.03) to determine BF421 (LOQs 0.002 mg/kg in milk and 0.01 mg/kg in tissues) and with analytical method 573/0 to determine fenpropimorph and BF421 2 (LOQ 0.0019 mg/kg for milk and 0.0091 mg/kg for tissues).

Table 113 Recoveries for methods 573/0 and 987, both corrected and uncorrected for residues in controls

Matrix		Method No. 573/0				Method No 987	
		corrected		uncorrected		corrected	uncorrected
	Fortification level	fenpropimorph	BF421-2	fenpropimorph	BF421-2	BF421-2	BF421-2
Milk	0.001 [A]	90±14	93±9.7	98±14	97±13	80±30	90±17
	0.01 [A]	98±12	89±9.7	99±13	90±10	74±13	75±10

Matrix	Fortification level	Method No. 573/0 corrected fenpropimorph	Method No. 573/0 corrected BF421-2	Method No. 573/0 uncorrected fenpropimorph	Method No. 573/0 uncorrected BF421-2	Method No 987 corrected BF421-2	Method No 987 uncorrected BF421-2
	0.05 [A]	91±14	87±8.6	91±14	87±8.6	71±13	72±12
Skim milk	0.001 [A]	81	94	81	96	121±34	141±44
	0.01	83	98	83	98		
Cream	0.001 [A]	115	110	127	111	89±27	126±51
	0.005	97	108	100	108		
	0.01	106	101	108	101		
Muscle	0.005/0.05/0.5 [A]	80±6.2	95±5.1	82±6.0	95±5.1	67±22	82±3.4
Liver	0.005/0.05/0.5/2.5 [A]	78±10	95±6.7	79±10	95±6.8	78±10	80±8.1
kidney	0.01/0.05/0.5 [A]	96±5.6	96±2.2	96±5.4	96±2.2	73±8.1	73±8.6
Fat	0.005/0.05/0.5 [A]	83±13	87±11	83±13	87±11	77±4.5	77±4.4

[A] fortification levels for REM167-03 = Method 987 were milk 0.002, 0.02, 0.06 skim milk 0.002/0.06, cream 0..002/0.06, muscle 0.01/0.2, liver 0.01/0.1/2.0, kidney 0.04,0.4 and fat 0.01/0.2 m/kg.

All animals remained healthy for the duration of the study with the exception of two cows: one of which was sacrificed on study day 18 due to severe lameness, while the other was treated for mastitis observed on study day 11.

Table 114 Mean milk residues (fenpropimorph and BF421 2 and their sum) measured using method 573/0

Study day	Mean fenpropimorph residues in milk (mg/kg)								
	2.0 ppm			6.0 ppm			20 ppm		
	Parent	BF421-2	Total [A]	Parent	BF421-2	Total [A]	Parent	BF421-2	Total [A]
-1	< 0.001	< 0.0009	< 0.0019	< 0.001	< 0.0009	< 0.0019	< 0.001	< 0.0009	< 0.0019
1	< 0.001	< 0.001	< 0.002	< 0.001	0.003	0.004	0.002	0.010	0.013
3	< 0.001	0.004	0.005	0.002	0.008	0.009	0.005	0.034	0.039
5	< 0.001	0.004	0.005	0.001	0.014	0.016	0.005	0.036	0.041
7	< 0.001	0.005	0.006	0.002	0.014	0.015	0.007	0.044	0.051
10	< 0.001	0.006	0.007	0.002	0.012	0.014	0.005	0.045	0.049
14	< 0.001	0.004	0.005	0.001	0.011	0.013	0.005	0.037	0.043
17	< 0.001	0.005	0.006	0.002	0.010	0.013	0.007	0.047	0.053
21	< 0.001	0.004	0.005	0.002	0.017	0.019	0.007	0.047	0.053
24	< 0.001	0.005	0.006	0.002	0.012	0.014	0.007	0.037	0.044
28	< 0.001	0.006	0.007	0.001	0.012	0.013	0.007	0.039	0.046
29							0.003	0.036	0.038
30							< 0.001	0.024	0.025
32							< 0.001	0.010	0.011
34							< 0.001	0.004	0.005
35							< 0.001	0.002	0.003
Skim Milk	< 0.001	0.006	0.007	< 0.001	0.0165	0.018	0.002	0.051	0.053
Cream	0.011	0.004	0.015	0.024	0.011	0.035	0.094	0.027	0.121

[A] total = sum of fenpropimorph and BF421-2 expressed as fenpropimorph equivalents

Table 115 Summary of residue levels in tissues (fenpropimorph, BF421 2 and their sum [A]) measured using method 573/0

Dose group (ppm)		Muscle			Liver			Kidney			Fat		
		Parent	BF421-2	Total	Parent	BF421-2	Total	Parent	BF421-2	Total	Parent	BF421-2	Total
2.0	Mean	< 0.005	0.02	0.02	0.02	0.35	0.37	0.01	0.07	0.08	0.01	0.02	0.03
	Max	< 0.005	0.02	0.02	0.02	0.44	0.46	0.01	0.07	0.08	0.01	0.02	0.03
6.0 [B]	Mean	< 0.005	0.035	0.04	0.03	0.74	0.77	0.01	0.15	0.16	0.02	0.04	0.06
	Max	< 0.005	0.05	0.06	0.03	0.75	0.78	0.01	0.19	0.20	0.02	0.05	0.07
20	Mean	< 0.005	0.13	0.14	0.07	2.82	2.89	0.01	0.46	0.47	0.04	0.14	0.18
	Max	< 0.005	0.16	0.16	0.05	3.46	3.51	0.01	0.53	0.54	0.02	0.20	0.22
20 (2 days withdrawal)	Mean												
	Max	< 0.005	0.05	0.06	0.01	1.24	1.25	0.01	0.21	0.22	0.05	0.05	0.10

Dose group (ppm)		Muscle			Liver			Kidney			Fat		
		Parent	BF421-2	Total	Parent	BF421-2	Total	Parent	BF421-2	Total	Parent	BF421-2	Total
20 (7 days withdrawal)	Mean												
	Max	< 0.005	0.01	0.02	< 0.005	0.17	0.17	< 0.005	0.03	0.03	< 0.005	0.01	0.01

[A] total = sum of fenpropimorph and BF421-2 expressed as fenpropimorph equivalents

[B] One of the three cows in the 6 ppm dose group was slaughtered early, after 18 days of dosing. The animal was sacrificed 6.5 hours after the last dose. Residues for cow 9 were: muscle < 0.005 + 0.05 = 0.06 mg/kg, liver 0.03+0.99 = 1.02 mg/kg, kidney 0.01+0.19 = 0.20 mg/kg and fat 0.02 + 0.05 = 0.07 mg/kg.

The muscle fat content was determined (chloroform extraction). The mean muscle fat content was 2.37% (range 0.87–4.55%) and was similar for all animals.

Table 116 Summary of mean milk residues (BF421 2; expressed as parent equivalents) measured using method 987

Day of dosing	Mean BF421-2 residues in milk (mg/kg)		
	2.0 ppm	6.0 ppm	20 ppm
-1	< 0.0018	< 0.0018	< 0.0018
1	< 0.0018	0.002	0.009
3	0.003	0.007	0.029
5	0.004	0.005	0.021
7	0.005	0.014	0.043
10	0.007	0.009	0.028
14	0.003	0.010	0.041
17	0.004	0.010	0.039
21	0.006	0.013	0.035
24	0.005	0.012	0.035
28	0.006	0.009	0.035
29	-	-	0.030
30	-	-	0.018
32	-	-	0.005
34	-	-	< 0.0018
35	-	-	< 0.0018
Skim Milk	0.006	0.016	0.046
Cream	< 0.0018	< 0.0018	< 0.0018

Table 117. Summary of residue levels in tissues (BF421 2; expressed as parent equivalents) measured using method 987

Dose Group		Mean and maximum individual BF421-2 residues in tissues, (mg/kg)			
		Muscle	Liver	Kidney	Fat
2.0 ppm	Mean	0.01	0.19	0.05	0.02
	Max	0.02	0.24	0.07	0.02
6.0 ppm [A]	Mean	0.03	0.46	0.13	0.04
	Max	0.04	0.47	0.15	0.04
20 ppm	Mean	0.13	1.89	0.42	0.13
	Max	0.16	2.33	0.49	0.16
20 (2 days withdrawal)	Mean				
	Max	0.04	0.66	0.19	0.03
20 (7 days withdrawal)	Mean				
	Max	< 0.009	0.08	0.02	< 0.009

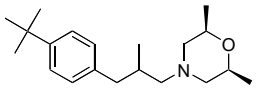
[A] results for cow sacrificed 6.5 hours after the last of 18 days of dosing were not included. The residues for this cow were: muscle 0.03, fat 0.03, liver 0.90, kidney 0.09 mg/kg.

APPRAISAL

Fenpropimorph is a systemic morpholine fungicide for the control of various diseases primarily in cereals but also finds use in controlling Sigatoka diseases in bananas. It acts by inhibiting the sterol pathway of fungus. The ADI for fenpropimorph was re-established as 0–0.004 mg/kg bw in 2016 and ARfDs of 0.1 mg/kg bw for women of child-bearing age and 0.4 mg/kg bw for the general population established. At the 47th Session of the CCPR (2015), it was scheduled for the evaluation of residues by 2017 JMPR under the periodic review program of CCPR.

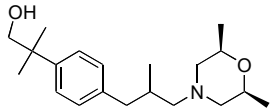
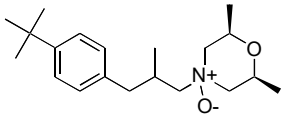
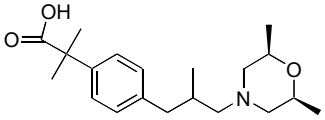
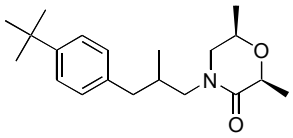
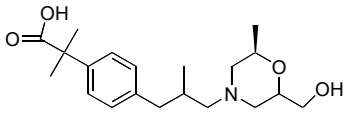
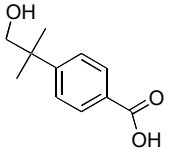
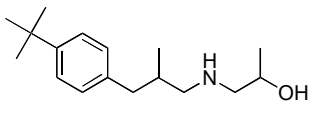
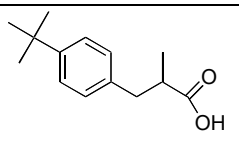
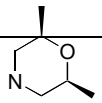
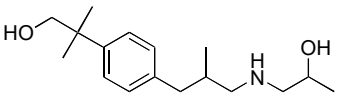
The Meeting received information on the metabolism of fenpropimorph in lactating goats and laying hens, banana, sugar beet, barley and wheat, follow crops, methods of residue analysis, freezer storage stability, GAP information, supervised residue trials on banana, sugar beet, barley, oats and wheat as well as a livestock feeding study (lactating cow).

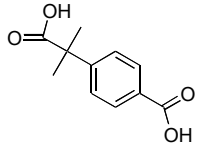
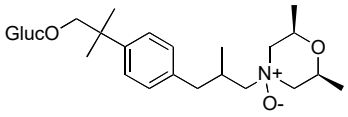
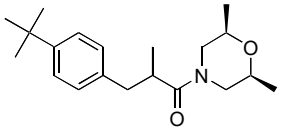
Fenpropimorph is (±)-*cis*-4-[3-(4-*tert*-butylphenyl)-2-methylpropyl]-2,6-dimethylmorpholine.



Fenpropimorph is a racemate, that is it consists of equal amounts of two enantiomers.

Metabolites referred to in the appraisal are addressed by their company codes:

Code	Structure	Code	Structure
BF421-1		BF421-14	
BF421-2		BF421-15	
BF421-3		BF421-16	
BF421-7		BF421-18	
BF421-10		BF421-20	

Code	Structure	Code	Structure
BF421-12		BF421-36	
BF421-13			

Studies on the metabolism in plants and livestock and environmental fate utilised [phenyl U-^{14}C]-fenpropimorph = P-label, [morpholine-2,(6)-^{14}C]-fenpropimorph = M1-label, [morpholine-2,6-^{14}C]-fenpropimorph =M2-label, and [benzylic-^{14}C]-fenpropimorph = B-label.

Plant metabolism

The Meeting received plant metabolism studies with fenpropimorph following foliar applications to banana, sugar beet, barley and wheat.

Banana

The metabolism of [^{14}C]-fenpropimorph, labelled in the morpholine (M2-label) or phenyl (P-label) rings, in banana plants was studied in plants grown outdoors following four foliar sprays at 0.9 kg ai/ha at intervals of 14, 51 and 12 days. The experiment included plants where bunches were protected (bagged) after the first spray and those where bunches remained unprotected (unbagged). Fruits are unripe at harvest and a sample of fruit was ripened using acetylene.

TRRs found in whole fruit at harvest (0 days after the last application) were higher in unbagged (0.09–0.67 mg eq/kg) compared to bagged bunches (0.025–0.35 mg eq/kg). TRR was also higher in experiments using the M2-label compared to the P-label.

In whole fruit, methanol extracted ^{14}C ranged from 27 to 83% TRR for the M2-label, whereas the corresponding values varied between 49 to 88% TRR for the P-label.

Fenpropimorph accounted for 3.2 to 16% of the TRR in whole fruit from the M2-label experiments, with sugars the only other major component identified (accounting for 9.7 to 76% TRR, the higher levels occurring in ripened fruit). For the P-label, fenpropimorph accounted for 14 to 60% of the TRR in whole fruit with no other component identified. Levels were too low to permit identification of ^{14}C in PES.

In summary, metabolism of ^{14}C-fenpropimorph in banana proceeds with hydroxylation of the *t*-butyl at the phenyl ring, followed by glucosidation. The morpholine ring can be degraded with its C_1 fragments utilised during assimilation processes in the plant for the biosynthesis of carbohydrates.

Sugar beet

Two studies were made available on the metabolism of ^{14}C-fenpropimorph in sugar beet.

In the first, two foliar applications of an EC formulation of ^{14}C-fenpropimorph labelled in the morpholine ring (M1-label) were made to sugar beet in a glasshouse at 0.15 kg ai/ha at BBCH 39 and at 32 days before harvest. TRRs were lower in roots (0.026 to 0.034 mg eq/kg) and the higher in leaves (0.596 to 1.885 mg eq/kg) demonstrating limited translocation from treated leaves into the roots.

Methanol extracted >70% of the ^{14}C in roots (70–79% TRR) and >79% from leaves (79–92% TRR).

In roots, other than natural products, the major component identified was fenpropimorph, comprising 40% TRR at 0 days and 14% TRR at 32 days after the second application. In leaves, fenpropimorph accounted for 14–15% TRR. Three metabolites were identified; BF421-1 glucoside (max 10% TRR), BF421-1 diglucoside (max 15% TRR) and BF421-1 glucoside sulphate (maximum 22% TRR). Glucose accounted for 34–56% TRR in roots and 4.3–9% TRR in leaves. A small amount of ^{14}C in leaves was associated with other natural products such as cellulose and lignins.

The second study utilised EC formulations of ^{14}C-fenpropimorph labelled in either the morpholine or phenyl rings. A single application was made to sugar beet maintained in a glasshouse at 0.75 kg ai/ha with harvest 112 days after treatment. The highest levels of TRRs were found in sugar beet plants sampled on the day of application (55.2 mg eq/kg P-label, 47.2 mg eq/kg M1-label). The TRR values for sugar beet leaves harvested 112 DALA amounted to 0.97 mg eq/kg for the P-label or 0.52 mg eq/kg for the M1-label. In sugar beet root, the ^{14}C levels were much lower, accounting for 0.03 mg eq/kg (P-label) to 0.04 mg eq/kg (M-label).

The extractability of ^{14}C with methanol was high for sugar beet leaves (89–93% TRR) and quite good for sugar beet root (64–76% TRR).

The major components identified in roots were fenpropimorph (17–34% TRR), BF421-20 glucoside (11% TRR), BF421-1 diglucoside (15% TRR) and sugars (23% TRR). In leaves the major components were fenpropimorph (5–19% TRR), BF421-14 (7–10% TRR), BF421-20 glucoside 10–18% TRR), BF421-36 glucoside (19–25% TRR) and BF421-10 (11% TRR).

In summary, for sugar beet the major degradation reactions were N oxidation of the morpholine ring, hydroxylation at the *t*-butyl moiety and subsequent conjugation reactions (glucosylations and malonylation), cleavage (detachment of the morpholine ring) or decomposition of the morpholine ring system to form hydroxypropylamine derivatives.

Barley

The metabolic fate of [^{14}C]-fenpropimorph (B-label) in barley plants maintained in a greenhouse was examined following topical application to selected leaves when plants were at the five leaf tillering stage. The application rate was equivalent to 0.9 kg ai/ha. Fenpropimorph accounted for 62% of the applied radioactivity on treated leaves at day 0, declining to 10% of applied radioactivity at day 20 after application with surface wash accounting for most of the fenpropimorph recovered (7.8%TRR). Limited translocation of radioactivity occurred to other plant parts. Fenpropimorph is a significant component of the residue in barley leaves, declining as a proportion of the total residues with increasing time after application.

Wheat

Three studies were made available on the metabolism of ^{14}C-fenpropimorph in wheat.

The metabolism of [^{14}C]-fenpropimorph in wheat grown in an open greenhouse was studied following application of the M2-label to plants at 1.3 kg ai/ha 55 days after seeding. At harvest, 84 days after treatment, TRR in grain and straw were 0.43 and 12 mg eq/kg respectively.

Methanol extracted 9% TRR in grain, 62% TRR in straw and 79–87% TRR in forage.

Extracted residues in grain were too low to characterise while residues unextracted by methanol were identified as starch 49% TRR, protein 16% TRR and polysaccharides 5% TRR.

Fenpropimorph was the major component of the residue in forage (38–47% TRR) and straw (22% TRR) with BF421-1 (7–8% TRR) and BF421-7 (5–26% TRR) the other significant components.

In a separate study with ^{14}C-fenpropimorph labelled in either the morpholine (M1-label) or phenyl (P-label) rings, wheat was treated at 0.75 kg ai/ha five weeks after sowing with harvest 56–57 days later.

Methanol extracted 52–97% TRR from forage, 56–61% TRR from straw and 12–13% TRR from grain.

TRR in methanol extracts of grain were too low to permit their characterisation. Only small amounts of ^{14}C were incorporated into polysaccharides and proteins (<10% TRR) with the majority associated with starch (31–32% TRR).

Fenpropimorph was the main component of the residue found in forage 21 days after application at 16–27% TRR, with no other single component accounting for more than 4.9% TRR.

Fenpropimorph was also the major component in straw at 20–24% TRR with BF421-2 accounting for up to 7.0% TRR. The largest individual unidentified component accounted for 7 to 12% TRR. About 12% TRR in straw was associated with lignin fractions.

In the third and most comprehensive study, two foliar applications of ^{14}C-fenpropimorph were made to wheat, each at 0.75 kg ai/ha. Fenpropimorph was labelled in the morpholine (M1-label) or phenyl (P-label) rings. Harvest was 49 days after the last application.

TRRs in hay 25 DALA were 25–48 mg eq/kg. At harvest, 49 DALA, levels of ^{14}C in grain and straw were 0.13–0.34 and 9.6–15 mg eq/kg respectively.

The extractability of ^{14}C with methanol depended on the matrix under investigation and was high for wheat hay (83–83% TRR) and moderate for wheat straw (67–71% TRR). From the wheat grain, 15% TRR (M1-label) and 35% TRR (P-label) were extracted with methanol.

For the M1-label experiment, major identified components in grain extracts were the parent compound (1.7% TRR) and its N oxide (BF421-14, 0.6% TRR) as well as 2,6-DMM (BF421-10, 0.6% TRR). Additional components were minor amounts of the BF421-1 malonylglucoside (0.3% TRR) and the non-separated metabolites BF421-1 diglucoside and/or BF421-1 glucoside and/or BF421-20 (0.3% TRR). Sugar, predominantly fructose, accounted for 6.5% TRR.

In grain extracts (P-label), the major components were fenpropimorph (16% TRR) and its N oxide (BF421-14, 4.1% TRR) together with BF421-1 malonylglucoside (4.7% TRR), and the non-separated metabolites BF421-1 diglucoside and/or BF421-1 glucoside and/or BF421-20 (3.2% TRR).

Residues remaining in grain solids after methanol extraction, 85% TRR for M1-label and 65% TRR for the P-label, were further characterised using enzymes, acid and base, which released an additional 56% TRR from the M1-label solids, mostly as sugars, and 40% from the P-label.

In hay extracts from the M1-label experiment the major components identified were BF421-1 malonylglucoside (20% TRR), parent compound (21% TRR) and its N oxide (BF421-14, 14% TRR), 2,6-dimethylmorpholine (BF421-10; 6.5% TRR) and the non-separated metabolites BF421-1 diglucoside and/or BF421-1 glucoside and/or BF421 20 (7.2% TRR).

In hay (P-label) the major components in extracts were parent compound (20% TRR) and its N oxide (BF421-14, 14% TRR) and BF421-1 malonylglucoside (22% TRR). A component that represented the metabolites BF421-1 diglucoside and/or BF421-1 glucoside and/or BF421-20 accounted for 7.9% TRR.

Similar metabolic profiles to hay were obtained for straw extracts. The M1-label straw extracts contained BF421-1 malonylglucoside (16% TRR), fenpropimorph (7.1% TRR) and its N oxide (BF421-14, 14% TRR), BF421-10 (6.4% TRR) and the non-separated metabolites BF421-1 diglucoside and/or BF421-1 glucoside and/or BF421-20 (6.7% TRR).

The major components in the P-label experiment were BF421-1 malonylglucoside (15% TRR), the parent compound (6.9% TRR) and its N oxide (BF421-14, 12% TRR) and the non-separated metabolites BF421-1 diglucoside and/or BF421-1 glucoside and/or BF421-20 (7.8% TRR).

The solids after methanol extraction of hay accounted for 17% TRR for the M1-label or 17% TRR for the P-label, respectively. In straw, the ^{14}C in solids after extraction were 29% TRR for the M1-label or 33% TRR for the P-label. More than 90% of the ^{14}C in the solids was released by sequential solubilisation treatments, which included aqueous ammonia, Macerozyme and cellulase,

amylases and amyloglucosidase, tyrosinase and laccase, or reflux with NaOH. The more severe treatments released additional portions of the more polar metabolites (particularly of glucoside conjugates and of BF421-10), hydroxylated metabolite BF421-1 and BF421-10 (M1-label only) and in some cases minor amounts of the parent compound and the metabolite BF421-7. Considerable amounts of the ^{14}C in solids after methanol extraction consisted of fenpropimorph or its degradation products associated with or embedded/incorporated in insoluble natural polymers (starch and cell wall components like hemicelluloses, lignin, lignin-carbohydrate complexes or pectin).

In summary, the metabolism of fenpropimorph by plants is well understood consisting of oxidative processes affecting the morpholine ring, the tertiary-butyl side chain and the methyl-propyl bridge. In addition, cleavage of the morpholine bond and glucose conjugation of a number of metabolites was also observed. A number of metabolites were not observed in laboratory animal (rat) studies. Plant specific metabolites were BF421-13 (wheat forage/straw), BF421-14 (sugar beet leaves, wheat forage/straw/grain), BF421-15 (wheat forage/straw) and BF421-36 (sugar beet leaves, wheat straw).

The possible impact of plant metabolism on the isomer ratio of fenpropimorph indicates slight enrichment of the (-)-enantiomer over the (+)-enantiomer.

Animal metabolism

Metabolism studies were made available to the meeting for lactating goats and laying hens dosed with fenpropimorph.

Lactating goats were orally dosed by intubation once daily for five consecutive days with [^{14}C]-fenpropimorph at a dose equivalent to 2336 ppm in the diet for the P-label or 1421 ppm in the diet for the M2-label.

By five hours after the last dose, the majority of the ^{14}C residues was recovered in the excreta (M2-label; urine 21% administered dose (AD), faeces 29% AD: P-label; urine 14% AD, faeces 20% AD). For tissues, ^{14}C residues were highest in kidney, (53 M2-label, 233 P-label mg eq/kg) and liver (124 M2-label, 141 P-label mg eq/kg) with muscle (6.3 M2-label, 8.9 P-label mg eq/kg) and fat (19 M2-label, 4.3 P-label mg eq/kg) containing low residues. TRR in milk reached 23 (M2-label) and 9.7 (P-label) mg equivalents/kg before the end of dosing. Overall accountability of the administered dose was poor at 55% for the M2-label and 40% for the P-label.

The whey fraction of milk from the M-label experiment contained 60% TRR (9.8% *n*-hexane soluble; 7.5% CH_2Cl_2 soluble; 43% TRR water-soluble) with the protein fraction accounting for 33% TRR (4.7%TRR CH_3OH soluble). Radioactivity in milk from the P-label experiment was predominantly found in the whey fraction, at 76% TRR with the remainder in the protein fraction.

Extractability of kidney, liver and muscle tissues with methanol was high at >83% TRR.

Parent fenpropimorph was not detected in any tissue or in milk. The major components of the ^{14}C in tissues and milk were BF421-2 (11–68% TRR) and BF421-1 (25–26% TRR).

In an additional study, lactating goats were orally dosed by gavage once daily for seven consecutive days with [^{14}C]-fenpropimorph at a dose equivalent to 13 ppm in the diet for the M1-label or 12 ppm in the diet for the P-label.

By 23 hours after the last dose, the majority of the ^{14}C residues were recovered in the excreta (M1-label; urine 24% AD, faeces 43% AD: P-label; urine 26% AD, faeces 50% AD). For tissues, ^{14}C residues were highest in liver (0.78 M1-label, 0.64 P-label mg eq/kg), fat (0.24 M1-label, 0.19 P-label mg eq/kg) and kidney, (0.26 M1-label, 0.15 P-label mg eq/kg) with muscle (0.06 M1-label, 0.02 P-label mg eq/kg) containing low residues.

Residues in milk appeared to reach plateau levels of 0.16 M1-label and 0.016 P-label mg eq/kg by day four of dosing with significant differences in ^{14}C levels between milk collected in the morning prior to dosing compared to evening milk, suggesting fenpropimorph residues are rapidly eliminated following dosing. The rapid elimination was confirmed by ^{14}C in plasma, which peaked at

1–6 hours after last dosing and thereafter declined with a half-life of 14–18 hours (M1-label) and 43–67 hours (P-label).

Extractability was high with > 70% TRR in kidney, liver and muscle samples extracted with methanol (71–78% M1-label, ≥ 93% P-label). For fat, methanol extracted 49% (M1-label) to 57% TRR (P-label), with iso-hexane extracting an additional 19% (P-label) to 21% (M1-label) TRR. In the case of milk, acetone/CH_3CN extracted 22% (M1-label) to 60% TRR (P-label), with methanol extracting an additional 9.7% (M1-label) to 17% (P-label) TRR.

In solvent extracts of milk, fenpropimorph (1.4–14% TRR) and the metabolites BF421-2 (2.9–27% TRR) and BF421-3 (1.1–10% TRR) were the main components identified. For the M1-label experiment, lactose accounted for up to 19% TRR with an additional 41% TRR associated with metabolites derived from 2,6-DMM. The protein precipitate accounted for 22–33% TRR (M1 and P-labels).

In liver extracts, the predominant components identified were BF421-2 (57–73% TRR) together with smaller amounts of BF421-3 (5.1–6.4% TRR), BF421-1 (1.9–10% TRR) and fenpropimorph (2.3–6.9% TRR).

The main components identified in kidney extracts were BF421-2 (25–40% TRR) and BF421-3 (21–40% TRR) and fenpropimorph (10–15% TRR). The metabolites BF421-10 (1.7% TRR) and BF421-19 (5.0% TRR) were identified exclusively in M1-label extract.

The main radioactive residue in muscle extract was BF421-2 (30–79% TRR) while components identified in fat extracts were mainly BF421-2 (29–34% TRR), BF421-2 glucuronide (14–15% TRR) and fenpropimorph (2.9–14% TRR).

The majority of ^{14}C unextracted with solvent was released on treatment of solids with pronase or acid suggesting incorporation into natural products.

Two studies on laying hens were made available to the Meeting.

In the first laying hens were orally dosed by intubation once a day for five doses with [^{14}C]-fenpropimorph at a dose equivalent to 21 ppm (M2-label) or 56 ppm (P-label) in the feed.

Excretion of fenpropimorph was fast, with 79% AD (M2-label) and 83% AD (P-label) found in the excreta by 5 hours after the last dose.

TRR for the M2-label were greatest in liver (3.9 mg eq/kg), egg yolk (3.0 mg eq/kg) and kidneys (2.4 mg eq/kg) with lower levels in the fat (1.1 mg eq/kg) and muscle (0.34 mg eq/kg). For hens dosed with the P-label, TRR were highest in liver (2.8 mg eq/kg) and kidneys (2.8 mg eq/kg), followed by fat (1.4 mg eq/kg), muscle (0.42 mg eq/kg) and eggs (0.39 mg eq/kg). 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, a pattern consistent with the physiology of egg formation.

Extractability with solvents and water was high at 87% for liver and kidney (80% methanol, methanol, acetone), 97% muscle (80% methanol, methanol, Soxhlet), 99% fat (methanol, ethyl acetate) and > 62% egg white and yolks (acetone).

In liver, fenpropimorph was extensively metabolised with up to 10 metabolites identified in the organosoluble extracts but no parent compound. Fenpropimorph was detected in kidney for birds dosed with the P-label. Metabolites identified were BF421-1 (plasma), BF421-2 (plasma, liver 3.9% TRR and kidney 3.5% TRR), and BF421-3 (kidney 1.9% TRR).

In a more recent study, laying hens were orally dosed once a day for ten days with [^{14}C]-fenpropimorph at a dose equivalent to 12–14 ppm in the feed (M1-label or P-label).

Radioactivity recovered from excreta together with cage wash amounted to 85% and 106% AD for the M1-label and the P-label, respectively.

The TRR in tissues were 0.097–0.63 mg eq/kg for the M1-label and 0.036–0.34 mg eq/kg for the P-label with highest residues in liver, fat, and lowest residues in muscle. In eggs, by the end of dosing the TRRs were 0.64 mg eq/kg and 0.11 mg eq/kg, for the M1- and P-label, respectively.

Extractability with methanol was generally low to moderate, at 18–56% TRR depending on label and tissue type. Subsequent extraction with iso-hexane was especially successful for fat for both labels (extracting 60–83% TRR), for the P-label muscle (66% TRR) and the M1-label egg (45% TRR). The overall extracted radioactivity using methanol + iso-hexane ranged from 46 to 101% TRR in edible matrices for the M1-label, and from 63 to 92% TRR for the P-label.

Parent fenpropimorph was a major component in eggs, fat and liver of the P-label, accounting for 18, 18 and 29% TRR respectively, whereas in the M1-label tissues fenpropimorph accounted for only 2.7% TRR in egg, 13% TRR in fat and 11% TRR in liver. In muscle, fenpropimorph represented 5.1% TRR (M1-label) and 5.5% TRR (P-label). Other compounds identified were the acid metabolite BF421-2 (eggs, muscle and liver) which was a minor component in all tissues for the M1-label (egg 1.8% TRR; muscle 0.5% TRR; liver: 1.8% TRR), but accounting for 16% TRR in eggs, 3.4% TRR in muscle and 7.5% TRR in liver for the P-label. BF421-3 was also identified in hens in amounts similar to BF421-2, representing 13%, 2.9% and 8.1% TRR in eggs, muscle and liver of the P-label and 0.8%, 1.4% and 1.3% TRR in the respective matrices of the M1-label.

Of the M1-label specific metabolites that could be unambiguously identified, BF421-10 was found in eggs (1.5% TRR), muscle (3.4% TRR) and liver (3.3% TRR). By far the most predominant metabolites identified in the M1-label experiment were lipids in fat (79.5% TRR) and egg (45.2% TRR) with lower levels in muscle (7.6% TRR) and liver (9.8% TRR). These lipid metabolites were identified after a saponification/esterification reaction via their fatty acid methyl esters to represent triacyl glycerides primarily containing the most abundant endogenous fatty acids in hens, palmitic, oleic, and stearic acid.

The P-label specific metabolites BF421-12 and BF421-16, were identified in eggs, muscle and fat, accounting for 5.1%, 2.4% and 4.5% of the TRR. Another metabolite exclusively found in the P-label tissues was BF421-18, which was detected in the muscle in free form (4.5% TRR) and identified in all tissues as its ethyl ester after saponification/esterification, indicating an incorporation into/conjugation with endogenous lipids. Radioactive triacyl glycerides represented the major metabolites in muscle and fat, accounting for 53% and 52% TRR. They were also detected in eggs and liver, at 9.5% and 1.8% TRR, respectively.

The ^{14}C unextracted with solvent of all matrices except fat contained considerable amounts of radioactivity and were subjected to enzymatic digestion with pronase which solubilised 82 to 100% of the unextracted radioactivity. Analysis of the ^{14}C liberated by pronase treatment allowed the identification of minor amounts of parent fenpropimorph, and metabolites BF421-3, BF421-12 and BF421-16 in the egg and the identification of BF421-2 and BF421-3 in the liver of the P-label experiment. Solubilised residues of the M1-label egg, muscle and liver could not be unambiguously identified, however they were comprised of numerous compounds, presumably small polar molecules, and it is likely that a considerable portion consists of endogenous molecules.

In summary, the metabolism of fenpropimorph in lactating goats and laying hens is similar to metabolism in laboratory animals. A number of metabolites were observed in livestock but not reported in studies on laboratory animals (rats): BF421-12 (hen), BF421-18 (hen), BF421-19 (goat), BF421-21 (goat), BF421-22 (goat), BF421-26 (goat) and BF421-30 (goat). These metabolites are structurally related to the parent compound and resulted from metabolic pathways that are similar to those observed in the rat.

No information was available on the possibility for stereo-selective metabolism in livestock.

Environmental fate

The Meeting received information on soil aerobic metabolism, aqueous photolysis and aqueous hydrolysis properties of [^{14}C]-fenpropimorph. Studies were also received on the behaviour of [^{14}C]-fenpropimorph in a rotational crop situation.

The degradation of fenpropimorph in soil maintained under aerobic conditions is rapid with fenpropimorph acid (BF421-2) and BF421-10 the major degradation products formed. While parent fenpropimorph is degraded moderately fast in soils, the degradates formed can be classified as non-persistent to moderately persistent. In the laboratory studies, normalised soil DT_{50} values (20 °C pF2) for parent fenpropimorph ranged from 9.4–134 days (geometric mean 16.2 days) while for fenpropimorph acid (BF421-2) normalised DT_{50} values ranged from 3.4 to 8.9 days (geometric mean 4.6 days) and 149 days for BF421-10. In field studies an additional degradate, BF421-7 was observed for which a DT_{50} value of 82 days was estimated, consistent with laboratory studies for this metabolite for which DT_{50} values ranged from 2.5 to 209 days.

Fenpropimorph was stable to hydrolysis in aqueous solutions at pH 3 to pH 9 suggesting hydrolysis plays a negligible role in its degradation.

The soil photolysis of fenpropimorph in loamy sand soil was investigated. The half-life for degradation was estimated to be 30 days with main degradates detected BF421-13 and BF421-15. Photochemical degradation is not expected to contribute significantly to the degradation of fenpropimorph in the environment

In a confined rotational crop study with lettuce, radish and wheat, bare loamy sand soil was treated with [^{14}C]-fenpropimorph (P- and M1-labels) at the equivalent of 1.5 kg ai/ha and crops sown 30, 120 and 365 days after the soil application. Lettuce was sampled at maturity (50–56 DAA), radish was sampled at 64–76 DAA, wheat forage at 56–60 DAA and straw, chaff and grain at 95–116 DAA.

The TRRs in lettuce head were highest in the samples from the 30 days PBI (0.06–0.13 mg eq/kg) declining for subsequent plant back periods. TRRs in radish leaves (30 PBI: 0.03–0.23 mg eq/kg,) were higher than in radish roots (30 PBI: 0.02–0.04 mg eq/kg). In both matrices and both labels, the TRRs were lower for the longer PBIs. In wheat forage residues (highest 30 PBI 0.13–0.65 mg eq/kg) declined for the longer PBIs. In wheat straw and chaff, the TRRs were higher (0.36–1.2 mg eq/kg, straw) in the samples of the 30 days PBI and were 0.16–0.36 mg eq/kg for the 365 days PBI. In wheat grain TRRs at the different PBIs followed the order 120 > 30 > 365 days for the M1-label and 365 > 120 > 230 days for the P-label.

In addition to BF421-1 (free and conjugated) and BF421-10, other compounds identified were natural sugars like glucose, fructose and saccharose as well as starch.

Residues above 0.01 mg/kg were observed in the M1-label experiment at the 30-day PBI for BF421-10 (0.06 mg/kg lettuce, 0.1 mg/kg radish leaf, 0.011 mg/kg radish root, 0.42 mg/kg wheat forage, 0.58 mg/kg straw and 0.14 mg/kg chaff) and BF421-1 free and conjugated (0.016 mg/kg wheat forage, 0.031 mg/kg straw), at the 120-day PBI for BF421-10 (0.02 mg/kg lettuce, 0.03 mg/kg radish leaf, 0.04 mg/kg wheat forage, 0.13 mg/kg straw, 0.27 mg/kg chaff) and the 365-day PBI for BF421-10 (0.02 mg/kg wheat forage, 0.09 mg/kg straw and 0.02 mg/kg chaff).

In the P-label experiment fenpropimorph-related components above 0.01 mg/kg at the 30 day PBI were fenpropimorph (0.02 mg/kg lettuce, 0.05 mg/kg wheat forage) and BF421-1 free and conjugated (0.01 mg/kg lettuce, 0.06 mg/kg wheat forage, 0.28 mg/kg straw, 0.05 mg/kg chaff). No components other than natural products were above 0.01 mg/kg in the 120-day or 365-day PBI experiments.

Comparison with primary metabolism studies shows that the pathway in rotational crops is consistent with that in primary crops.

Noting that in the rotational fenpropimorph application was to bare soil and in practice a crop would be present to intercept most of the active ingredient together with the low levels detected, the Meeting concluded that, with the exception of BF421-10, fenpropimorph and related residues are unlikely to be observed in rotational crops.

Methods of analysis

The methods all involve homogenisation followed by extraction of the homogenised samples with an organic/aqueous solvent mixture, typically CH_3OH. The main differences between methods involve

clean-up conditions and instrumentation for quantification (GC-N-FID, GC-ECD, GC-NPD, GC-MS, HPLC-UV, LC-MS/MS). In plant commodities, fenpropimorph is the analyte determined while in animal commodities in addition to fenpropimorph, BF421-2 may also be determined. The LOQs for plant commodities are typically 0.01–0.05 mg/kg for fenpropimorph while for animal commodities they are 0.005 to 0.01 mg/kg for tissues and eggs and 0.001–0.002 mg/kg for milk for fenpropimorph and BF421-2.

Radiovalidation studies supported the use of CH_3OH for the extraction of residues.

Multi-residue methods are currently available for fenpropimorph in both plant and animal matrices.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the stability of fenpropimorph and BF421-2 (fenpropimorph acid) in various matrices on freezer storage (-18 °C).

The periods of demonstrated stability cover the frozen storage intervals used in the residue studies on crops.

Fenpropimorph is stable in dry commodities (wheat grain) and high starch commodities (sugar beet root), high water content commodities (banana, wheat forage, sugar beet tops), high oil content commodities (sunflower seed) and wheat straw for at least 723 days.

Fenpropimorph is stable in muscle for 760 days, and at least 880 days in fat, kidney, liver, milk and cream. Fenpropimorph acid (BF421-2) is stable in liver for 760 days and muscle, fat, kidney, milk and cream for at least 880 days.

The demonstrated frozen storage stability intervals encompass the storage intervals encountered in the supervised residue trials.

Definition of the residue

The fate of fenpropimorph was investigated after foliar application to banana, sugar beet, barley and wheat plants. Fenpropimorph was degraded in all crops to a number of metabolites and their conjugates.

In edible commodities, fenpropimorph was a significant component of the radioactive residue, cereals (barley, wheat grain 2–16% TRR), root and tuber vegetables (sugar beet roots 17–34% and leaves 5–19% TRR) and tropical fruit (banana 3–60% TRR). Other significant components were BF421-36 glucoside in beet leaves (19–25% TRR), BF421-10 in beet leaves (11% TRR), BF421-20 glucoside in beet roots (11% TRR) and leaves (10–18% TRR), BF421-14 in grain (0.6–4% TRR) and beet leaves (7–10% TRR) and BF421-1 glucoside in beet leaves (10% TRR).

In livestock feeds, fenpropimorph was the major component of the residue in wheat forage (16–47% TRR). Apart from incorporation into natural products, BF421-1 and its conjugates were the most abundant components of the ^{14}C residue in wheat hay and straw (8–36% TRR) together with BF421-14 (12–14% TRR), BF421-7 (1–26% TRR) and BF421-10 (3–12% TRR).

With the exception of BF421-10, residues derived from fenpropimorph are unlikely to occur in rotational (follow) crops.

Fenpropimorph was the only compound that is a significant residue in most commodities and validated analytical methods are available for its determination.

The Meeting decided the residue definition for compliance with MRLs in plants should be fenpropimorph.

In deciding which additional compounds should be included in the residue definition for risk assessment the Meeting considered the likely occurrence of the compounds and the toxicological properties of the candidates BF421-1 (free and conjugated), BF421-7, BF421-10, BF421-14, BF421-20 conjugates and BF421-36 conjugates.

The metabolites fenpropimorph alcohol (BF 421-1 free and conjugated) and BF 421-10 are major rat metabolites and their toxicity is therefore covered by the toxicological properties of the parent compound. Testing did not reveal evidence that BF421-10 is genotoxic. For BF421-14 (fenpropimorph N-oxide) no structural alerts indicating genotoxicity were identified with the structure-activity relationship models OASIS-TIMES and VEGA.

The other plant metabolites were shown to be structurally related to the parent compound and resulted from metabolic pathways that are similar to those observed in the rat. It is considered that these metabolites are of no greater potency compared to the parent compound.

The levels of metabolites were considered in the edible commodities banana, cereal grain and young sugar beet leaves (32 DALA). Residues of BF421-1 (free and conjugated) were only present at greater than 0.01 mg/kg in sugar beet leaves. BF421-7 was only detected in grain at 49 DALA but only at levels below 0.01 mg/kg. BF421-10 and BF421-14 were detected in grain 49 DALA but also at levels below 0.01 mg/kg. BF421-20 glucoside was detected in beet roots at levels below 0.01 mg/kg. BF421-36 glucoside was not detected in commodities suitable for human consumption considered by the current Meeting.

It was noted that the typical PHIs for the approved use patterns made available to the Meeting were sometimes considerably shorter than the interval between last application and sampling in the metabolism studies, which could result in over-emphasising the relative importance of metabolites:

- Cereals (barley, rye, oats, triticale, wheat): PHI 35 days, range 28–56 days versus 49–84 days for metabolism, with the critical study samples at 49 days for grain and straw.
- Sugar beet: 35 days (range 21–46 days) versus 32 to 112 days with the critical metabolism study samples at 112 days.

Noting the above the Meeting considered only fenpropimorph and BF421-1 (free and conjugated) and BF421-10 would make significant contributions to the overall toxicological burden.

It is possible BF421-10 is not a metabolite unique to fenpropimorph as other morpholine fungicides such as aldimorph, dodemorph and tridemorph also contain the 2,6-dimethylmorpholine moiety, though their structures suggest limited potential for release of intact BF421-10 as cleavage of an amine alkyl bond would be required.

The Meeting decided the residue definition for estimation of dietary intake in plants should be the sum of fenpropimorph, BF421-1 (free and conjugated) and BF421-10 (2,6-DMM).

To facilitate estimation of exposure, a conversion factor could be used to provide a conservative estimate of the sum based solely on residues of fenpropimorph. Based on the available metabolism studies with the M-label, appropriate conversion factors (CF = [fenpropimorph+BF421-1 free and conjugated + BF421-10]/fenpropimorph) are banana 1 (metabolism sample 0 DALA), cereal grains 1.7 (44 DALA), sugar beet roots 1.3 (32 DALA) and beet leaves 3.6 (32 DALA).

For livestock dietary burden it is noted that the main residue in livestock is BF421-2 (fenpropimorph acid) and that both fenpropimorph and BF421-1 (fenpropimorph alcohol) could be transformed into BF421-2 by livestock. In considering livestock dietary burden, the sum of fenpropimorph and BF421-1(free and conjugated) residues needs to be considered. Residue trials did not measure BF421-1 and to enable a more reliable estimate of livestock dietary burden a conversion factor should also be employed (CF = [fenpropimorph+BF421-1 free and conjugated]/fenpropimorph). Based on the metabolism studies a factor of 6.15 (mean of 5.7, 6.6; 49 DALA) for straw and 1.95 (median of 1.1, 1.2, 2.7, 3.0; 21-28 DALA) for forage/hay, 1.5 (mean of 1.4, 1.6; 49 DALA) for grain, 3.6 (32 DALA) for beet tops and 1.3 (32 DALA) for beet roots should be used.

Livestock may be exposed to fenpropimorph derived residues present in feeds. The nature of fenpropimorph residues in commodities of animal origin was investigated in metabolism studies including two studies in lactating goats and two studies in laying hens. In the early studies on lactating goats and laying hens, sacrifice was 5 hours after the last dose while in the more recent studies

sacrifice was 23 hours after the last dose. The interval between last dose and sacrifice is expected to have a significant impact on the relative proportions of the metabolites detected in tissues whereas metabolite profiles for milk and eggs would be expected to be similar in the two studies.

In the more recent study on lactating goats with sacrifice 23 hours after the last dose, fenpropimorph was detected in all tissues and milk (as% TRR: milk 1.4–14%, kidney 10–15%, liver 2–7%, muscle 5% and fat 3–14%). The major components of the radioactive residues were BF421-2 free (as% TRR milk 2.9–27%, kidney 25–40%, liver 57–73%, muscle 30–79%, fat 29–34%) and conjugated (milk 0.9–3.7%, muscle 7% and fat 14–15%) and BF421-3 free and conjugated (milk 7–14% TRR, kidney 20–41% TRR, liver 6–6% TRR, muscle 6–22% TRR and fat 8–9% TRR).

The earlier study in laying hens with sacrifice at the more relevant 5 hours after the last dose did not detect fenpropimorph in tissues or eggs. The only metabolite identified was BF421-2 and then only in kidney. In the more recent study fenpropimorph was the major component identified (egg 2.7–18% TRR, liver 11–29% TRR, muscle 5% TRR, fat 13–18% TRR) with BF421-2 free and conjugated (egg 1.8–16% TRR, liver 1.8–8% TRR, muscle 0.5–3.4% TRR) and BF421-3 free and conjugated (egg 0.8–13% TRR, liver 1.3–8.1% TRR, muscle 1.4–2.9% TRR).

In livestock, fenpropimorph and BF421-2 comprised the major portion of the residue in tissues, milk and eggs and validated analytical methods are available for their determination.

The Meeting decided the residue definition for compliance with MRLs in livestock commodities should be the sum of fenpropimorph and BF421-2.

In deciding which additional compounds to fenpropimorph should be included in the residue definition for risk assessment the Meeting considered the likely occurrence of the compounds and the toxicological properties of the candidates BF421-2 (free and conjugated) and BF421-3 (free and conjugated).

The metabolites BF421-2 (free and conjugated) and BF421-3 (free and conjugated) are major rat metabolites and their toxicity is therefore covered by the toxicological properties of the parent compound.

As the contribution of the additional metabolites BF421-2 glucuronide and BF421-3 and its glucuronide to the overall consumer exposure would be small, the Meeting decided the definition for estimation of dietary exposure should be the sum of fenpropimorph and BF421-2.

In summary, based on the above the Meeting decided the residue definitions for compliance with MRLs and estimation of dietary intake should be as follows:

Definition of the residue for compliance with MRL for plant commodities: fenpropimorph

Definition of the residue estimation of dietary intake for plant commodities:

sum of fenpropimorph, fenpropimorph alcohol (free and conjugated) and BF421-10 (2,6-DMM), expressed as fenpropimorph.

Definition of the residue for compliance with MRL and estimation of dietary intake for animal commodities: sum of fenpropimorph and fenpropimorph acid (BF421-2) expressed as fenpropimorph

Residues (sum of fenpropimorph and BF421-2) in fat were more than 5× greater than muscle in the lactating goat and laying hen metabolism studies. However, in the livestock transfer study with lactating cows, residues in fat are only slightly greater than muscle and residues in cream are only about 2× residues in skim milk. On the weight of evidence, the Meeting decided residues measured as the sum of fenpropimorph and BF421-2 are not fat soluble.

The residue is not fat soluble.

Results of supervised residue trials on crops

Supervised residue trial data for were available for fenpropimorph on bananas, sugar beet, barley, oats, rye and wheat.

Banana

The Meeting received supervised residue trial data for fenpropimorph on banana from Brazil, Colombia, Costa Rica, Ecuador, Guatemala, Honduras, Martinique and Mexico. In Columbia critical GAP is applications at up to 0.6 kg ai/ha with a PHI 0 days. In trials approximating critical GAP in Columbia (0.6±25% kg ai/ha) residues in unbagged bananas were: (n=18): < 0.05, < 0.05, < 0.05, 0.07, 0.1, 0.12, 0.13, 0.16, 0.26, 0.32, 0.36, 0.43, 0.65, 0.7, 0.7, 0.8, 1.2, 1.4 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg for bananas, confirming its previous recommendation.

Residues in the edible portion (pulp) were (n=11): < 0.05, < 0.05, < 0.05, < 0.05, 0.06, 0.08, 0.14, 0.18, 0.28, 0.30, 0.43 mg/kg. The Meeting recommended an STMR and HR of 0.08 and 0.43 mg/kg respectively for banana (pulp).

The Meeting also noted that the STMR and HR are conservative as residues in bagged bananas, a common commercial practice, were lower than for unbagged bananas with residues in pulp for bagged bananas that ranged from < 0.05 to 0.2 mg/kg.

Sugar beet

In Poland critical GAP is a single application at 0.25 kg ai/ha at growth stage BBCH 39–49 with a 35 day PHI. In trials conducted in Europe on sugar beet the application rate (0.75 kg ai/ha) was higher than the critical GAP of Poland (0.25 kg ai/ha) and the Meeting agreed to utilise the proportionality approach (scaling factor 0.75/0.25=0.33) to estimate residues matching cGAP. Unscaled residues for sugar beet were (n=8) < 0.01 (7), 0.06 mg/kg. Residues below LOQ were not scaled. After scaling the following residues were obtained (n=8), < 0.01 (7) and 0.02 mg/kg.

The Meeting recommended a maximum residue level of 0.03 mg/kg to replace its previous recommendation of 0.05 * mg/kg and an STMR for dietary intake estimation and median residue for livestock feeds of 0.013 (1.3×0.01) mg/kg for sugar beet.

Barley

Supervised residue trial data for fenpropimorph on barley were made available. The critical GAP in Belgium is up to two applications at 0.75 kg ai/ha with a PHI of 28 days.

In trials conducted in Europe and Brazil approximating critical GAP in Belgium (0.75±25% kg ai/ha), residues in barley were (n=15): 0.01, 0.02, 0.02, 0.02, < 0.05, < 0.05, < 0.05, < 0.05, < 0.05, 0.07, 0.07, 0.07, 0.09, 0.11 and 0.11 mg/kg.

The Meeting recommended a maximum residue level of 0.2 mg/kg, an STMR of 0.085 (1.7×0.05) mg/kg for dietary intake estimation and a median residue of 0.075 (1.5×0.05) mg/kg for livestock dietary burden estimation for barley. Use patterns exist for oats and the Meeting decided to extrapolate the values to oats. The recommendations for barley and oats replace the previous recommendations of 0.5 mg/kg for these commodities.

Wheat

Supervised residue trial data for fenpropimorph on wheat were available. Critical GAP in Belgium is two applications at 0.75 kg ai/ha with a PHI of 28 days.

In trials conducted in Europe approximating critical GAP in Belgium residues in wheat grain were (n=7): < 0.01, < 0.01, < 0.01, < 0.01, 0.01, 0.02, and 0.04 mg/kg.

The Meeting recommended a maximum residue level of 0.07 mg/kg, an STMR of 0.017 (1.7×0.01) mg/kg for dietary intake estimation and a median residue of 0.015 (1.5×0.01) mg/kg for livestock dietary burden estimation for wheat. Use patterns exist for rye and triticale and the Meeting decided to extrapolate the values to rye and triticale.

The recommendations for rye, triticale and wheat replace the previous recommendations for these commodities.

Animal feedstuffs

Sugar beet tops

In Poland critical GAP is a single application at 0.25 kg ai/ha at growth stage BBCH 39–49 with a 35 day PHI. In trials conducted in Europe on sugar beet the application rate (0.75 kg ai/ha) was higher than the critical GAP of Poland (0.25 kg ai/ha) and the Meeting agreed to utilise the proportionality approach (scaling factor 0.31–0.35) to estimate residues matching cGAP. Unscaled residues for sugar beet tops were (n=8) 0.11, 0.15, 0.17, 0.2, 0.21, 0.22, 0.33 and 0.44 mg/kg. After scaling the following residues were obtained: 0.04, 0.05, 0.06, 0.06, 0.07, 0.07, 0.11 and 0.14 mg/kg. Sugar beet tops contain approximately 23% DM.

The Meeting noted that sugar beet tops are not a traded commodity and decided to withdraw its previous recommendation for fodder beet leaves and tops of 1 mg/kg. The Meeting also estimated a median residue of 0.367 (1.3×0.065/0.23) mg/kg and a highest residue of 0.848 (1.3×0.15/0.23) mg/kg (all on dry matter basis) for livestock dietary burden estimation for sugar beet leaves and tops.

Cereal straw

Supervised residue trial data for fenpropimorph on barley were made available. The critical GAP in Belgium is up to two applications at 0.75 kg ai/ha with a PHI of 28 days. Residues in barley straw from trials complying with critical GAP were (n=16): < 0.05, < 0.05, 0.03, 0.04, 0.04, 0.07, 0.08, 0.08, 0.1, 0.13, 0.14, 0.14, 0.15, 0.16, 0.17 and 0.39 mg/kg.

For wheat, the critical GAP in Belgium is up to two applications at 0.75 kg ai/ha with a PHI of 28 days. Residues in wheat straw in trials approximating cGAP were (n=7): 0.07, 0.07, 0.11, 0.12, 0.14, 0.21 and 0.34 mg/kg.

The Meeting noted that residues in barley and wheat straw are similar, confirmed by a Mann-Whitney U test, and decided to combine the data sets for mutual support. The combined data is: < 0.05, < 0.05, 0.03, 0.04, 0.04, 0.07, 0.07, 0.07, 0.08, 0.08, 0.1, 0.11, 0.12, 0.13, 0.14, 0.14, 0.14, 0.15, 0.16, 0.17, 0.21, 0.34 and 0.39 mg/kg.

The Meeting recommended a maximum residue level of 0.5 mg/kg and median 0.68 (6.15×0.11) mg/kg and highest 2.40 (6.15×0.39) mg/kg residues for use in estimation of livestock dietary burden for barley and wheat straw and fodder, dry. The Meeting agreed these values should be extrapolated to oats, rye and triticale straw and fodder, dry and replace the previous recommendations.

Fate of residues during processing

The Meeting received information on the nature of the residue of fenpropimorph under simulated processing conditions (pasteurization 20 minutes at 90 °C, pH 4, baking/brewing/boiling 60 minutes at 100 °C, pH 5, sterilization 20 minutes at 120 °C, pH 6) showed fenpropimorph, if present, is hydrolytically stable under processing conditions representative of pasteurisation, baking/boiling/brewing and under sterilisation conditions.

A number of processing studies on sugar beet, barley, oats and wheat were reviewed by the Meeting. A summary of relevant fenpropimorph processing factors is provided below.

	Processed Fraction	Processing Factor (PF)[A]	Best estimate PF	RAC STMR or median	STMR×PF	CF	STMR×PF×CF = STMR-P
Sugar beet	Molasses	< 0.04 < 0.06	0.05	0.01	0.0005	1.3	0.00065
	Refined sugar	< 0.04 < 0.06	0.05		0.0005		0.00065
	Dried pulp	2.9 3.9	3.4		0.034		0.0442
	Ensiled pulp	0.8 1	0.9		0.009		0.0117
Wheat	Bran	0.19 2.9 6.5	2.9	0.01	0.029	1.7	0.0493
	Flour	0.28 0.35 < 1	0.35		0.0035		0.00595
	Wholemeal flour	1.3 1.4 1.5	1.4		0.014		0.0238

	Processed Fraction	Processing Factor (PF)[A]	Best estimate PF	RAC STMR or median	STMR×PF	CF	STMR×PF×CF = STMR-P
	Wholemeal bread	1.1 1.4 3.2	1.4		0.014		0.0238
	Germ	2.4 4.2	3.3		0.033		0.0561
Barley	Brewing malt	0.7 0.8 1.2 1.4	1	0.02	0.02	1.7	0.034
	Beer	< 0.004 < 0.004 < 0.4 < 0.5	0.004		0.00008		0.000136
	Pot barley	0.6 0.8 1.0 1.6	0.9		0.018		0.0306
	Flour	2.5 2.5	2.5		0.05		0.085
Oats	Husks/dust	1 1.4 1.6 1.9	1.6	0.02	0.032	1.5	0.0544
	Oat flakes	0.5 0.8 1 1	0.8		0.016	1.7	0.0272

[A] PF = residues fenpropimorph in processed commodity divided by fenpropimorph in RAC

Residues of fenpropimorph concentrated in sugar beet pulp (dry), wheat bran, wholemeal flour, wholemeal bread, wheat germ and barley flour

The Meeting recommended the following maximum residue levels for processed commodities for which there are codes in the commodity classification: Sugar beet pulp (dry) 0.1 mg/kg (0.03×3.4=0.1 mg/kg), wheat bran (unprocessed) 0.2 mg/kg (0.07×2.9=0.2 mg/kg), wheat germ 0.3 mg/kg (0.07×3.3=0.23 mg/kg) and wheat wholemeal 0.1 mg/kg (0.07×1.4=0.098 mg/kg).

Residues in animal commodities

Farm animal feeding studies

The Meeting received information on the residue levels in tissues and milk of dairy cows dosed with fenpropimorph at the equivalent of 5, 16 and 52 ppm in the feed for 28 consecutive days.

Mean residues (max in brackets) of BF421-2 in milk were 0.012, muscle 0.03 (0.04), liver 0.75 (0.94), kidney 0.09 (0.11), fat 0.02 (0.02) mg/kg for the 5 ppm dose group and in milk were 0.065, muscle 0.19 (0.31), liver 5.6 (7.8), kidney 0.73 (0.92), fat 0.15 (0.22) mg/kg for the 52 ppm dose group.

An additional study was made available to the Meeting where dairy cows dosed with fenpropimorph at the equivalent of 2, 6 and 20 ppm in the feed for 28 consecutive days. Residues of fenpropimorph and BF421-2 in milk were 0.007 mg/kg, muscle 0.02 (0.02), liver 0.37 (0.46), kidney 0.08 (0.08), fat 0.03 (0.03) mg/kg for the 2 ppm dose group and in milk 0.046 mg/kg, muscle 0.14 (16), liver 2.9 (3.5), kidney 0.47 (54), fat 0.18 (0.22) mg/kg for the 20 ppm dose group.

A laying hens feeding study was not available.

Estimation of livestock dietary burdens

Dietary burden calculations for beef cattle and dairy cattle and poultry are provided below. The dietary burdens were estimated using the OECD diets listed in Appendix IX of the 2016 edition of the FAO Manual.

Potential cattle feed items include: cereal grain and straw and sugar beet tops.

Summary of livestock dietary burden (ppm fenpropimorph equivalents of dry matter diet)

	US-Canada		EU		Australia		Japan	
	Max	mean	Max	Mean	max	Mean	Max	Mean
Beef cattle	0.47	0.18	1.0	0.35	2.7 [A]	0.77 [C]	0.07	0.07
Dairy cattle	0.86	0.28	1.1	0.38	2.7 [B]	0.76 [D]	0.20	0.10
Broilers	0.08	0.08	0.07	0.07	0.03	0.03	0.01	0.01
Layers	0.08	0.08	0.25 [E]	0.13 [F]	0.03	0.03	0.01	0.01

[A] Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian meat

[B] Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

[C] Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian meat.

[D] Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

[E] Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs

[F] Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs

Animal commodity maximum residue levels

The calculations used to estimate highest total residues for use in estimating maximum residue levels, STMR and HR values are shown below.

	Feed level (ppm) for milk residues	Residues (mg/kg) in milk	Feed level (ppm) for tissue residues	Residues (mg/kg) in			
				Muscle	Liver	Kidney	Fat
MRL beef or dairy cattle							
Feeding study [a]	6.0	0.013	6.0	0.06	0.78	0.20	0.07
	2.0	0.007	2.0	0.02	0.46	0.08	0.03
Dietary burden and high residue	2.7	0.0077	2.7	0.027	0.516	0.101	0.037
STMR beef or dairy cattle							
Feeding study [b]	2.0	0.007	2.0	0.02	0.37	0.08	0.03
Dietary burden and median residue estimate	0.76	0.0027	0.77	0.0077	0.142	0.031	0.012

[a] highest residues for tissues and mean residues for milk

[b] mean residues for tissues and mean residues for milk

The Meeting estimated the following maximum residue levels: milk 0.01 mg/kg; meat (mammalian except marine mammals) 0.04 mg/kg, mammalian fat (except milk fat) 0.05 mg/kg and edible offal 0.7 mg/kg. The Meeting estimated the following HRs: mammalian meat 0.027 mg/kg; mammalian fat 0.037 mg/kg; liver 0.516 mg/kg, kidney 0.101 mg/kg and milk 0.0077 mg/kg and STMRs: mammalian meat 0.0077 mg/kg; mammalian fat 0.012 mg/kg; liver 0.142 mg/kg, kidney 0.031 mg/kg and milk 0.0027 mg/kg.

The Meeting agreed to withdraw its previous recommendation for Kidney of cattle, goats, pigs and sheep of 0.05 mg/kg and for Liver of cattle, goats, pigs and sheep of 0.3 mg/kg.

Although a laying hen feeding study was not available, in a metabolism study where hens were dosed at the equivalent of 12–14 ppm in the diet for ten days, residues of fenpropimorph and BF421-2 were 0.03–0.04 mg/kg in eggs, 0.003–0.005 mg/kg in muscle, 0.06 mg/kg in fat and 0.08–0.09 mg/kg in liver.

At the maximum dietary burden for poultry (0.25 ppm), no residues above typical LOQs for analytical methods are expected in eggs and tissues. The Meeting estimated the following maximum residue levels for poultry commodities: poultry meat 0.005* mg/kg; poultry edible offal 0.005* mg/kg, poultry fat 0.005* mg/kg and eggs 0.005* mg/kg to replace its previous recommendations for these commodities. The Meeting estimated the following STMR and HR values: poultry meat 0 mg/kg; poultry fat 0 mg/kg; poultry edible offal 0 mg/kg and eggs 0 mg/kg.

RECOMMENDATIONS

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

Definition of the residue (for compliance with MRLs) for plant commodities*: fenpropimorph*

Definition of the residue (for estimation of dietary intake) for plant commodities*: sum of fenpropimorph, 2-(4-{(2RS)-3-[(2R,6S)-2,6- dimethylmorpholin-4-yl]-2- methylpropyl}phenyl)-2-methylpropan-1-ol (free and conjugated) (BF421-1) and (2R,6S)-2,6-dimethylmorpholine (BF421-10) expressed as fenpropimorph.*

Definition of the residue (for compliance with MRLs and estimation of dietary intake) for animal commodities*: sum of fenpropimorph and 2-(4-{(2RS)-3-[(2R,6S)-2,6- dimethylmorpholin-4-yl]-2- methylpropyl}phenyl)-2- methylpropanoic acid (BF421-2) expressed as fenpropimorph*

The residue is not fat soluble.

Table of recommendations

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P (mg/kg)	HR, HR-P, highest residue (mg/kg)
CCN	Name	New	Previous		
FI 0327	Banana	2	2	0.08	0.43
GC 0640	Barley	0.2	0.5	0.085 (0.075[b])	
AS 0640	Barley straw and fodder, dry	0.5	5	0.68	2.4
MO 0105	Edible offal (mammalian)	0.7		0.142 Liver 0.031 Kidney	0.516 Liver 0.101 Kidney
PE 0112	Eggs	0.005*	0.01*	0	0
MO 0098	Kidney of cattle, goats, pigs and sheep	W [a]	0.05		
MO 0099	Liver of cattle, goats, pigs and sheep	W [a]	0.3		
MF 0100	Mammalian fats (except milk fats)	0.05	0.01	0.012	0.037
MM 0095	Meat (from mammals other than marine mammals)	0.04	0.02	0.0077	0.027
ML 0106	Milks	0.01	0.01	0.0027	0.0077
GC 0647	Oats	0.2	0.5	0.085 (0.075[b])	
AS 0647	Oats straw and fodder, dry	0.5	5	0.68	2.4
PF 0111	Poultry fats	0.005*	0.01*	0	0
PM 0110	Poultry meat	0.005*	0.01*	0	0
PO 0111	Poultry, Edible offal of	0.005*	0.01*	0	0
GC 650	Rye	0.07	0.5	0.017 (0.015[b])	
AS 650	Rye straw and fodder, dry	0.5	5	0.68	2.4
VR 0596	Sugar beet	0.03	0.05*	0.013	
AV 1051	Fodder beet leaves or tops	W	1		
AV 0596	Sugar beet leaves or tops			0.367 dw	0.848 dw
AB 0596	Sugar beet pulp, dry	0.1		0.0442 dw	
GC 0653	Triticale	0.07		0.017 (0.015[b])	
AS 0653	Triticale straw and fodder, dry	0.5		0.68	2.4
GC 0654	Wheat	0.07	0.5	0.017 (0.015[b])	
CM 0654	Wheat bran, unprocessed	0.2		0.0493	
CF 1210	Wheat germ	0.3		0.0561	
AS 0654	Wheat straw and fodder, dry	0.5	5	0.68	2.4
CF 1212	Wheat wholemeal	0.1		0.0238	

dw = dry weight basis

[a] to be replaced by edible offal (mammalian) recommendation

[b] for use in livestock dietary burden estimation, residues of fenpropimorph and BF421-1

Table of additional STMR/median and HR/highest residue values for use in dietary intake and livestock dietary burden estimation.

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P, median residue (mg/kg)	HR, HR-P, highest residue (mg/kg)
CCN	Name	New	Previous		
	Beer			0.000136	
	Pot Barley			0.0306	
	Barley flour			0.085	
CF 1211	Wheat flour			0.00595	

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P, median residue (mg/kg)	HR, HR-P, highest residue (mg/kg)
CCN	Name	New	Previous		
	Oat flakes			0.0272	
DM 0596	Sugar beet molasses			0.00065	
	Sugar, refined			0.00065	

dw = dry weight basis

DIETARY RISK ASSESSMENT

Long-term exposure

The 2016 JMPR established an Acceptable Daily Intake (ADI) of 0–0.004 mg/kg bw for fenpropimorph.

The evaluation of fenpropimorph resulted in recommendations for MRLs and STMR values for raw and processed commodities. Where data on consumption were available for the listed food commodities, dietary intakes were calculated for the 17 GEMS/Food Consumption Cluster Diets. The results are shown in Annex 3 to the 2017 Report.

The IEDIs in the seventeen Cluster Diets, based on the estimated STMRs were 1–10% of the maximum ADI (0.004 mg/kg bw). The Meeting concluded that the long-term dietary exposure to residues of fenpropimorph from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term exposure

The 2016 JMPR established an Acute Reference Dose (ARfD) of 0.1 mg/kg for women of child-bearing age and 0.4 mg/kg bw for the general population for fenpropimorph. The IESTI of fenpropimorph, for the commodities for which STMR, HR and maximum residue levels were estimated by the current Meeting are shown in Annex 4 to the 2017 Report. The IESTI represented 0–5% of the ARfD for women of child-bearing age and 0–9% for the general population.

The Meeting concluded that the short-term dietary exposure to residues of fenpropimorph resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

REFERENCES

Code	Author(s)	Year	Reference
	Almvik, M., Bolli, R.I., Christiansen, A., Eklo, O.M., Holten, R.	2015	Slow degradation of fungicides in soils from cold temperate climate. Page 70 in XV Symposoium in Pesticide Chemistry. Environmental Risk Assessment and Management, Cigolini M, Luzzani G and Sacchettini G editors. Mattioli 1885. See also http://www.bioforsk.no/ikbViewer/Content/112600/Almvik.pdf
10076	Anonymous	1978a	Method of analysis for gas chromatographic determination of 108 406 F in grain, straw, soil and water, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1978/10076, Unpublished
10088	Anonymous	1979a	Analytical method No. 156: Fenpropimorph - GC determination. Grain, straw, green plants, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1979/10088, Unpublished
10160	Anonymous	1986	BAS 421 00 F - Pesticide residue analysis: Sugar beet, Faculte des Sciences Agronomiques, Gembloux, Belgium, 1986/10160, Unpublished

Code	Author(s)	Year	Reference
10161	Anonymous	1986	Pesticide residue analysis: Sugar beet, Faculte des Sciences Agronomiques, Gembloux, Belgium, 1986/10161, Unpublished
10162	Anonymous	1986	BAS 421 00 F - Pesticide residue analysis: Sugar beet, Faculte des Sciences Agronomiques, Gembloux, Belgium, 1986/10162, Unpublished
10261	Anonymous	1979	Pflanzenschutzmittelrueckstands-Untersuchungen: SommerweizenBASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep., 1979/10261, Unpublished
10262	Anonymous	1979	Pflanzenschutzmittelrueckstands-Untersuchungen: Sommerweizen, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1979/10262, Unpublished
10263	Anonymous	1979	Pflanzenschutzmittelrueckstands-Untersuchungen: Sommerweizen, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1979/10263, Unpublished
10330	Anonymous	1979	Pflanzenschutzmittelrueckstands-Untersuchungen: SommerweizenBASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1979/10330, Unpublished
10331	Anonymous	1979	Pflanzenschutzmittelrueckstands-Untersuchungen: SommerweizenBASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1979/10331, Unpublished
10332	Anonymous	1979	Pflanzenschutzmittelrueckstands-Untersuchungen: SommerweizenBASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1979/10332, Unpublished
10333	Anonymous	1979	Pflanzenschutzmittelrueckstands-Untersuchungen: Sommerweizen, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1979/10333, Unpublished
10334	Anonymous	1979	Pflanzenschutzmittelrueckstands-Untersuchungen: Sommerweizen, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1979/10334, Unpublished
10335	Anonymous	1979	Pflanzenschutzmittelrueckstands-Untersuchungen: Sommerweizen, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1979/10335, Unpublished
10351	Anonymous	1983	BAS 421 00 F - Pflanzenschutzmittelrueckstands-Untersuchungen: Zuckerrueben, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1983/10351, Unpublished
10352	Anonymous	1983	BAS 421 00 F - Pflanzenschutzmittelrueckstands-Untersuchungen: Zuckerrueben, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1983/10352, Unpublished
10353	Anonymous	1983	BAS 421 00 F: Pflanzenschutzmittelrueckstands-Untersuchungen – Zuckerrueben, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1983/10353, Unpublished
10357	Anonymous	1980	Pflanzenschutzmittelrueckstands-Untersuchungen: Sommergerste, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1980/10357, Unpublished
10358	Anonymous	1980	Pflanzenschutzmittelrueckstands-Untersuchungen: Sommergerste, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1980/10358, Unpublished
1985/10359	Anonymous	1985	BAS 421 00 F - Pflanzenschutzmittelrueckstands-Untersuchungen: Zuckerrueben, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1985/10359, Unpublished
1980/10359	Anonymous	1980	Pflanzenschutzmittelrueckstands-Untersuchungen: Sommergerste, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1980/10359, Unpublished
10360	Anonymous	1980	Pflanzenschutzmittelrueckstands-Untersuchungen: Sommergerste, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1980/10360, Unpublished
10463	Anonymous	1987	Pflanzenschutzmittelrueckstands-Untersuchungen: Zuckerrueben, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1987/10463, Unpublished
10504	Anonymous	1989	BAS 421 00 F - Pflanzenschutzmittelrueckstands-Untersuchungen: Zuckerrueben BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1989/10504, Unpublished

Code	Author(s)	Year	Reference
10505	Anonymous	1989	BAS 421 00 F - Pflanzenschutzmittelrueckstands-Untersuchungen: Zuckerruebe, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1989/10505, Unpublished
10768	Anonymous	1989	BAS 421 00 F - Pesticide residue analysis: Sugar beet, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1989/10768, Unpublished
10770	Anonymous	1989	BAS 421 00 F - Pesticide residue analysis: Sugar beet, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1989/10770, Unpublished
10795	Anonymous	1980	Pflanzenschutzmittelrueckstands-Untersuchungen: Sommergerste, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1980/10795, Unpublished
10797	Anonymous	1980	Pflanzenschutzmittelrueckstands-Untersuchungen: Sommergerste, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1980/10797, Unpublished
10823	Anonymous	1988	BAS 421 00 F - Pflanzenschutzmittelrueckstands-Untersuchungen: ZuckerruebeBASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1988/10823, Unpublished
11785	Anonymous	1991	BAS 421 00 F - Pesticide residue analysis: Sugar beet, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1991/11785, Unpublished
11786	Anonymous	1991	BAS 421 00 F (Corbel): Pesticide residue analysis - Sugar beets, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1991/11786, Unpublished
5077	Artz S.C., Malinsky S.	1997 a	Independent method validation of BASF analytical method no. 241/1: Gas chromatographic determination of Fenpropimorph in banana fruit, peel and pulp at BASF, BASF Corporation Agricultural Products Center, Research Triangle Park NC 27709, USA, 1997/5077, GLP, Unpublished
1007053	Atkinson S.	2002 a	Independent laboratory validation of the S19 method for the determination of Fenpropimorph in poultry matrices, CEMAS - CEM Analytical Services Ltd., North Ascot Berkshire SL5 8JB, United Kingdom, 2002/1007053, GLP, Unpublished
1019510	Bacher R.	2001 a	Assessment and validation of the adapted multi-residue method DFG S19 for the determination of BAS 421 F (Fenpropimorph) in poultry matrices PTRL Europe GmbH, Ulm, Germany Fed. Rep. 2001/1019510, GLP, Unpublished
	Baloch, R.I.; Mercer, E.I.	1987	Inhibition of sterol Δ8→Δ7-isomerase and Δ14-reductase by fenpropimorph, tridemorph and fenpropidin in cell-free enzyme systems from Saccharomyces cerevisiae. Phytochemistry 26:663−668.
10470	Baumann R.A. et al.	1996 a	Pesticides amenable to gas chromatography: Multi residue method 1National Institute of Public Health and the Environment, Bilthoven, Netherlands, 1996/10470, Unpublished
1000985	Benz A., Mackenroth C.	2001	Validation of BASF method No. 456/0: Determination of Fenpropimorph (BAS 421 F) in various plant matrices, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2001/1000985, GLP, Unpublished
1001287	Benz A., Mackenroth C.	2003 b	Determination of the storage stability of BAS 421 F (Fenpropimorph) in various plant matrices, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2003/1001287, GLP, Unpublished
1004669	Benz A., Mackenroth C.	2003 a	Determination of the storage stability of BAS 421 F (Fenpropimorph) in various plant matrices, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2003/1004669, GLP, Unpublished
1026504	Benz A., Mackenroth C.	2006 a	Validation of the analytical method 573/0: Method for the determination of Fenpropimorph (BAS 421 F) and its metabolite BF 421-2 in animal matrices, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2005/1026504, GLP, Unpublished
1000021	Beutel P	1978	Soil dissipation and metabolism of 108 406 (=BAS 42100F). BASF Doc ID 1978/1000021, Unpublished

Code	Author(s)	Year	Reference
10142	Beutel P., Tilting N.	1987 a	Analytical method No. 241: Fenpropimorph - GC determination. Wheat: forage, spindles, stalks, straw, grains; Rape: forage, seeds; Brussels sprouts; Tangerines; Oranges, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1987/10142, GLP, Unpublished
3005401	Bissetti Costa D.L. La	2012 a	Determinacao de residuo de Fenpropimorph, em banana, apos a aplicacao de CorbelPLANTEC - Planejamento e Tecnologia Agricola Ltda., Iracemapolis, Brazil, 2012/3005401, GLP, Unpublished
1009068	Blaschke U.	2001	Determination of the magnitude of the residue of BAS 528 00 F and BAS 500 01 F in/on cereal raw agricultural commodity specimens from supervised field trials in Northern Europe in 2000, Huntingdon Life Sciences Ltd., Huntingdon Cambridgeshire PE28 4HS, United Kingdom, 2001/1009068, GLP, Unpublished
10739	Blaschke U.G.	1995 a	Raw agricultural commodity study with LX 1262-02 (CORBEL = BAS 421 12 F) applied to bananas in Mexico, Paragon Global Services, Eye Suffolk IP23 7PX, United Kingdom, 1995/10739, GLP, Unpublished
	Buerge IJ, Krauss J, López-Cabeza R, Siegfried W, Stüssi M, Wettstein FE, Poiger T	2016	Stereoselective Metabolism of the Sterol Biosynthesis Inhibitor Fungicides Fenpropidin, Fenpropimorph, and Spiroxamine in Grapes, Sugar Beets, and Wheat. J. Agric. Food Chem., 64: 5301–5309
3003426	Costa D.L.B. La	2012	Determinacao de residuo de Fenpropimorph, em graos de cevada, apos a aplicacao de Corbel, PLANTEC - Planejamento e Tecnologia Agricola Ltda., Iracemapolis, Brazil, 2012/3003426, GLP, Unpublished
11214	Daum A.	1999 a	Determination of the melting point and the appearance of Fenpropimorph (Reg. No. 108 406, BAS 421 F), BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1999/11214, GLP, Unpublished
1008316	Daum A.	1999 b	Fenpropimorph PAI: DSC raw data for GLP Study 1999/11214, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1999/1008316, GLP, Unpublished
1010873	Ebert D, Harder U	2005	Aerobic rate of degradation of BF421-2 (metabolite of BAS 421 F, fenpropimorph) BASF DocID 2005/1010873, Unpublished
1005601	Ebert D.	2010 a	Rate of degradation of BF 421-7 (Reg.No. 4994030) in different soils under aerobic conditions, BASF SE Agricultural Center Limburgerhof, Limburgerhof, Germany Fed. Rep. 2010/1005601, GLP, Unpublished
10250	Elsom LF	1998	^{14}C-Dimethylmorpholine – aerobic soil metabolism and route of degradation. Huntingdon Life Sciences Ltd, Huntingdon Cambridgeshire PE28 4HS UK. 1998/10250, Unpublished
1050094	Erdmann H.-P.	2007	Study on the residue behaviour of BAS 421 F in wheat and barley after application of BAS 421 12 F under field condition in France and Germany, 2007, Agro-Check, Lentzke, Germany Fed. Rep. 2007/1050094, Unpublished
1050096	Erdmann H.-P.	2008 b	Study on the residue behaviour of BAS 421 F in wheat after application of BAS 421 12 F under field condition in France, 2007, Agro-Check, Lentzke, Germany Fed.Rep. 2007/1050096, GLP, Unpublished
1014911	Fabian E., Knoell H.-E.	2003 a	The metabolism of ^{14}C-BAS 421 F (Fenpropimorph) in lactating goats, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep., 2001/1014911, GLP, Unpublished
10822	Ford R.T.G	1980	Residues of Fenpropimorph in cereals following applications of Corbel (Mistral - M & B) in the United Kingdom during 1979, BASF United Kingdom Ltd., Ipswich Suffolk IP7 6BQ, United Kingdom, 1980/10822, Unpublished
10736	Ford R.T.G.	1981	Addendum to report No. 139: Further residues of Fenpropimorph in cereals following applications of Corbel (Mistral - M & B) in the United Kingdom during 1979, BASF United Kingdom Ltd., Ipswich Suffolk IP7 6BQ, United Kingdom, 1981/10736, Unpublished

Code	Author(s)	Year	Reference
10737	Ford R.T.G.	1981	Residues of fenpropimorph in cereals following applications of Corbel (Mistral - M & B) in the United Kingdom during 1980, BASF United Kingdom Ltd., Ipswich Suffolk IP7 6BQ, United Kingdom, 1981/10737, Unpublished
1000062	Ford R.T.G.	1978	Residues of BAS 421 F in spring wheat following applications of BAS 421 00 F in West Germany during 1977, BASF United Kingdom Ltd., Ipswich Suffolk IP7 6BQ, United Kingdom, 1978/1000062, Unpublished
1000101	Ford RTG	1984	Residues of fenpropimorph in rye and field beans following application of Corbel in the United Kingdom during 1983. BASF Doc ID 1984/1000101, Unpublished
10189	Haenni R.	1980	Compilation of Fenpropimorph (Ro 14-3169/000 formulated as ACR 3320) residue data 1979 from Great Britain, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1980/10189, Unpublished
1021394	Hafemann C., Gläßgen W.E.	2006 a	Residues of Fenpropimorph (BAS 421 F) in milk and tissues of dairy cows following multiple oral administrations, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2005/1021394, GLP, Unpublished
1025538	Hafemann C., Mackenroth C.	2005	Storage stability investigations of BF 421-2 in cow samples, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2005/1025538, GLP, Unpublished
10536	Hamm R.T.	1997 a	Metabolism of ^{14}C-Fenpropimorph (BAS 421 F) in banana, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1997/10536, GLP, Unpublished
10709	Hamm R.T.	1995 a	Plant uptake study with ^{14}C-Fenpropimorph [morpholine-2,6-^{14}C) in banana, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1995/10709, GLP, Unpublished
10710	Hamm R.T.	1995 b	Plant uptake study with ^{14}C-Fenpropimorph [phenyl-U-^{14}C) in banana, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1995/10710, GLP, Unpublished
1018643	Hamm R.T.	2000 a	Metabolism of BAS 421 F in sugar beets, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2000/1018643, GLP, Unpublished
1025931	Harant H.	2006 a	Determination of residues of Fenpropimorph in spring barley and its processing products after two applications of BAS 421 12 F in Germany, BioChem agrar Labor fuer biologische und chemische Analytik GmbH, Gerichshain, Germany Fed. Rep. 2006/1025931, GLP, Unpublished
1014901	Hartl M.	2002 a	The metabolism of ^{14}C-BAS 421 F in laying hens, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2001/1014901, GLP, Unpublished
1022507	Hassink J, Stephan A	2005	Metabolism of BAS 421F in soil under aerobic conditions. BASF Doc ID 2004/1022507, Unpublished
0433	Herrchen M.	1988 a	Soil photolysis of Fenpropimorph, Fraunhofer-Institut fuer Umweltchemie und Oekotoxikologie, Schmallenberg, Germany Fed.Rep. 1988/0433, GLP, Unpublished
10760	Hesse B, Tilting N	1991	Dissipation of fenpropimorph in the soil under field conditions. BASF AG 1991/10760, Unpublished
1000183	Hesse B, Tilting N	1992	The determination of the dissipation behaviour of fenpropimorph in soil under field conditions. BASF AG 1992/1000183, Unpublished
	Himmele, W., Pommer, E.-H.	1980	3-Phenylpropylamines, a new class of systemic fungicides. Angew. Chem., Int. Ed. Engl. 19: 184−189
1000141	Hofmann M.	1986 a	^{14}C-Trial-lay-out/metabolism wheat, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1986/1000141, Unpublished
1000142	Hofmann M.	1986 b	^{14}C-Trial-lay-out/metabolism spring wheat, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1986/1000142, Unpublished
	Houbraken M, Senaeve D, Fevery D, Spanoghe P	2015	Influence of adjuvants on the dissipation of fenpropimorph, pyrimethanil, chlorpyrifos and lindane on the solid/gas interface. Chemosphere 138: 357-363.
10024	Huber R	1979	Stage on the investigations of the metabolism of fenpropimorph in soil. BASF Doc ID 1979/10024, Unpublished

Code	Author(s)	Year	Reference
0148	Huber R.	1979 a	Investigations on the metabolism of Fenpropimorph (Reg.Nr. 108 406, BAS 421 F) in wheat, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1979/0148, Unpublished
10392	Kästel R.	1994	Physical and chemical properties report for Fenpropimorph (technical active ingredient), BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep., 1994/10392, GLP, yes Unpublished
10395	Kästel R.	1994 a	Physical and chemical properties report for Fenpropimorph (108 406), BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1994/10395, GLP, Unpublished
1016297	Kästel R.	2004	Vapour pressure of Fenpropimorph, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2004/1016297, GLP, Unpublished
10061	Keller W	1985	Influence of temperature and moisture on the degradation behaviour of fenpropimorph in the soil BASF Doc ID 1985/10061, Unpublished
10693	Knecht K-E, Tilting N	1993	Prüfung des Rückstandsverhaltens von BAS 421 12 F (Corbel) an Getreide unter Feldbedingungen. Reg Doc #BASF 93/10693, Unpublished
1025930	Kniep R.	2006 a	Determination of residues of Fenpropimorph in winter wheat and its processing products after two applications of BAS 421 12 F in Germany, BioChem agrar Labor fuer biologische und chemische Analytik GmbH, Gerichshain, Germany Fed. Rep., 2006/1025930, GLP, Unpublished
1013434	Lantos J.	2000	BAS 481 07 F: Residue study with Epoxiconazole and Fenpropimorph in sugar beet - Treatment with Tango Star, 2000Plant Health and Soil Conservation Station of Szabolcs-Szatmar-Bereg County, Nyiregyhaza, Hungary, 2000/1013434, Unpublished
1013272	Leibold E., Hoffmann H.D.	2000 a	^{14}C-BAS 421 F - Absorption, distribution and excretion after repeated oral administration in lactating goats, BASF AG, Ludwigshafen/Rhein, Germany Fed. Rep. 2000/1013272, GLP, Unpublished
1013365	Leibold E., Hoffmann H.D.	2000 b	^{14}C-BAS 421 F - Absorption, distribution and excretion after repeated oral administration in laying hens, BASF AG, Ludwigshafen/Rhein, Germany Fed. Rep. 2000/1013365, GLP, Unpublished
1396627	Linder G.	2014 a	Study on the residue behaviour of Fenpropimorph (BAS 421 F) in sugar beets after treatment with BAS 421 12 F under field conditions in Germany, France (North), United Kingdom and The Netherlands, 2013, BASF SE, Limburgerhof, Germany Fed. Rep. 2013/1396627, GLP, Unpublished
1039427	Mackenroth C., Lehmann A.	2006	Validation of BASF method No. 535/1 in plant matrices, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2006/1039427, GLP, Unpublished
10384	Maxwell J. G.	1993 a	Validation of the method of analysis for Fenpropimorph in plant matter, Huntingdon Research Centre Ltd., Huntingdon Cambridgeshire PE18 6ES, United Kingdom, 1993/10384, GLP, Unpublished
1016476	Ohnsorge U.	2004	Calculation of the pKa value of Fenpropimorph hydrochloride and of pH dependent log Pow values for Fenpropimorph, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2004/1016476, Unpublished
1031205	Ohnsorge U.	2004	Henrys law constant for Fenpropimorph, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2004/1031205, Unpublished
1012810	Oxspring S.	2009 a	Study on the residue behaviour of Fenpropimorph in sugar beet after treatment with BAS 421 12 F under field conditions in Northern and Southern Europe during 2007, Agrisearch UK Ltd., Melbourne Derbyshire DE73 8AG, United Kingdom, 2009/1012810, GLP, Unpublished

Code	Author(s)	Year	Reference
1024749	Perny A.	2004	Study on the residue behaviour of Fenprophimorph and Epoxiconazole on sugar beets after application of BAS 481 08 F under field conditions in Greece and Spain, 2003, Anadiag SA, Haguenau, France, 2004/1024749, GLP, Unpublished
1007746	Platz K	2002	Standardisation of laboratory half-lives of BAS 421 F – fenpropimorph to reference conditions according to the recommendations of FOCUS. BASF Doc ID 2002/1007746, Unpublished
1020649	Platz K.	2005 a	Summary of laboratory half-lives in soil of BAS 421 F (Fenpropimorph) and BF 421-2 and normalisation to 20 °C and moisture at field capacity, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed.Rep. 2005/1020649, Unpublished
1037958	Plier S.	2013 a	Determination of residues of BAS 421 F (Fenpropimorph) in sugar beets and their processed products after one application of BAS 421 12 F in Germany, 2012, BioChem agrar Labor fuer biologische und chemische Analytik GmbH, Gerichshain, Germany Fed. Rep. 2013/1037958, GLP, Unpublished
	Pommer E-H	1984	Chemical structure-fungicidal activity relationships in substituted morpholines. Pestic. Sci. 15: 285−295
10097	Pryde A. ,Etterli M.	1979	Investigation of the relative contribution of the systemic and gas phase activities to the total activity of ^{14}C-Rol4- 3169/001 formulated as ACR3320 in barley plants using chemical and autoradiographic techniques, Dr. R. Maag AG, Dielsdorf, Switzerland, 1979/10097, Unpublished
10102	Pryde A. ,Etterli M.	1980	Crop rotation studies with radioactive fenpropimorph (Ro 14-3169/001). Internal Report 1980/10102, Unpublished
1001380	Rabe U.	2003 a	Confined rotational crop study with ^{14}C-BAS 421 F, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2003/1001380, GLP, Unpublished
1034290	Rabe U.	2006 b	Fenpropimorph - Reanalyses of wheat grain samples from the wheat metabolism study (BASF DocID 2003/1001288), BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2006/1034290, GLP, Unpublished
1036924	Rabe U.	2006	Reanalysis of Dimethylmorpholine in wheat and sugar beet samples from the metabolism studies 151744 and 151741, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2006/1036924, GLP, Unpublished
1001288	Rabe U., Gläßgen W.E.	2005 b	Metabolism of ^{14}C-BAS 421 F in spring wheat, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2003/1001288, GLP, Unpublished
1021389	Rabe U., Gläßgen W.E.	2005 a	Metabolism of ^{14}C-BAS 421 F in sugar beet, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2005/1021389, GLP, Unpublished
1010542	Raunft E. et al.	2004	Study on the residue behaviour of Fenpropimorph and BAS 560 F in cereals after application of BAS 421 12 F, BAS 560 00 F and BAS 564 AF F under field conditions in France, Germany, Denmark, United Kingdom, Italy and Spain, 2003, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2004/1010542, GLP, Unpublished
11302	Redeker J.	1988 a	Water solubility of Fenpropimorph in the neutral range and at pH 9-11, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1988/11302, GLP, Unpublished
11596	Redeker J.	1992	Determination of the solubility of Fenpropimorph in organic solvents at 20 °C, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1992/11596, GLP, Unpublished
11671	Redeker J.	1988 b	Determination of the pKb value of Fenpropimorph in water, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1988/11671, GLP, Unpublished
11913	Redeker J.	1988 c	Determination of the solubility of Fenpropimorph hydrochloride in water, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1988/11913, GLP, Unpublished

Code	Author(s)	Year	Reference
1014084	Renner G.	2005 a	Determination of residues of Fenpropimorph in oat and its processing products after two applications of BAS 421 12 F in Germany, BioChem agrar Labor fuer biologische und chemische Analytik GmbH, Gerichshain, Germany Fed. Rep. 2005/1014084, GLP, Unpublished
0595	Ritter A.	1989 b	Distribution, excretion and metabolism of ^{14}C-BAS 421 F after repeated oral administration to laying hens, RCC Umweltchemie AG, Itingen, Switzerland, 1989/0595, GLP, Unpublished
10215	Ritter A.	1989 a	Distribution, degradation and excretion of ^{14}C-BAS 421 F after repeated oral administration to lactating goats, RCC Umweltchemie AG, Itingen, Switzerland, 1989/10215, GLP, Unpublished
0443	Rüdel H.	1988	Hydrolysis studies on BAS 421 F at pH 3, 5, 7 and 9, Fraunhofer-Institut fuer Umweltchemie und Oekotoxikologie, Schmallenberg, Germany Fed. Rep. 1988/0443, GLP, Unpublished
0524	Rüdel H.	1990 a	Investigations into the metabolism of Fenpropimorph in wheat, Fraunhofer-Institut fuer Umweltchemie und Oekotoxikologie, Schmallenberg, Germany Fed. Rep. 1990/0524, GLP, Unpublished
0525	Rüdel H.	1990 b	Addendum to the final report: Investigations into the metabolism of Fenpropimorph in wheat, Fraunhofer-Institut fuer Umweltchemie und Oekotoxikologie, Schmallenberg, Germany Fed. Rep. 1990/0525, GLP, Unpublished
10301	Sarafin R.	1991 a	Photochemical oxidative degradation of Fenpropimorph (Atkinson), BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1991/10301, Unpublished
10344	Sarafin R.	1991 b	Fenpropimorph (BAS 421 .. F) Absorption coefficients, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1991/10344, GLP, Unpublished
1012400	Sasturain J., Mackenroth C.	2000	Validation of BASF method No. 241/3 for determination of Fenpropimorph in plant matrices, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2000/1012400, GLP, Unpublished
11092	Scharf J.	1999 a	Hydrolysis of Fenpropimorph (BAS 421 F) at 90 °C, 100 °C, and 120 °C, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed.Rep. 1999/11092, GLP, Unpublished
1009081	Schroth E.	2001	Determination of residues of Fenpropimorph in/on barley following two applications of BAS 421 12 F (Fenpropimorph) under field conditions in Spain and Italy, 2000, Agrologia SL, Palomares, Spain 2001/1009081, GLP, Unpublished
1004082	Schulz H.	2002	Determination of Fenpropimorph in processed products of wheat and barley - Validation of the Method No. 241/3, Institut Fresenius Chemische und Biologische Laboratorien GmbH, Taunusstein, Germany Fed. Rep. 2002/1004082, GLP, Unpublished
1009082	Schulz H.	2002 a	Determination of the residues of Fenpropimorph in wheat and processed products following treatment with BAS 421 12 F under field conditions in Germany 2000, Institut Fresenius Chemische und Biologische Laboratorien AG, Taunusstein, Germany Fed. Rep. 2001/1009082, GLP, Unpublished
1009083	Schulz H.	2002 b	Determination of the residues of Fenpropimorph in barley and processed products following treatment with BAS 421 12 F under field conditions in Germany 2000, Institut Fresenius Chemische und Biologische Laboratorien GmbH, Taunusstein, Germany Fed. Rep. 2001/1009083, GLP, Unpublished
1026505	Schulz H.	2006 a	Determination of Fenpropimorph (Reg.No. 108 406) and its metabolite BF 421-2 in animal matrices ILV - Independent laboratory validation of the method No. 573/0, SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep. 2005/1026505, GLP, Unpublished
1075091	Schulz H.	2009 a	Study on the residue behaviour of Fenpropimorph in sugar beet after treatment with BAS 421 12 F under field conditions in Germany, Northern France, Greece and Spain, 2008, SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep. 2009/1075091, GLP, Unpublished

Code	Author(s)	Year	Reference
5003	Shaffer S.R., Artz S.C.	1997 a	Specificty test for BASF analytical method no. 241/1: Gas chromatographic determination of Fenpropimorph in banana fruit, peel and pulp, Horizon Laboratories Inc., Columbia MO, United States of America, 1997/5003, GLP, Unpublished
	Smelt, J.H., Smidt RA, van den Berg F, Matser AM, Stork A, Ophoff H	1997	Volatilization of fenpropimorph and clopyralid after spraying onto a sugarbeet crop. Wageningen (The Netherlands), DLO Winand Staring Centre. Report 136. 46 pp
10365	Stockmaier M, Robert Kreuzig, Müfit Bahadir	1996	Investigations on the behaviour of fenpropimorph and its metabolite fenpropimorphic acid in soils. Pest Sci 46:361-367
1050905	Thiaener J.	2009	Investigation of the storage stability of BAS 421 F (Reg.No. 108406) and BF 421-2 (Reg. No. 231346) in cow samples, BASF SE, Limburgerhof, Germany Fed. Rep. 2007/1050905, GLP, Unpublished
10207	Tilting N.	1989	Applicability of DFG multiresidue methods S 8 and S 19 for the determination of Fenpropimorph, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1989/10207, GLP Unpublished
10550	Tilting N.	1994 a	Method for the determination of Fenpropimorph in soil and plant samples by gas chromatography, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1994/10550, GLP, Unpublished
10781	Tilting N.	1993 a	Two years storage stability study of Fenpropimorph residues in wheat and soil, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1993/10781, GLP, Unpublished
11464	Tilting N.	1993 a	Gas chromatographic determination of Fenpropimorph in banana fruit, peel and pulp, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1993/11464, GLP, Unpublished
1014760	Tilting N.	2001 a	Independent validation of Ciba method REM 167.03 for the determination of the Fenpropimorph metabolite CGA 294975 (BF 421-2) in matrices of animal origin and description of a confirmatory method, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 2001/1014760, GLP, Unpublished
10747	Tilting N., Mackenroth C.	1995	Study on the residue behaviour of BAS 421 12 F (Fenpropimorph) and BAS 480 21 F (Epoxiconazole) in bananas, BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1995/10747, GLP, Unpublished
10433	Tribolet R.	1999 a	Residues of CGA 294975 (metabolite of Fenpropimorph) in milk, blood and tissues (muscle, fat, liver, kidney) of dairy cattle resulting from feeding of Fenpropimorph at three dose levels, Novartis Crop Protection AG, Basel, Switzerland 1999/10433, GLP, Unpublished
11081	Tribolet R.	1995 a	Fenpropimorph (CGA 101031). Determination of metabolite CGA 294975 by high performance liquid chromatography (HPLC). Animal tissues, egg and milk, Ciba-Geigy AG, Basel, Switzerland, 1995/11081, Unpublished
10288	Türk W.	1996	Spectra of Fenpropimorph Reg.No. 108 406 (PAI), BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed. Rep. 1996/10288, GLP, Unpublished
10042	Von der Mühll et al.	1980	Soil field dissipation, metabolism and leaching study with radioactive fenpropimorph. BASF 1980/10042
11462	Weeren R.D., Pelz S.	1999 a	Validation of DFG method S 19 for the determination of Dimethenamid, Epoxiconazole, Fenpropimorph, Kresoxim-methyl, Metazachlor and Vinclozolin in various plant materials, Dr. Specht & Partner Chemische Laboratorien GmbH, Hamburg, Germany Fed. Rep. 1999/11462, Unpublished
7004267	White M.T., Stewart J.	2005	Residue of Metrafenone (BAS 560 F) Fenpropimorph, (BAS 421 F) Epoxiconazol (BAS 480 F) in formulation bridging on wheat, barley after BAS 565 00 F BAS 562 00 F BAS 560 00 F BAS 421 12 F BAS 480 31 F field, in DE, DK, N-FR, S-FR, SE, UK 2005 BASF Agro Research RTP, Research Triangle Park NC, United States of America 2005/7004267, GLP, Unpublished

Code	Author(s)	Year	Reference
5004	Wofford J.T. ,Artz S.C.	1997 a	Magnitude of Fenpropimorph residues in banana, BASF Corp. Agricultural Products Center, Research Triangle Park NC, United States of America, 1997/5004, GLP, Unpublished