FENPYROXIMATE(193)

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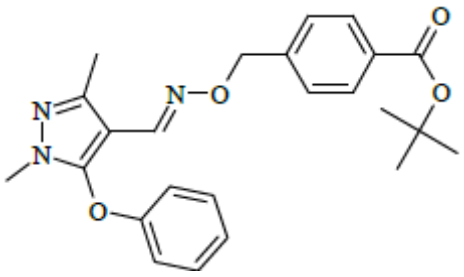
EXPLANATION

Fenpyroximate is a pyrazole non-systemic selective acaricide/insecticide for the control of mites and hoppers in a wide range of crops including fruits and vegetables. It was first evaluated by JMPR in 1995 and then in 1999 and 2010 for maximum residue levels, and in 2004 and 2007 for toxicology. The currently standing recommendations of JMPR are as follows: ADI: 0–0.01 mg/kg bw (1995), ARfD: 0.02 mg/kg bw (2007), and residue definition: Fenpyroximate for compliance with the MRL and for estimation of dietary intake (both for animal and plant commodities). The residue is fat soluble (1999).

Fenpyroximate was scheduled at the 48th session of the CCPR for Periodic Re-evaluation for residues and toxicology by 2017 JMPR. The meeting received information from manufacturer on the metabolism of fenpyroximate in crops, rotational crop studies, metabolism in animals, environmental fate in soil and water, method of residue analysis, stability in stored analytical samples, use patterns, supervised residue trials, fate of residue during storage and processing, and livestock feeding studies.

IDENTITY

ISO common:	fenpyroximate
Chemical name:	
IUPAC:	tert-butyl (*E*)-alpha-(1, 3-dimethyl-5-phenoxypyrazol-4-ylmethyleneamino-oxy)-*p* toluate
CAS:	(*E*)-1, 1-dimethylethyl 4-[[[[(1, 3-dimethyl-5-phenoxy-1H-pyrazol-4-yl) methylene] amino] oxy]methyl]benzoate
CAS Registry No:	134098-61-6
CIPAC No:	695
Synonyms and trade names:	NNI-850
Molecular formula : $C_{24}H_{27}N_3O_4$	
Structural fomular :	



Molecular mass:	421.5 g/mol

PHYSICAL AND CHEMICAL PROPERTIES

The information on chemical and physical properties of pure and technical material of fenpyroximate is shown in Table 1.

Table 1 The chemical and physical properties of fenpyroximate pure and technical material

Property	Results	Reference
Melting point	Melting range 100 to 101 °C (purity 98.6%)	Krips, H.J.,2001a (Report: PC-4037)
Boiling point	Evaporation of the test substance was observed at temperatures above 215 to 219 °C, but reaction or decomposition of the test substance started in the same temperature range. Therefore no boiling point can be evaluated. (purity 98.6%)	Krips, H.J.,2001a (Report: PC-4037)
Temperature of decomposition	Evaporation of the test substance was observed at temperatures above 215 to 219 °C, but reaction or decomposition of the test substance started in the same temperature range. (purity 98.6%)	Krips, H.J.,2001a (Report: PC-4037)
Relative density	Pure fenpyroximate at 20 °C is 1.25 g/cm^3 (purity 98.6)	Rijsbergen van, L.M., 2001 (Report PC-4033)
Appearance	Pure fenpyroximate: white powder (purity 99.6%) Technical grade fenpyroximate: white crystalline powder (purity 98.6%)	Ota, Y., 2015a (Report: PC-4141) Ota, Y., 2015b (Report: PC-4142) Krips, H.J.,2001b (Report: PC-4039)
Vapour pressure	Pure fenpyroximate at 25 °C is ≤ 9.21 × 10^{-6} Pa (purity 99.6%)	Ota, Y., 2015c (Report: PC-4149)
Volatility at 20ºC	Henry's law constant has been calculated from water solubility and vapour pressure data at 25 °C, and is < 0.0168 Pa.m^3.mol^{-1}.	Murata, S., 2016 (Report: PC-4169)
Solubility in water	Buffer, pH 5: 21.4 ± 1.6 µg/L Buffer, pH 7: 23.1 ± 2.8 µg/L Buffer, pH 9: 29.8 ± 4.6 µg/L All tests at 25 °C, No effect of pH observed. (purity 99.8%)	Hori, K. 1991 (Report: PC-4003)
Solubility in organic solvents at 25ºC	Solubility at 20 °C (purity 99.6%): Toluene: 199 g/L Methanol: 13.4 g/L Acetone: 116 g/L Ethyl acetate: 148 g/L Heptane: 3.71 g/L	Furutani, E., 2015a (Report: PC-4150)
Partition coefficient	Partition coefficient: log Pow 5.01 (20 °C) (purity 99.9%) Partition coefficient: log Pow 5.70 (25 °C, pH 6.4-6.5) (purity 99.6%)	Kudo, M., 2001 (Report: PC-4009) Furutani, E., 2015b (Report: PC-4156)
Dissociation constant	The test item does not dissociate as the UV spectra in acid, neutral and alkaline solution are identical. (purity 99.6%)	Furutani, E., 2015c (Report: PC-4147)
Photochemical degradation	Fenpyroximate half-life of 1.5 hours (pH 7 at 25 °C, with 73 hours of a Xenon arc lamp). Fenpyroximate half-life: 13.6 hours (pH 4), 115.5 hours (pH 7) and 99 hours (pH 9) at 25 °C with illuminance of 4000 l× for periods up to 360 minutes hours.	Swanson, M.B, 1993 (Report: E-4015) Zhao, Y., Li, J., Xi, P., XU, R., Duan, Y. Yang, H., Tan, H, 2014 (Scientific paper)

Property	Results	Reference
Hydrolysis	Fenpyroximate's half-lives at 25 °C in the dark for periods of up to 30 days: 180 days (pH 5), 226 days (pH 7) and 221 days (pH 9) Fenpyroximate's half-life at 25 °C with illuminance of 4000 lx for periods up to 360 hours: 13.6 hours (pH 4), 115.5 hours (pH7) and 99 hours (pH 9)	Saxena, A., McCann, D., 1992 (Report: E-4013)

Formulations

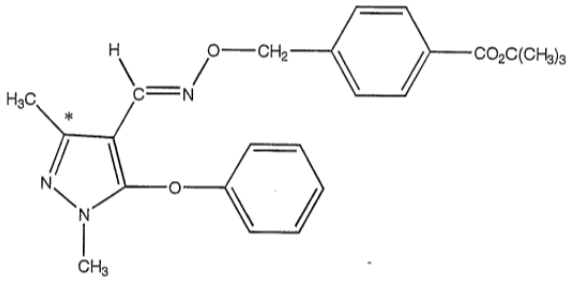
Fenpyroximate is primarily available as suspension concentrates containing 51.2 g ai/L, 5.25% fenpyroximate and 53 g ai/L fenpyroximate.

METABOLISM AND ENVIRONMENTAL FATE

Radiolabel Position

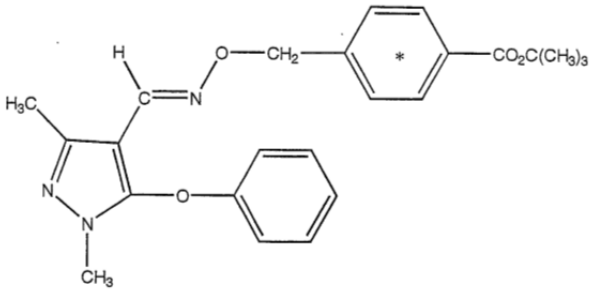
Table 2 Radiolabel Position and Chemical Structure of Test Compound

[pyrazole-3-^{14}C]-Fenpyroximate:



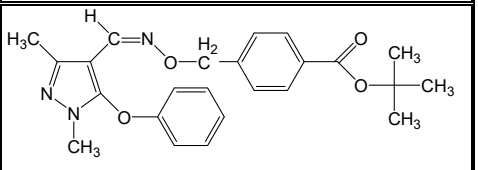
* indicates position of radiolabelling

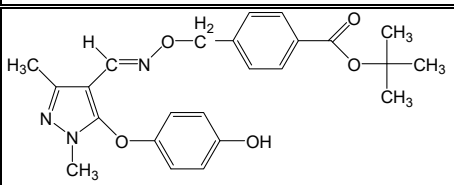
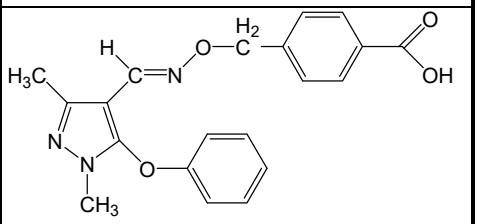
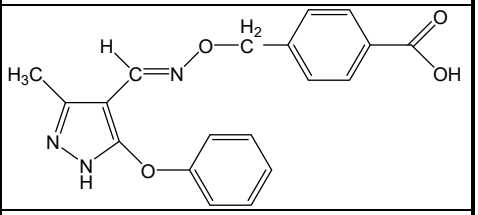
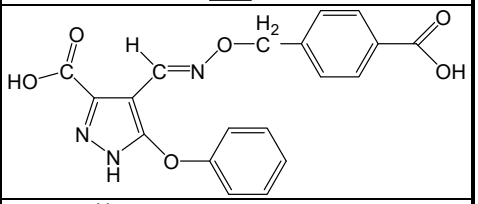
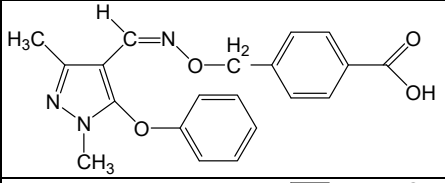
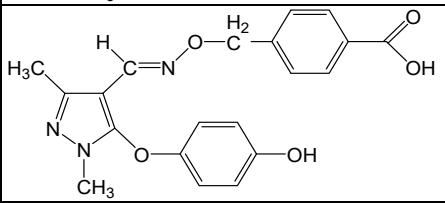
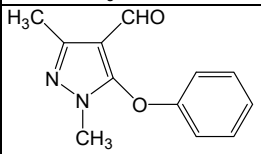
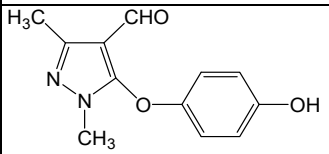
[benzyl-(U)-^{14}C]-Fenpyroximate:

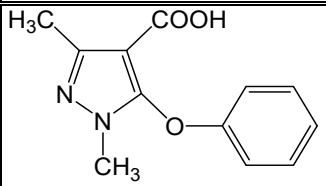
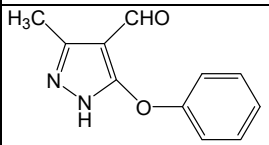
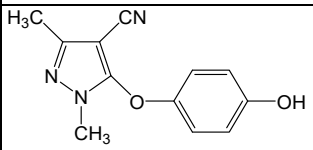
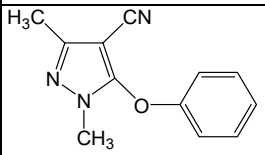
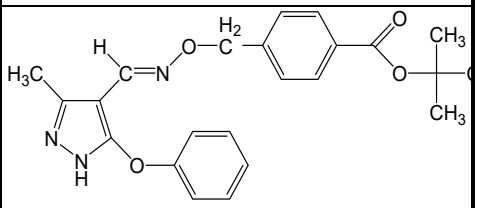
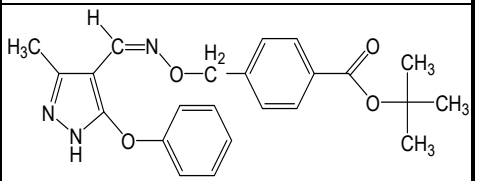
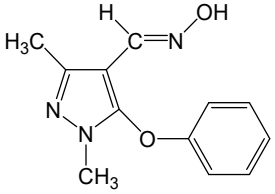


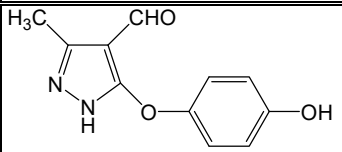
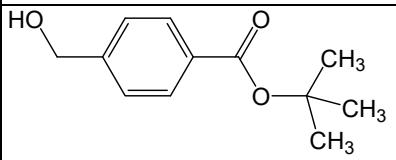
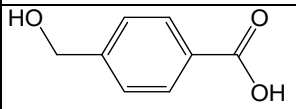
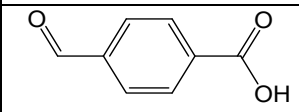
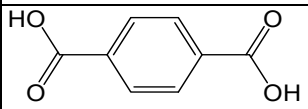
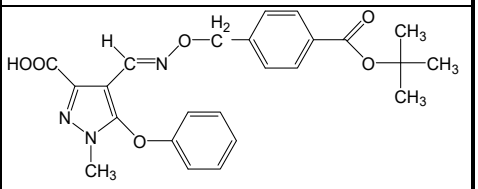
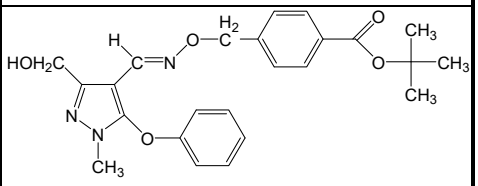
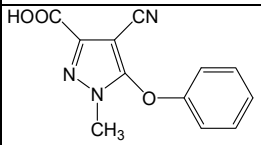
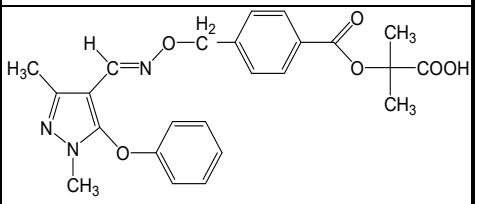
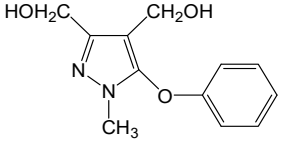
* indicates position of radiolabelling

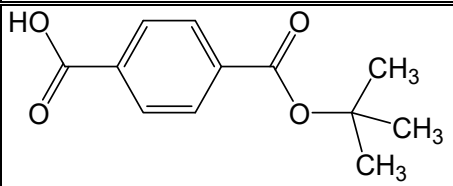
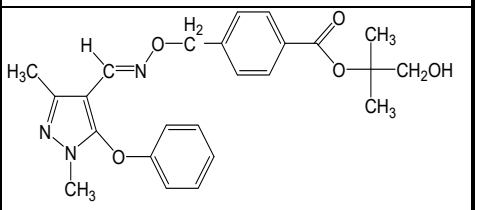
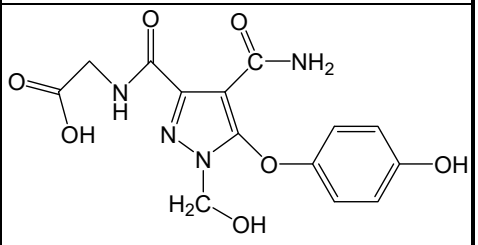
Table 3 Metabolite Codes and Their Related Chemical Structures

Code Number (Synonyms)	Description	Compound found in:	Structure
M-1 (*Z*-isomer)	IUPAC: tert-butyl (*Z*)-α-(1,3-dimethyl-5-phenoxypyrazol-4-ylmethyleneamino-oxy)-*p*-toluate CAS: 1,1-dimethylethyl (*Z*)-4-[[[[(1,3-dimethyl-5-phenoxy-1*H*-pyrazol-4-yl)methylene]amino]oxy]methyl]benzoate	Soil photolysis, aqueous photolysis, surface water, citrus leaves, citrus rind, apple leaves, apple fruit, grape leaves, grape fruit, snap beans, Swiss chard leaves, goat, rotated crops, rat	

Code Number (Synonyms)	Description	Compound found in:	Structure
M-2	Tert-butyl (*E*)-4-{[1,3-dimethyl-5-(4-hydroxyphenoxy)pyrazol-4-yl]-methyleneaminooxymethyl}benzoate	Citrus leaves, rat	
M-3	(*E*)-4-[(1,3-dimethyl-5-phenoxypyrazol-4-yl)methyleneaminooxymethyl]benzoic acid	Aerobic soil, anaerobic soil, groundwater, surface water, sediment, apple leaves, goat, rotated crops, rat	
N-desmethyl M-3	(*E*)-4-[(3-methyl-5-phenoxypyrazol-4-yl)methyleneaminooxymethyl]benzoic acid	Goat	
N-desmethyl M-3 acid	(*E*)-4-[(3-carboxy-5-phenoxypyrazol-4-yl)methyleneaminooxymethyl]benzoic acid	Goat	
M-4	(*Z*)-4-[(1,3-dimethyl-5-phenocypyrazol-4-yl) methyleneaminooxymethyl]benzoic acid		
M-5	(*E*)-4-{[(1,3-dimethyl-5-(4-hydroxyphenoxy)pyrazol-4-yl]methyleneaminooxymethyl}benzoic acid	Goat (also present as phenoxy glucuronide), rotated crops, rat	
M-6	1,3-dimethyl-5-phenoxypyrzole-4-carbaldehyde	Apple leaves, grape leaves, rat	
M-7	1,3-dimethyl-5-(4-hydroxyphenoxy)pyrzole-4-carbaldehyde		

Code Number (Synonyms)	Description	Compound found in:	Structure
M-8	1,3-dimethyl-5-phenoxypyrazole-4-carboxylic acid	Aerobic soil, anaerobic soil, surface water, sediment, apple leaves, grape leaves, rotated crops, rat	
M-9	3-methyl-5-phenoxypyrzole-4-carbaldehyde	Citrus leaves, apple leaves, grape leaves, rat	
M-10	1,3-dimethyl-5-(4-hydroxyphenoxy)pyrzole-4-carbonitrile	Rat	
M-11	1,3-dimethyl-5-phenoxypyrazole-4-carbonitrile	Aerobic soil, anaerobic soil, aqueous photolysis, surface water, sediment, apple leaves, grape leaves, rat	
M-12	Tert-butyl (*E*)--4-[(3-methyl-5-phenoxypyrazol-4-yl)methyleneaminooxymethyl]benzoate	Soil photolysis, citrus leaves, citrus rind, apple leaves, grape leaves, rotated crops, rat	
M-12 isomer	Tert-butyl (*Z*)-4-[(3-methyl-5-phenoxypyrazol-4-yl)methyleneaminooxymethyl]benzoate	Soil photolysis	
M-13	(*E*)-1,3-dimethyl-5-phenoxypyrazole-4-carbaldehyde oxime	Rat, citrus leave	

Code Number (Synonyms)	Description	Compound found in:	Structure
M-14	3-methyl-5-(4-hydroxyphenoxy)pyrzole-4-carbaldehyde	Rat	
M-15	Tert-butyl 4-hydroxymethylbenzoate	Citrus leaves, rat	
M-16	4-hydroxymethylbenzoic acid	Rat	
M-17	4-formylbenzoic acid	Citrus leaves, citrus rind, rat	
M-18	Terephthalic acid	Rat	
M-19	(*E*)-4-[(4-tert-butoxycarbonylphenyl)methyloxyiminomethyl]-1-methyl-5-phenoxypyrazole-3-carboxylic acid	Citrus leaves, apple leaves, grape leaves	
M-20	Tert-butyl (*E*)-4-[(3-hydroxymethyl-1-methyl-5-phenoxypyrazol-4-yl)methyleneaminooxymethyl]benzoate	Citrus leaves, apple leaves, grape leaves	
M-21	4-cyano-1-methyl-5-phenoxypyrazole-3-carboxylic acid	Goat, rat	
M-22	(*E*)-2-[4-[(1,3-dimethyl-5-phenoxypyrazol-4-yl)methyleneaminooxymethyl]benzoyloxy]-2-methylpropanoic acid	Goat, rat	
M-24	3, 4-di(hydroxymethyl)-1-methyl-5-phenoxy-1*H*-pyrazole	Apple leaves	

Code Number (Synonyms)	Description	Compound found in:	Structure
MTBT	Mono-(tert-butyl) terephthalate	Soil photolysis, surface water, sediment	
Fen-OH	2-hydroxymethyl-2-propyl (*E*)-4-[(1,3-dimethyl-5-phenoxypyrazol-4-yl)-methylenaminooxymethyl]benzoate	Goat	
Metabolite 2	2-(5-(4-hydroxyphenoxy)-1-(hydroxymethyl)-1*H*-pyrazole-4-carbamoyl-3-carbonylamino)acetic acid	Goat	

Plant metabolism

The meeting received information on metabolism studies of fenpyroximate after foliar spray (representative use patterns) in citrus, apples, grapes (Fruit crop group), snap beans and cotton (Pulses and Oilseeds crop group) and Swiss chard (Leafy crop group) labelled either in the pyrazole or the benzyl ring.

Citrus (Crop group Citrus fruits)

In one study, 5–6 years old Satsuma mandarin tangerine (*Citrus unshiu*) trees (Krautter G.R., Downs J., Gibson B.S., Lawrence L.J. 1989, report No. R-4003) were treated with [pyrazole-^{14}C] fenpyroximate (specific activity 23.02 mCi/mM, radiochemical purity > 99%, mixed with aliquots of an emulsion concentrate containing 5% fenpyroximate, at the application rate of 22.4 ± 1.5 mg [^{14}C] fenpyroximate /tree for phase I trees (sampled at 0-28 DAT) and 33.5 ± 0.5 mg [^{14}C] fenpyroximate /tree for phase II trees (sampled at normal harvest, 137 DAT）. 10 phase I trees received 61.9 ± 4.3 µCi/tree and two trees were harvested at time of 0, 3, 7, 14 and 28 days after treatment. Phase II trees received 704.7 ± 11.2 µCi/tree and were harvested 137 days after treatment at maturity. Radioactive residues in samples of fruits, leaves were quantified by combustion and liquid scintillation spectroscopy. Samples were transferred to the study for further identification of metabolite (Funayama S., 1990, report No. R-4005). Citrus leaves, fruit and rind were extracted with 2 × acetone/methanol (1:1) and were combusted with an automatic sample combustion system for radioassay. The metabolites were analysed with two-dimensional thin-layer chromatography. Radioactive spots observed by TLC were compared with UV spots of fenpyroximate and its authentic compounds for metabolite identification. Radioactivity at the origin on TLC plates developed with solvent system C was defined as polar metabolites. The polar metabolites were extracted and analysed with enzyme hydrolysis with β-glucosidase or cellulose (Suzuki T., 1995, report no. R-4101).

The residue levels of [^{14}C] fenpyroximate equivalents found in phase I leaves and rind ranged from 1.78 to 5.54 and 0.48 to 0.63 mg eq/kg. No residues were detected in phase one fruit from any

collection period where the detection limit was 0.03 mg eq/kg. The residue levels in Phase II leaves and rind were 1.37 and 0.36 mg eq/kg. Phase II fruit had a residue level of 0.004 mg eq/kg, where the detection limit was 0.004 mg eq/kg. The results are summarised in Table 4

Table 4 Residue levels in leaves, rind and fruit (pulp) of tangerine trees treated with [pyrazole-^{14}C]-fenpyroximate

Days after treatment	Matrix	Mean residues (mg equiv/kg)[a]	Limit of detection [b] (mg eq/kg)
0	Leaves	5.33	0.04
	Rind	0.49	0.19
	Fruit	< 0.03	0.03
3	Leaves	5.54	0.04
	Rind	0.63	0.19
	Fruit	0.025	0.03
7	Leaves	3.37	0.04
	Rind	0.52	0.19
	Fruit	0.01	0.03
14	Leaves	2.27	0.04
	Rind	0.48	0.19
	Fruit	0.02	0.03
28	Leaves	1.78	0.04
	Rind	0.49	0.19
	Fruit	0.02	0.03
137	Leaves	1.37	0.005
	Rind	0.36	0.025
	Fruit	0.0043	0.004

[a] Average calculated from 2 trees for day 0-28 (phase I study) and 3 trees for day 137 (phase II study)

[b] calculated as (2x background dpm–background dpm)/specific activity/sample size (0.1g for rind, 0.3g for fruit, 0.5g for leaves)

The extracted radioactivity in citrus leaves just after [^{14}C]-fenpyroximate application amounted to 99.1% of the total radioactivity and the concentration was 5.28 mg eq/kg (fenpyroximate equivalents). At day 7, 28 and 137 the extracted radioactivity in the leaves amounted to 76.6, 74.7 and 76.6% of the residual radioactivity. More than 90% of the residual radioactivity in the fruit rind was extracted.

In the leaves the level of fenpyroximate was 4.88 mg eq/kg just after application and then decreased to 0.24 mg eq/kg 137 days after application. In the fruit rind the level of fenpyroximate was 0.44 mg eq/kg just after application and then decreased to 0.12 mg eq/kg 137 days after application. Major metabolites in citrus leaves and rind were M-1 and M-12. Concentrations of M-1 and M-12 were 0.13 and 0.08 mg eq/kg in the leaves, and 0.02 and 0.06 mg eq/kg in the fruit rind at harvest, respectively.

Polar metabolite hydrolysis with β-glucosidase or cellulase revealed M-20 glucoside and several glucosides to exist in the leaves and the fruit rind. Among them, Cp-10 (4-hydroxymethyl-1-methyl-5-phenoxypyrazole-3-ylmethyl β-D-glucopyranoside, the glucose conjugate of M-24) was one of the major products both in leaves and in fruit rind. In the leaves, Cp-10 accounted for 11% (0.12 mg eq/kg) of the extracted radioactivity at harvest. The concentrations of fenpyroximate and metabolites in citrus leaves and rind after application of [^{14}C]-fenpyroximate are summarised in Tables 5 and 6.

Table 5 Concentration of fenpyroximate and its metabolites in citrus leaves after foliar application of [pyrazole-^{14}C]-fenpyroximate

Fenpyroximate and its metabolites	Residues (mg fenpyroximate equivalents/kg fresh weight)					
	Days after treatment (phase I study)					Phase II study
	0	3	7	14	28	137
Fenpyroximate	4.88 (92)	3.37 (61)	1.01 (30)	0.56 (25)	0.51 (29)	0.24 (18)
M-1	0.23 (4)	0.29 (5)	0.15 (4.5)	0.23 (10)	0.25 (14)	0.13 (9)

Fenpyroximate and its metabolites	Residues (mg fenpyroximate equivalents/kg fresh weight)					
	Days after treatment (phase I study)					Phase II study
	0	3	7	14	28	137
M-2	n.d.	n.d.	n.d.	n.d.	n.d.	< 0.01
M-3	n.d.	< 0.06	< 0.06	< 0.06	< 0.06	< 0.01
M-6	n.d.	< 0.06	< 0.06	< 0.06	< 0.06	0.01 (0.7)
M-8	n.d.	< 0.06	< 0.06	< 0.06	< 0.06	< 0.01
M-9	n.d.	0.20 (3.6)	0.13 (3.9)	0.08 (3.5)	< 0.06	< 0.01
M-11	n.d.	< 0.06	< 0.06	< 0.06	< 0.06	< 0.01
M-12	0.12 (2)	0.44 (7.9)	0.27 (8)	0.09 (4)	< 0.06	0.08 (5.8)
M-13	n.d.	< 0.06	< 0.06	< 0.06	< 0.06	< 0.01
M-14	n.d.	n.d.	n.d.	n.d.	n.d.	< 0.01
M-19	n.d.	< 0.06	< 0.06	< 0.06	< 0.06	< 0.01
M-20	n.d.	n.d.	n.d.	0.06 (2.6)	< 0.06	0.01 (0.7)
Others	n.d.	0.10 (1.8)	0.60 (18)	0.10 (4.4)	0.12 (6.7)	0.03 (2.2)
Origin	0.05 (1)	0.49 (8.8)	0.42 (12)	0.41 (18)	0.45 (25)	0.55 (40)
Total Extracted	5.28 (99) 2)	4.89 (88.3)	2.58 (76.6)	1.53 (67.4)	1.33 (74.7)	1.05 (76.6)
Non-extracted	0.05 (0.9)	0.65 (11.7)	0.79 (23.4)	0.74 (32.6)	0.45 (25.3)	0.32 (23.4)
Total-^{14}C	5.33	5.54	3.37	2.27	1.78	1.371

n.d.–not detected; Values in parenthesis represent % of the total radioactivity in each sample

Table 6 Concentration of fenpyroximate and its metabolites in citrus fruit rind after foliar application of [pyrazole-^{14}C]-fenpyroximate

Fenpyroximate and its metabolites	Residues (mg fenpyroximate equivalents/kg fresh weight)					
	Days after treatment (phase I study)					Phase II study
	0	3	7	14	28	137
Fenpyroximate	0.44 (90)	0.52 (83)	0.42 (81)	0.32 (67)	0.30 (61)	0.12 (33)
M-1	0.05 (10)	0.03 (4.8)	0.02 (3.8)	0.05 (10)	0.02 (4.1)	0.02 (5.5)
M-2	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
M-3	n.d.	< 0.02	< 0.02	< 0.02	< 0.02	< 0.01
M-6	n.d.	< 0.02	< 0.02	< 0.02	< 0.02	< 0.01
M-8	n.d.	< 0.02	< 0.02	< 0.02	< 0.02	< 0.01
M-9	n.d.	< 0.02	< 0.02	< 0.02	< 0.02	< 0.01
M-11	n.d.	< 0.02	< 0.02	< 0.02	< 0.02	0.01 (2.8)
M-12	n.d.	< 0.02	< 0.02	< 0.02	0.02 (4.1)	0.06 (17)
M-13	n.d.	< 0.02	< 0.02	< 0.02	< 0.02	< 0.01
M-14	n.d.	n.d.	n.d.	n.d.	n.d.	< 0.01
M-19	n.d.	n.d.	n.d.	n.d.	n.d.	< 0.01
M-20	n.d.	n.d.	n.d.	n.d.	n.d.	< 0.01
Others	n.d.	0.03 (4.8)	0.02 (3.8)	0.02 (4.2)	0.06 (12)	0.02 (5.5)
Origin	n.d.	0.04 (6.3)	0.04 (7.7)	0.07 (15)	0.06 (12)	0.10 (28)
Extracted-^{14}C	0.49 (100)	0.62 (98.4)	0.50 (96.2)	0.46 (95.8)	0.46 (93.9)	0.33 (91.9)
Unextracted-^{14}C	0.00 (0)	0.01 (1.6)	0.02 (3.8)	0.02 (4.2)	0.03 (6.1)	0.03 (8.1)
Total-^{14}C	0.49	0.63	0.52	0.48	0.49	0.361

n.d.–not detected; values in parentheses represent % of total radioactive residues in each sample;

In a second study (Funayama S., 1991, report No. R-2006), 3 year old citrus trees (*Citrus unshu* variety *Okitsuwase*) were treated with [benzyl-^{14}C]-fenpyroximate. Ten phase I trees (sampled 0–28 DAT) were sprayed 10 μCi of 50 mg eq/kg solution (20 mL/tree) and four phase II trees (sampled at normal harvest, 98 DAT) were sprayed 35 μCi of 50mg equiv/kg solution (20 mL/tree). Citrus leaves and fruits were taken from two trees at day 0, 3, 7, 14 and 28 after treatment for phase I study, and from four trees at day 98 for phase II study. The fruit was peeled and the fruit rind and edible fruit were separated. Citrus leaves and fruit were extracted with acetone/methanol (1:1) and were combusted with an automatic sample combustion system for radioassay. The metabolites were analysed with two-dimensional thin-layer chromatography. Radioactive spots observed by TLC were compared with UV spots of fenpyroximate and its authentic compounds for metabolite identification. Radioactivity at the origin on TLC plates developed with solvent system C was defined as polar

metabolites. The polar metabolites were extracted and further analysed with enzyme hydrolysis (β-glucosidase or cellulase).

The radioactivity levels in citrus leaves and fruit rind was 9.80 and 1.13 mg eq/kg at day 0. Radioactivity levels in the pulps were all less than 0.01 mg eq/kg. The radioactivity in the leaves from the phase I study was 4.23 and 2.47 mg eq/kg at day 14 and 28. The radioactivity level in the leaves at day 98 had decreased to 0.86 mg eq/kg. The radioactivity level in fruit rind was 0.87 mg eq/kg at day 28 and 0.21 mg eq/kg at day 98. Detailed results are summarised in Table 7.

Table 7 Extraction of radioactivity in citrus leaves, pulp and rind after foliar application of [benzyl-^{14}C]-fenpyroximate

Citrus samples	Average concentration (mg fenpyroximate equivalents /kg fresh weight) [1), 2)]					
	Phase I study (days post treatment)					Phase II study
	0	3	7	14	28	98
Leaves						
Extracted-^{14}C	9.77 (100)	9.11 (99)	6.30 (98)	4.08 (96)	2.32 (94)	0.62 (72)
Unextracted-^{14}C	0.03 (<1)	0.06 (1)	0.10 (2)	0.15 (4)	0.15 (6)	0.24 (28)
Total-^{14}C	9.80	9.17	6.40	4.23	2.47	0.86
Rind						
Extracted-^{14}C	1.12 (99)	1.24 (99)	0.98 (96)	1.05 (94)	0.76 (87)	0.17 (81)
Unextracted-^{14}C	0.01 (1)	0.01 (1)	0.04 (4)	0.08 (6)	0.11 (13)	0.04 (19)
Total-^{14}C	1.13	1.25	1.02	1.13	0.87	0.21
Pulp [3)]						
Total-^{14}C	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01 [4)]

[a] Average calculated from n=2 for phase I study, and n=4 from phase II study

[b] Values in parenthesis represent % of the total radioactivity in each sample

[c] Radioactivity in fruits were measured as $^{14}CO_2$ by combustion

[d] Average of two values only

In the leaves the level of fenpyroximate was 9.75 mg eq/kg just after application and decreased to be 1.07 mg eq/kg at day 28. At day 98 the residue level of fenpyroximate in the leaves was 0.21 mg eq/kg. In the fruit rind the level of fenpyroximate was 1.12 mg eq/kg just after application and then decreased to 0.09 mg eq/kg after 98 days.

The major metabolites in leaves and fruit rind were M-1 and M-12. M-1 level in leaves showed a maximum of 2.03 mg eq/kg at day 7 and then decreased to be 0.14 mg eq/kg at day 98. M-12 level became a maximum of 0.21 mg eq/kg at day 3 and 7 and then gradually decreased to 0.07 mg eq/kg at day 98. The fenpyroximate level was 1.12 mg eq/kg just after spray, 0.81 mg eq/kg at day 7 and 0.53 mg eq/kg at day 28. The sum of fenpyroximate, M-1 and M-12 accounted for 69% of residue radioactivity in the leaves and 83% of that in the fruit rind at day 98, respectively. Two new metabolites, M-15 and M-17 were identified in this study. They were detected maximum of 0.04 mg eq/kg at day 7 and 0.16 mg eq/kg at day 14, respectively, after fenpyroximate application, and then decreased to be less than 0.01 mg eq/kg afterward. All other metabolites found have already been identified in the [pyrazole-^{14}C]-fenpyroximate metabolism study. Hydrolysis of polar metabolites with β-glucosidase or cellulase gave M-15 glucoside and several unknown glucosides at a level in the leaves and fruit rind of less than 0.01 mg eq/kg at day 98. The concentrations of fenpyroximate and metabolites in citrus leaves and rind after application of [^{14}C]-fenpyroximate are summarised in Tables 8 and 9.

Table 8 Concentration of fenpyroximate and its metabolites in citrus leaves after foliar application of [benzyl-^{14}C]-fenpyroximate

Fenpyroximate and its metabolites	Concentration (mg fenpyroximate equivalents/kg fresh weight)					
	Phase I study (days after treatment)					Phase II study
	0	3	7	14	28	98
Fenpyroximate	9.75 (100)	6.40 (70)	3.09 (48)	1.75 (41)	1.07 (43)	0.21 (24)
M-1	0.02 (<1)	1.85 (20)	2.03 (30)	1.29 (32)	0.49 (20)	0.14 (16)
M-2	n.d.	0.04 (<1)	0.03 (<1)	0.02 (<1)	< 0.01	< 0.01

Fenpyroximate and its metabolites	Concentration (mg fenpyroximate equivalents/kg fresh weight)					
	Phase I study (days after treatment)					Phase II study
	0	3	7	14	28	98
M-3	n.d.	0.02 (<1)	0.01 (<1)	0.02 (<1)	< 0.01	< 0.01
M-12	n.d.	0.21 (2)	0.21 (3)	0.14 (3)	0.09 (4)	0.07 (8)
M-15	n.d.	< 0.01	0.04 (<1)	0.03 (<1)	< 0.01	< 0.01
M-16	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
M-17	n.d.	0.03 (<1)	0.14 (2)	0.16 (4)	< 0.01	< 0.01
M-18	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
M-19	n.d.	< 0.01	0.05 (<1)	0.06 (1)	< 0.01	< 0.01
M-20	n.d.	0.07 (<1)	0.05 (<1)	0.02 (<1)	< 0.01	< 0.01
Others	n.d.	0.33 (4)	0.38 (6)	0.29 (7)	0.15 (6)	0.02 (2)
Origin	< 0.01	0.16 (2)	0.27 (4)	0.30 (7)	0.52 (21)	0.18 (21)
Extracted-^{14}C	9.77 (100)	9.11 (99)	6.30 (98)	4.08 (96)	2.32 (94)	0.62 (72)
Non-extracted-^{14}C	0.03 (<1)	0.06 (1)	0.10 (2)	0.15 (4)	0.15 (6)	0.24 (28)
Total-^{14}C	9.80	9.17	6.40	4.23	2.47	0.86
n.d.–not detected; values in parentheses represent % of total radioactive residues;						

Table 9 Concentration of fenpyroximate and its metabolites in citrus fruit rind after foliar application of [benzyl-^{14}C]-fenpyroximate

Fenpyroximate and its metabolites	Concentration (mg fenpyroximate equivalents//kg fresh weight)				
	Phase I study (days after treatment)				Phase II study
	0	7	14	28	98
Fenpyroximate	1.12 (100)	0.81 (80)	0.83 (73)	0.53 (61)	0.09 (43)
M-1	n.d.	0.04 (4)	0.05 (4)	0.02 (2)	0.01 (5)
M-2	-	-	-	-	< 0.01
M-3	-	-	-	-	< 0.01
M-12	n.d.	0.12 (12)	0.12 (11)	0.10 (11)	0.04 (19)
M-15	-	-	-	-	< 0.01
M-16	-	-	-	-	n.d.
M-17	n.d.	n.d.	n.d.	n.d.	< 0.01
M-18	-	-	-	-	n.d.
M-19	-	-	-	-	< 0.01
M-20	-	-	-	-	< 0.01
Others	-	-	-	-	0.01 (5)
Origin	-	-	-	-	0.02 (10)
Extracted-^{14}C	1.12 (99)	0.98 (96)	1.05 (94)	0.76 (87)	0.17 (81)
Unextracted-^{14}C	0.01 (1)	0.04 (4)	0.08 (6)	0.11 (13)	0.04 (19)
Total-^{14}C	1.13	1.02	1.13	0.87	0.21

n.d.–not detected; values in parentheses represent % of total radioactive residues

In a third study (Krautter G.R., Downs J., Gibson B.S., Lawrence L.J., 1988, report No. R-4004), 5 years old Dancy tangerine trees (*Citrus nobilis*) were treated with [pyrazole-^{14}C]fenpyroximate (specific activity 14.6 mCi/mM, radiochemical purity 99%) at the rates of 21.2 mg [^{14}C]fenpyroximate/tree for phase I trees and 32.0 mg [^{14}C]fenpyroximate/tree for phase II trees. Phase I trees received 38.2 µCi/tree and 2 trees were harvested 0, 1, 7, 14 and 28 days post treatment. Phase II trees received 409.6 µCi/tree and 3 trees were harvested at maturity (65 days after treatment). Radioactive residues in samples of leaves, whole fruit were quantified by combustion and liquid scintillation spectroscopy. The citrus leaves and rind were extracted twice with acetone/methanol, the metabolites were identified by two dimensional TLC using authentic reference standards (Funayama S., 1991, report No. R-4007).

In Phase I trees, residue levels of 11.24–13.76 and 0.96–1.36 mg eq/kg were found in leaves and rind, respectively. No residues were detected in Phase I fruit for any collection period (LOD was 0.13 mg equiv/kg). In Phase II trees residue levels at the time of harvest were 14.263, 1.026 and 0.022 mg eq/kg in leaves, rind and fruit pulp, respectively. Results are shown in Table 10.

Table 10 Mean residue levels in leaves, rind and fruit (pulp) of tangerine trees treated with [pyrazole-^{14}C]-fenpyroximate, expressed as mg eq/kg

Matrix	Sample day					
	0	1	7	14	28	65
Leaves	13.76	12.24	12.07	13.24	11.24	14.263
Rind	0.96	0.97	1.04	1.36	1.10	1.026
Pulp	ND	ND	ND	ND	ND	0.022

ND not detected

Extractability of radioactivity from leaves ranged 70-96% and approximately 90% from rind. Fourteen radioactive areas were identified by TLC of which 7 were identified. The major radioactive areas corresponded to fenpyroximate, M-1, and M-12; the other identified metabolites were M-3, M-6, M-8, M-9, M-11, M-13 and M-19. Results are presented in Table 11 and 12.In leaves, the level of fenpyroximate was 12.02 mg eq/kg just after application and 5.25 and 3.63 mg eq/kg after 7 and 28 days, respectively. The half-life of fenpyroximate in the leaves from Phase I was calculated to be 18.2 days. In leaves from Phase II the residue levels of fenpyroximate, M-1 and M-12 were 4.44, 1.73 and 0.41 mg eq/kg, respectively and, in total, accounted for 65% of the extracted radioactivity at day 65. Radioactive material remaining at the origin accounted for 19% extracted radioactivity and the levels of other metabolites were < 0.02 mg eq/kg. In rind at day 65 the levels of fenpyroximate, M-1, M-12 and M-3 were 0.60, 0.13, 0.04 and 0.04 mg eq/kg, respectively and were considered as the major metabolites, accounting for 87% of the extracted radioactivity. M-6 was detected at 0.02 mg eq/kg and the level of other metabolites was < 0.02 mg eq/kg.

Table 11 Extraction of radioactivity in citrus leaves and fruit rind after foliar application of [pyrazole-^{14}C]-fenpyroximate

Citrus samples	Average concentration (mg equiv/kg fenpyroximate equivalents/fresh weight) [a, b]				
	Phase I study (days post treatment)				Phase II study
	0	7	14	28	137
Leaves					
Extracted-^{14}C	13.21 (96.0)	8.47 (70.2)	9.80 (74.0)	9.08 (80.8)	10.15 (71.2)
Unextracted-^{14}C	0.55 (4.0)	3.60 (29.8)	3.44 (26.0)	2.16 (19.2)	4.11 (28.8)
Total-14C	13.76	12.07	13.24	11.24	14.263
Rind					
Extracted-^{14}C	0.88 (91.8)	0.94 (90.7)	1.25 (91.9)	0.99 (89.8)	0.92 (89.1)
Unextracted-^{14}C	0.08 (8.2)	0.10 (9.3)	0.11 (8.1)	0.11 (10.2)	0.11 (10.9)
Total-^{14}C	0.96	1.04	1.36	1.10	1.026

[a] Average calculated from n=2 for phase I study, and n=3 from phase II study

[b] Values in parenthesis represent % of the total radioactivity in each sample

Table 12 Concentration of fenpyroximate and its metabolites in citrus leaves after application of [pyrazole-^{14}C]-fenpyroximate

Fenpyroximate and its metabolites	Concentration (mg equiv/kg fenpyroximate equivalents/fresh weight)					
	Phase I study (days post treatment)				Phase II study	
	Leaves				Leaves	Rind
	0	7	14	28	65	65
Fenpyroximate	12.02 (87)	5.25 (43)	4.21 (32)	3.63 (32)	4.44 (31)	0.60 (58)
M-1	ND	1.19 (10)	1.08 (8)	1.82 (16)	1.73 (12)	0.13 (13)
M-3	ND	< 0.08	< 0.08	< 0.08	< 0.02	0.04 (4)
M-6	ND	< 0.08	< 0.08	< 0.08	< 0.02	0.02 (2)
M-8	ND	< 0.08	< 0.08	< 0.08	< 0.02	< 0.02
M-9	ND	< 0.08	< 0.08	< 0.08	< 0.02	< 0.02
M-11	ND	ND	ND	ND	< 0.02	< 0.02
M-12	ND	0.34 (3)	0.29 (2)	0.36 (3)	0.41 (3)	0.04 (4)
M-13	ND	0.08 (<1)	0.10 (1)	0.18 (2)	< 0.02	< 0.02
M-19	ND	ND	ND	ND	< 0.02	< 0.02
Others	ND	0.51 (4)	2.16 (16)	0.82 (7)	1.64 (11)	0.09 (9)

Fenpyroximate and its metabolites	Concentration (mg equiv/kg fenpyroximate equivalents/fresh weight)					
	Phase I study (days post treatment)				Phase II study	
	Leaves				Leaves	Rind
	0	7	14	28	65	65
Origin	1.19 (9)	1.10 (9)	1.96 (15)	2.27 (20)	1.93 (14)	< 0.02
Extracted-^{14}C	13.21 (96.0)	8.47 (70.2)	9.80 (74.0)	9.08 (80.8)	10.15 (71.2)	0.92 (89.1)
Non-extracted-^{14}C	0.55 (4.0)	3.60 (29.8)	3.44 (26.0)	2.16 (19.2)	4.11 (28.8)	0.11 (10.9)
Total-^{14}C	13.76	12.07	13.24	11.24	14.263	1.026

n.d.–not detected; values in parentheses represent % of total radioactive residues

Following foliar spray application of fenpyroximate to citrus trees, radioactive residues were mainly present in the leaves and rind with residues in fruit close to or below the limit of detection. The major metabolites identified were fenpyroximate, fenpyroximate isomer M-1 and metabolite M-12 with more polar metabolites detected. Fenpyroximate is proposed to be metabolised in citrus tree by hydrolysis of ester, oxime ether cleavage, N-demethylation, oxidation or conjugation to polar metabolites. An overall metabolic pathway for citrus can be found in Figure 1.

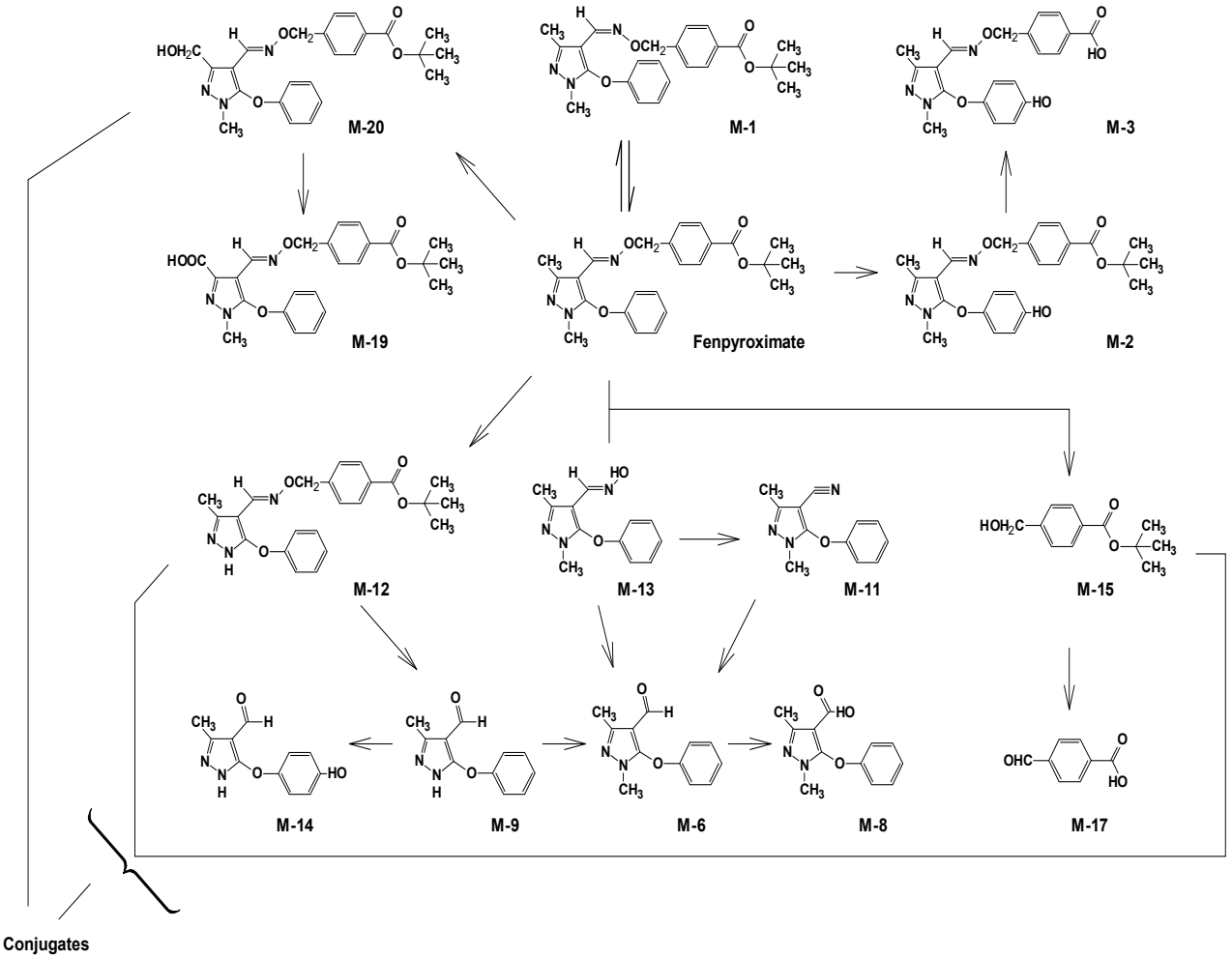


Figure 1 Proposed metabolic pathway of fenpyroximate in citrus rind and leaves

Apple (crop group pome fruit)

The meeting received 2 studies on metabolism of fenpyroximate in/on apple. In the first study (Galicia H., Wyss-Benz M., 1992, report No. R-4008), [pyrazole-^{14}C]-fenpyroximate was applied as

formulated test article (SC 5 %) once to apple trees by overtop spraying at a field rate of 7.5 g ai/100 L (application rate 500 L/ha per m crown height). Apple leaves and fruits were sampled at 0, 7, 14, 28 and 57 days after treatment. The last sampling interval represented mature apples. Samples were rinsed with acetone/water (9:1) solution and the radioactivity was determined by liquid scintillation counting (LSC). Homogenised apples were separated into juice and cake. The homogenised material was extracted with acetone/methanol (1:1), bi-distilled water and methanol and exhaustively under Soxhlet conditions with methanol. Combustion of solid residues was carried out to determine non-extracted radioactivity. The characterisation of the radioactive fractions was carried out with one dimensional thin layer chromatography on silica gel plates and on reversed phase in at least two different solvent systems and co-chromatography with reference compounds.

Residues in leaves amounted to 10.331 mg eq/kg at day 0 and decreased to 0.512 mg eq/kg leaves at harvest (day 57). The residues in apple fruits were 0.128 mg eq/kg at day 0 and 0.032 mg eq/kg at day 57. In apple leaves fenpyroximate and M-1 were the most abundant metabolites. 28 days after application, fenpyroximate and M-1 levels in leaves were 1.18 and 0.35 mg eq/kg, respectively. They decreased to 0.24 and 0.10 mg eq/kg by 57 days after application. Other minor metabolites detected at 28 days after application were: M-3, M-6, M-8, M-9, M-11, M-12, M-13, M-19, M-20 and M-24, these decreased to 0.01 mg eq/kg or less at 57 days after application. In fruit at 57 days after application, only fenpyroximate and M-1 were detected at 0.02 and 0.01 mg eq/kg, respectively. The other metabolites present in leaves were below the limit of detection (0.01 mg eq/kg) in fruit. Enzyme hydrolysis caused a slight increase in concentrations of M-8, M-9 and M-12 to 0.02-0.04 mg eq/kg in the 28 day leaf samples. There were no differences between hydrolysed and control fruit samples. A summary of ^{14}C-residue data in leaves and in fruits is given in Table 13.

Table 13 Residues in apple leaves and fruits after [pyrazole-^{14}C]-fenpyroximate application

Fraction	Concentration (mg parent equivalent/kg fresh weight)				
	Sampling (days post treatment)				
	0	7	14	28	57
Apple Leaves (combined organic phased from surface washing and extraction)					
Fenpyroximate	9.875 (96)	4.238(81)	1.075(59)	1.567(59)	0.221(43)
M-1	0.425(4)	0.369(7)	0.341(19)	0.499(19)	0.091(18))
Unknown-1	-	0.017(< 1)	0.025(1)	0.082(3)	0.024(5)
Unknown-2	-	0.331(6)	0.084(5)	0.170(6)	0.080(16)
Unknown-3	0.025(< 1)	-	0.143(8)	-	-
Unknown-4	-	0.124(2)	-	-	-
Unknown-5	-	0.019(< 1)	0.028(2)	0.087(3)	0.012(2)
Unknown-6, 7, 8, 9	-	-	-	-	-
Aqueous phase	0.005(< 1)	0.084(2)	0.078(4)	0.162(6)	0.054(11)
Total extracted	10.33(100)	5.183(100)	1.774(98)	2.566(97)	0.481(94)
Non-extracted	0.001(0.01)	0.037(<1%)	0.035(2)	0.068(3)	0.031(6)
Total	10.331	5.220	1.809	2.634	0.512
Washings	9.573	4.134	1.152	1.755	0.024
Apple Fruits (combined organic phased from surface washing and extraction)					
Fenpyroximate	0.119(93)	0.094(93)	0.062(77)	0.043(70)	0.015(47)
M-1	0.007(5)	0.009(8)	0.009(11)	0.008(13)	0.005(18)
Unknown-1	-	-	-	0.001(2)	-
Unknown-2	-	-	-	0.004(7)	0.001(3)
Unknown-3	-	-	0.003(4)	--	-
Unknown-4, 5, 6, 7, 8	-	-	-	-	-
Unknown-9	-	0.001(1)	-	-	-
Aqueous phase	-	0.002(2)	0.003(4)	0.003(5)	0.005(16)
Juice	0.001(1)	0.001(1)	0.001(1)	0.002(3)	0.003(9)
Total extracted	0.128(100)	0.108(100)	0.081(100)	0.06(98)	0.031(97)
Non-extracted	-	-	-	0.001(2)	0.001(3)
Total	0.128	0.108	0.081	0.061	0.032
Washings	0.122	0.094	0.064	0.042	< 0.001

-=not detected or below 0.001 mg parent equivalent/kg leaves or apples (fresh weight), values in parentheses represent % of total radioactive residues

Radioactive fractions having similar Rf-values at TLC were pooled to give the respective unknown fractions.

Unknown-1: Radioactive fractions WA3, OAC3, WL3, OL3 OAC6, WL4, OL5

Unknown-2: Radioactive fractions WA4,

Unknown-3: Radioactive fractions WA5, OAC9, WL5, OL6

Unknown-4: Radioactive fractions WL6

Unknown-5: Radioactive fractions OL4

Unknown-6: Radioactive fractions OAC4

Unknown-7: Radioactive fractions OAC5

Unknown-8: Radioactive fractions OAC7

Unknown-9: Radioactive fractions OAC8

Abbreviations: WL–washing leaves, WA–washing apples, OL–organic phase after extraction of leaves, AP–aqueous phase, OAC–organic phase from cake of apples

The identification of metabolites in washings and extracts of fruits and leaves samples harvested 28 and 57 DAT was further conducted (Nishizawa H., 1995, report No.: R-4100) by 2D-TLC and enzyme hydrolysis using beta-glucosidase. The metabolites identified were shown Table 14.

Table 14 Concentration of fenpyroximate and its metabolites in apple leaves and fruits after [[pyrazole-^{14}C]-fenpyroximate application

Fenpyroximate and its metabolites	Concentration (mg equiv/kg fenpyroximate equivalents/fresh weight)		
	Apple		
	Leaves		Fruit
	28 day	57 day	57 day
Fenpyroximate	1.18	0.24	0.02
M-1	0.35	0.10	0.01
M-2	n.d.	n.d.	n.d.
M-3	0.03	n.d.	< 0.01
M-6	0.01	n.d.	< 0.01
M-8	0.06	n.d.	n.d.
M-9	0.03	n.d..	< 0.01
M-10	n.d.	n.d.	n.d.
M-11	0.04	0.01	n.d.
M-12	0.04	n.d.	n.d.
M-13	trace	n.d.	< 0.01
M-14	n.d.	n.d.	n.d.
M-19	0.07	0.01	< 0.01
M-20	0.02	n.d.	< 0.01
M-21	n.d.	n.d.	< 0.01
M-24	0.01	0.01	< 0.01
U-1	0.09	0.02	n.d.
U-2	n.d.	n.d.	< 0.01
Others	0.48	0.02	< 0.01

n.d.–not detected

In the second study (Wyss-Benz M., Mamouni A., 1992a, report No. R-4009), [benzyl-^{14}C]-fenpyroximate was applied once to two apple trees by overtop spraying at a field rate of 7.5 g ai/100L (application rate 500 L/ha per m crown height). One tree separated from the others served as control. Apple leaves and fruits were sampled at 0, 7, 14, 28 and 57 days after treatment. The last sampling interval represented mature apples. Samples were rinsed with acetone/water (9:1) solution and the radioactivity was determined by liquid scintillation counting (LSC). Homogenised apples were separated into juice and cake. The homogenised material was extracted with acetone/methanol (1:1), bi-distilled water and methanol and exhaustively under Soxhlet conditions with methanol. Combustion of solid residues was carried out to determine non-extracted radioactivity. The characterisation of the radioactive fractions was carried out with one dimensional thin layer chromatography on silica gel plates and on reversed phase in at least two different solvent systems and co-chromatography with reference compounds.

Residues in leaves amounted to 12.212 mg parent equivalents/kg leaves at day 0 and decreased to 0.628 mg/kg leaves at harvest (day 57). The residues in fruits were 0.120 mg/kg at day 0 and 0.036 mg/kg apple at day 57. Residues in leaves were much higher than residues in fruits.

Fenpyroximate and its isomer M-1 were the only radioactive components detected at notable levels, numerous other minor metabolites were present at detectable levels in leaves and at levels approaching the limit of detection in fruit. A summary of ^{14}C-residue data in leaves and in fruits is given in Table 15.

Table 15 Residues in apple leaves and fruits after [benzyl-^{14}C]-fenpyroximate application

Fraction	Concentration (mg parent equivalent/kg fresh weight)				
	Sampling (days post treatment)				
	0	7	14	28	57
Apple **Leaves** (combined organic phased from surface washing and extraction)					
Fenpyroximate	11.371(93)	5.310(79)	1.815(70)	1.284(53)	0.219(35)
M-1	0.511(42)	0.906(13)	0.446(17)	0.613(25)	0.161(26)
Unknown-1	-	-	-	-	0.015(2)
Unknown-2	-	0.405(6)	0.064(2)	0.215(9)	0.080(13)
Unknown-3	0.314(3)	-	0.104(4)	-	-
Unknown-4	-	-	0.020(1)	0.052(2)	0.025(4)
Unknown-5	-	-	0.011(<1)	0.029(1)	0.012(2)
Unknown-6	-	0.027(<1%)	0.023(1)	0.046(2)	0.017(3)
Unknown-7	-	0.017(<1%)	0.016(1)	0.016(1)	0.017(3)
Unknown-8	-	-	-	-	-
Aqueous phase	0.016(<1)	0.026(<1)	0.034(1)	0.069(3)	0.049(8)
Total extracted	12.211(100)	6.69(100)	2.533(98)	2.325(97)	0.595(95)
Non-extracted	0.001(<1)	0.028(<1)	0.042(2)	0.082(3)	0.033(5)
Total	12.212	6.718	2.575	2.407	0.628
Washings	11.562	5.683	1.957	1.652	0.013
Apple **Fruits** (combined organic phased from surface washing and extraction)					
Fenpyroximate	0.109(91)	0.113(81)	0.086(78)	0.053(71)	0.017(47)
M-1	0.005(4)	0.017(12)	0.013(12)	0.011(15)	0.007(19)
Unknown-1	-	0.001(1)	-	-	0.001(3)
Unknown-2	0.004(3)	0.004(3)	0.003(3)	0.003(4)	0.001(3)
Unknown-3,4, 5, 6, 7, 8	-	-	-	-	-
Aqueous phase	0.002(2)	0.004(3)	0.004(4)	0.003(4)	0.007(19)
Juice	-	-	0.001(1)	0.001(1)	0.002(6)
Total extracted	0.120(100)	0.140(100)	0.109(99)	0.074(99)	0.034(94)
Non-extracted	-	-	0.001(1)	0.001(1)	0.002(6)
Total	0.120	0.140	0.110	0.075	0.036
Washings	0.115	0.120	0.085	0.050	< 0.001

-=not detected or below 0.001 mg parent equivalent/kg leaves or apples (fresh weight); values in parentheses represent % of total radioactive residues;

Radioactive fractions having similar Rf-values at TLC were pooled to give the respective unknown fractions.

Unknown-1: Radioactive fractions WA3, OAC3, WL3 Unknown-2: Radioactive fractions WA4, OAC4, WL4, OL5

Unknown-3: Radioactive fractions WL5, OL6 Unknown-4: Radioactive fractions OL3

Unknown-5: Radioactive fractions OL4 Unknown-6: Radioactive fractions OL7

Unknown-7: Radioactive fractions OL8 Unknown-8: Radioactive fractions OAC5

Abbreviations: WL–washing leaves, WA–washing apples, OL–organic phase after extraction of leaves, AP–aqueous phase, OAC–organic phase from cake of apples

Grape

The meeting received 3 studies on metabolism of Fenpyroximate in/on grape. In one study (Wyss-Benz M., Mamouni A., 1992b, report No.: R-4010), [Pyrazole-^{14}C]-fenpyroximate was applied as formulated test article (SC 5) once to two grapevines by hand spraying at a field rate of 7.5 g ai/100L (application rate 500 L/ha per m crown height). Leaves and grape fruit were sampled at 0, 7, 14, 28 and 57 days after treatment. The last sampling interval represented mature grapes. Samples were rinsed with acetone/water (9:1) solution and the radioactivity was determined by liquid scintillation counting (LSC). Grapes (without stem) were homogenised and separated into juice and cake by centrifugation. Aliquots of cake were combusted. Homogenised material was extracted with acetone/methanol (1:1), bi-distilled water and methanol and exhaustively under Soxhlet conditions

with methanol. One dimensional thin layer chromatography on silica gel plates and on reversed phase in at least two different solvent systems and co-chromatography with reference compounds was used to characterise the radioactive fractions. The identification of metabolites was conducted by 2D TLC. (Nishizawa H., 1995, report No.: R-4100).

Residues in leaves amounted to 6.234 mg parent equivalents/kg leaves at day 0 and decreased to 0.971 mg/kg leaves at harvest (day 57). The residues in grape fruits were 0.097 mg/kg at day 0 and 0.081 mg/kg at day 57. Juice of grapes was not extracted as ^{14}C-residues were lower than 0.01 mg/kg grapes over the whole sampling period. At all sampling times, ^{14}C-fenpyroximate was the main radioactive fraction and the only fraction present at day 0 (0.096 mg/kg grapes). A summary of ^{14}C-residue data in leaves and in grape bunches is given in Table 16.

Table 16 Residues in grape leaves and fruits after [pyrazole-^{14}C]-fenpyroximate application

Fraction	Concentration (mg parent equivalent/kg fresh weight)				
	Sampling (days post treatment)				
	0	7	14	28	57
Grape **Leaves** (combined organic phased from surface washing and extraction)					
Fenpyroximate	5.750（92）	3.125(71)	1.553(53)	1.475(50)	0.326(34)
M-1	0.335（5）	0.175(4)	0.124(4)	0.112(4)	0.052(5)
Unknown-1	-	0.503(11)	0.450(15)	0.314(11)	0.144(15)
Unknown-2	0.084（1）	0.234(5)	0.088(3)	0.143(5)	0.058(6)
Unknown-3	0.029（<1）	-	0.192(7)	0.086(3)	0.027(3)
Unknown-4	-	-	0.024(1)	0.033(1)	0.020(2)
Unknown-5	-	0.021(<1)	0.017(1)	-	0.019(2)
Unknown-6	-	0.045(1)	0.010(<1)	0.005(<1)	0.016(2)
Unknown-7	-	-	0.014(<1)	0.011(<1)	0.002(<1)
Unknown-8, 9, 10	-	-	-	-	-
Aqueous phase	0.028(<1)	0.066(2)	0.116(4)	0.273(9)	0.137(14)
Non-extracted	0.007(<1)	0.218(5)	0.329(11)	0.478(16)	0.169(17)
Total	6.234	4.413	2.917	2.930	0.971
Washings	5.626	3.582	2.130	1.874	0.348
Grape Bunches (combined organic phased from surface washing and extraction)					
Fenpyroximate	0.096(99)	0.140(72)	0.067(66)	0.028(55)	0.031(38)
M-1	-	0.004(2)	0.004(4)	0.002(4)	0.004(5)
Unknown-1	-	0.001(1)	-	0.002(4)	0.002(2)
Unknown-2	-	-	-	-	-
Unknown-3	-	0.015 (8)	0.010(10)	0.002(4)	0.016(20)
Unknown-4	-	-	-	-	-
Unknown-5	-	0.001(1)	0.001(1)	-	0.001(1)
Unknown-6	-	-	-	-	0.001(1)
Unknown-7	-	-	-	-	-
Unknown-8	-	-	-	-	0.001(1)
Unknown-9, 10	-	-	-	-	-
Aqueous phase	0.001(1)	0.014(7)	0.011(11)	0.008(16)	0.010(12)
Juice	-	0.005(3)	0.005(5)	0.004(8)	0.007(9)
Non-extracted	-	0.004(2)	0.004(4)	0.003(6)	0.006(7)
Total	0.097	0.195	0.102	0.051	0.081
Washings	0.088	0.136	0.055	0.022	0.032

-=not detected or below 0.001 mg parent equivalent/kg leaves or grape bunches (fresh weight), values in parentheses represent % of total radioactive residues;

Radioactive fractions having similar Rf-values at TLC were pooled to give the respective unknown fractions.

Unknown-1: Radioactive fractions WG2, OS3, OL3 Unknown-2: Radioactive fractions OS7, WL4, OL6

Unknown-3: Radioactive fractions WG3, OGC4, WL5, OL7 Unknown-4: Radioactive fractions OS5, OL4

Unknown-5: Radioactive fractions OGC3, OS6, OL6 Unknown-6: Radioactive fractions OGC5, OL8

Unknown-7: Radioactive fractions OS10, OL9 Unknown-8: Radioactive fractions OGC6, OS8

Unknown-9: Radioactive fractions OS4 Unknown-10: Radioactive fractions OS9

Abbreviations: WL–washing leaves, WG–washing grape bunches, OL–organic phase after extraction of leaves, AP–aqueous phase, OGC–organic phase from cake of grapes, OS–organic phase from stems

The identification of metabolites in washings and extracts of fruits and leaves samples harvested 28 and 57 DAT was further conducted (Nishizawa H., 1995, report No.: R-4100) by 2D TLC and enzyme hydrolysis using beta-glucosidase. The metabolites identified were shown in the following Table. The residues of M-9, M-12 and M-19 in leaves at 57 days after application were detected at 0.01, 0.01 and 0.02 mg eq/kg, respectively. In fruit only fenpyroximate and M-1 were detected at 0.02 and 0.01 mg eq/kg, respectively. The other metabolites were below the limit of detection (0.01 mg eq/kg). Results are presented in Table 17

Table 17 Concentration of fenpyroximate and its metabolites in grape leaves and fruit after [pyrazole-^{14}C]-fenpyroximate application

Fenpyroximate and its metabolites	Concentration (mg equiv/kg fenpyroximate equivalents/fresh weight)		
	Grapes		
	Leaves		Fruit
	28 day	57 day	57 day
Fenpyroximate	1.24	0.31	0.02
M-1	0.24	0.06	0.01
M-2	n.d.	n.d.	n.d.
M-3	n.d.	trace	< 0.01
M-6	0.01	trace	< 0.01
M-8	0.01	n.d.	n.d.
M-9	0.01	0.01	n.d.
M-10	n.d.	n.d.	n.d.
M-11	trace	n.d.	< 0.01
M-12	0.02	0.01	< 0.01
M-13	n.d.	n.d..	< 0.01
M-14	n.d.	n.d..	< 0.01
M-19	0.03	0.02	< 0.01
M-20	0.01	trace	< 0.01
M-21	n.d.	n.d.	< 0.01
M-24	n.d.	trace	< 0.01
U-1	0.01	trace	< 0.01
U-2	trace	0.01	n.d.
Others	0.89	0.27	0.03

n.d.–not detected

In another study (Wyss-Benz M., Mamouni A., 1992c, report No.: R-4011), [Benzyl-^{14}C]-fenpyroximate was applied as formulated test article (SC 5 %) once to two grapevines by hand spraying at a field rate of 7.5 g ai/100L (application rate 500 L/ha per m crown height). Leaves and fruits were sampled at 0, 7, 14, 28 and 57 (maturity) days after treatment. Samples were rinsed with acetone/water (9:1) solution and the radioactivity was determined by liquid scintillation counting (LSC). Grape fruits (without stem) were homogenised and separated into juice and cake by centrifugation. The homogenised material was extracted with acetone/methanol (1:1), bi-distilled water and methanol and exhaustively under Soxhlet conditions with methanol. The radioactive fractions were characterised with one dimensional thin layer chromatography on silica gel plates and on reversed phase in at least two different solvent systems and co-chromatography with reference compounds.

Following foliar spray application of radiolabelled fenpyroximate to grapes, residues were much lower in fruits than in leaves. Residues in leaves amounted to 7.492 mg parent equivalents/kg leaves at day 0 and decreased to 1.158 mg/kg leaves at harvest (day 57). The residues in grape fruits were 0.086 mg/kg bunches of grapes at day 0 and 0.060 mg/kg at day 57. Juice of grapes was not extracted as ^{14}C-residues were lower than 0.010 mg/kg grapes over the whole sampling period. Fenpyroximate and its isomer M-1 were the only radioactive components detected at notable levels, although other minor metabolites were present at detectable levels in leaves and at levels approaching the limit of detection in fruit. At all sampling times, ^{14}C-fenpyroximate was the main radioactive fraction. ^{14}C-fenpyroximate as well as metabolite M-1 slightly decreased their concentrations over the sampling period. Six unknown fractions, OGC3–OGC8, appeared at various times, but remained at 0.002 mg/kg or lower. All samples of stems were extracted, and the residues were all ≤ 0.010 mg/kg.

The same pattern of metabolites was showed in leaves and grape berry. A summary of ^{14}C-residue data in leaves and in grape bunches is given in Table18.

Table 18 Residues in grape leaves and fruits after [benzyl-^{14}C]-fenpyroximate application

Fraction	Concentration (mg parent equivalent/kg fresh weight)				
	Sampling (days post treatment)				
	0	7	14	28	57
Grape **Leaves** (combined organic phased from surface washing and extraction)					
Fenpyroximate	7.019(94)	2.943(72)	1.944(68)	1.492(60)	0.643(56)
M-1	0.308(4)	0.201(5)	0.162(6)	0.109(4)	0.054(5)
Unknown-1	-	0.203(5)	0.203(7)	0.087(4)	0.042(4)
Unknown-2	0.156(2)	0.262(6)	0.166(6)	0.124(5)	-
Unknown-3	-	-	0.104(4)	-	0.132(11)
Unknown-4	-	-	0.075(3)	0.017(1)	-
Unknown-5	-	0.046(1)	0.032(1)	0.036(1)	0.021(2)
Unknown-6, 7	-	-	-	-	-
Unknown-8	-	-	0.009(<1)	0.036(1)	0.006(1)
Unknown-9	-	-	0.011(<1)	-	0.005(<1)
Unknown-10	-	-	-	-	0.009(1)
Aqueous phase	0.006(<1)	0.392(10)	0.242(8)	0.515(21)	0.182(16)
Non-extracted	0.004(<1)	0.033(1)	0.028(1)	0.051(2)	0.064(6)
Total	7.492	4.080	2.872	2.467	1.158
Wahings	6.607	3.030	2.279	1.323	0.783
Grape **Bunches** (combined organic phases from surface washing and extraction)					
Fenpyroximate	0.079(92)	0.109(76)	0.049(65)	0.053(61)	0.027(45)
M-1	0.004(5)	0.013(9)	0.007(9)	0.016(18)	0.006(10)
Unknown-1	-	-	-	-	0.001(2)
Unknown-2	-	0.001(1)	0.002(3)	0.001(1)	0.002(3)
Unknown-3	-	0.008(6)	0.004(5)	0.006(7)	0.006(10)
Unknown-4	-	-	-	-	-
Unknown-5	-	0.001(1)	-	-	0.001(2)
Unknown-6	-	0.001(1)	0.001(1)	0.002(2)	-
Unknown-7, 8, 9, 10	-	-	-	-	-
Aqueous phase	0.001(1)	0.005(3)	0.005(7)	0.007(8)	0.006(10)
Juice	-	0.004(3)	0.005(7)	0.006(7)	0.005(8)
Non-extracted	-	0.001(1)	0.001(1)	0.002(2)	0.002(3)
Total	0.086	0.144	0.075	0.087	0.060*
Washing	0.073	0.101	0.038	0.037	0.021

-=not detected or below 0.001 mg parent equivalent/kg leaves or grape bunches (fresh weight); values in parentheses represent % of total radioactive residues;

* - most of the radioactivity showed the same Rf value as Unknown-1

Radioactive fractions having similar Rf-values at TLC were pooled to give the respective unknown fractions.

Unknown-1: Radioactive fractions WG2, OS3, OL3 Unknown-2: Radioactive fractions OS7, WL4, OL6

Unknown-3: Radioactive fractions WG3, OGC4, WL5, OL7 Unknown-4: Radioactive fractions OS5, OL4

Unknown-5: Radioactive fractions OGC3, OS6, OL6 Unknown-6: Radioactive fractions OGC5, OL8

Unknown-7: Radioactive fractions OS10, OL9 Unknown-8: Radioactive fractions OGC6, OS8

Unknown-9: Radioactive fractions OS4 Unknown-10: Radioactive fractions OS9

Abbreviations: WL–washing leaves, WG–washing grape bunches, OL–organic phase after extraction of leaves, AP–aqueous phase, OGC–organic phase from cake of grapes, OS–organic phase from stems

Snap beans

The snap bean plants (*Phaseolus vulgaris,* grown outdoors) were treated with [Pyrazole-^{14}C] - fenpyroximate at rate of 104 g/ha or [Benzyl-^{14}C]-fenpyroximate at rate of 105 g/ha (Dohn, D. R., 2007, report No. R-4183). Snap beans were harvested seven days after application. The homogenised samples were extracted with acetonitrile: water mixtures and acetonitrile. Radioactive components in the solvent extracts were characterised by reverse phase HPLC and normal phase TLC. Metabolite identification was by co-chromatography with authentic reference standards. The total radioactive residue values in beans were 0.107 to 0.124 mg eq/kg and >9 9% of the residue was extracted with

acetonitrile: water. The primary components of the residue were fenpyroximate, accounting for 85.5 to 88.8% TRR and M-1, accounting for 4.0 to 4.7% TRR. The remaining residue consisted of minor components extracted with acetonitrile: water (< 10% TRR) and a small residue of 0.001 mg eq/kg associated with the post extracted solids. All minor unknown components detected were present at concentrations < 0.01 mg eq/kg. Results are presented in Table 19.

Table 19 Components of radioactive residues in [pyrazole-^{14}C] fenpyroximate treated snap beans

	Pyrazole-^{14}C label		Benzyl-^{14}C label	
Component	mg equiv/kg	% TRR	mg equiv/kg	% TRR
Fenpyroximate	0.106	85.5	0.095	88.8
M-1	0.005	4.0	0.005	4.7
Minor extracted components	0.012[a]	9.7	0.006	5.6
Subtotal extracted	0.123	99.2	0.106	99.1
Post-extracted solids	0001	0.8	0.001	0.9
TRR	0.124	100	0.107	100
Subtotal identified (fenpyroximate and M-1)	0.111	89.5	0.100	93.5

[a] Multiple components. The two most abundant unknowns are both present at 0.003 mg eq/kg (2.4% TRR for each)

Swiss chard

Swiss Chard plants (35 days after the seeding) (Yoshizane T., 2014 report no. LSRC-M14-032A / R-4482) were treated once with [pyrazole-3-^{14}C] or [benzyl-^{14}C]-fenpyroximate at rate of 102.4 g ai/ha. The treated stems and leaves were collected at zero (just after the application), 14 and 35 days after the application, the roots were also collected at 35 days after the application. Sampled stems and leaves were rinsed with acetonitrile, and extracted with acetonitrile/distilled water (4/1, v/v) and acetonitrile/ 0.1N hydrochloric acid (4/1, v/v). Total radioactive residue (TRR) was determined as the sum of radioactivity in rinses, extracts and post extraction solid. Rinses and extracts containing significant radioactivity (> 0.05 mg eq./kg) were subjected to TLC-radioluminography to determine metabolite constituents. Identification of major metabolites including fenpyroximate was qualitatively confirmed by further analysis by HPLC.

The majority of the treated radioactivity was recovered from rinses and extracts (96.4 to 100% of TRR). Radioactive concentration in stems and leaves throughout testing duration ranged from 0.92 to 6.45 mg eq./kg for [pyrazole-3-^{14}C] fenpyroximate and from 0.89 to 7.65 mg eq./kg for [benzyl ring -^{14}C(U)] fenpyroximate, respectively. The most prominent residue in stems and leaves was unchanged fenpyroximate. High polar radioactivity (retaining at TLC origin) accounted for 30.7 to 48.7% TRR. RI-HPLC (radioisotope detection) analysis showed that the polar radioactivity at TLC origin consisted of several individual metabolites, each below 8.5% TRR. No M-3 was detected. M-1 and the other unknown metabolites were also detected at less than 3.8% TRR (0.04 mg/kg) as minor metabolites.

Table 20 Distribution of radioactivity in Swiss chard stems treated with ^{14}C-fenpyroximate

	Day 0		Day 14		Day 35	
	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg
Label	[Pyrazole 3-^{14}C] fenpyroximate					
Rinse + extract	100	6.45	97.5	1.55	96.9	0.90
Rinse	81.1	5.23	15.5	0.25	11.6	0.11
Acetonitrile/DW extract	18.8	1.21	81.6	1.30	84.3	0.78
Acetonitrile/0.1N HCl extract	< 0.1	< 0.01	0.5	< 0.01	1.0	< 0.01
PES	< 0.1	< 0.1	2.5	0.04	3.1	0.03
Total	100	6.45	100	1.59	100	0.92
Root	NS [a]	NS	NS	NS	100	0.08
Label	[Benzyl ring-^{14}C(U)] fenpyroximate					
Rinse + extract	100	7.65	98.2	1.94	96.4	0.86
Rinse	82.7	6.32	16.3	0.32	8.8	0.08
Acetonitrile/DW extract	17.3	1.32	81.6	1.61	86.6	0.77

	Day 0		Day 14		Day 35	
	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg
Acetonitrile/0.1N HCl extract	-	-	0.5	< 0.01	1.0	< 0.01
PES	< 0.1	< 0.01	1.8	0.03	3.6	0.04
Total	100	7.65	100	1.97	100	0.89
Root	NS	NS	NS	NS	100	0.07

[a] NS not sampled; 2) not detected

Table 21 Residues in Swiss chard leaves after ^{14}C-fenpyroximate application

	Day 0		Day 14		Day 35	
	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg
Label	[Pyrazole 3-^{14}C] fenpyroximate					
fenpyroximate	98.9	6.38	44.7	0.71	36.9	0.34
M-1	1.0	0.06	3.5	0.06	3.8	0.04
M-3	-[a]	-	-	-		-
Unk-1	-	-	2.4	0.04	2.7	0.02
Unk-2	-	-	1.8	0.03	1.6	0.01
Unk-3	-	-	2.0	0.03	2.1	0.02
TLC -origin	< 0.1	< 0.01	42.5	0.68	48.7	0.45
MeCN/0.1N HCl extract	< 0.1	< 0.01	0.5	< 0.01	1.0	< 0.01
Unextracted	< 0.1	< 0.01	2.5	0.04	3.1	0.03
Total	100.0	6.45	100.0	1.59	100	0.92
Label	[Benzyl ring-^{14}C(U)] fenpyroximate					
fenpyroximate	99.1	7.57	58.9	1.16	40.1	0.36
M-1	0.9	0.07	3.5	0.07	2.7	0.02
M-3	-	-	-	-	-	-
Unk-1	-	-	2.5	0.05	1.8	0.02
Unk-2	-	-	0.6	0.01	0.5	< 0.01
Unk-3	-	-	1.7	0.03	1.3	0.01
TLC -origin	< 0.1	< 0.01	30.7	0.60	48.9	0.43
MeCN/0.1N HCl extract	-	-	0.5	0.01	1.0	0.01
Unextracted	< 0.1	< 0.01	1.8	0.03	3.6	0.03
Total	100.0	7.65	100.0	1.97	100	0.89

[a] -: not detected

Cotton

The cotton plants were treated once with [pyrazole-3-^{14}C] fenpyroximate as a foliar spray at a rate of 194 g ai/ha (Baker F.C. *et al*, 2001, report no. 742W-1 / R-4116). The cotton forage of immature plants were harvested one week after the application. Mature plants were harvested after boll opening (30 days after application) and were separated into seed, lint and gin trash. Total radioactive residue (TRR) levels in cotton samples were determined by combustion. The cotton samples was extracted using methanol/acetone 1:1, MeOH, water or a combination of solvents including weak and strong acids and bases. Hexane extraction was useful for extracting the high level of fenpyroximate and M-1 from gin trash. Identification of radioactive components was conducted using reversed phase HPLC using UV and radio detection, with co-chromatography with certified reference standards. Certain extracts were also subject to LC/MS analysis to confirm the assignments of M-1 and fenpyroximate as major residues.

The major parts of the treated radioactivity were recovered from rinses and extracts (68-126% of TRR) in the various matrices. The proportion of radioactivity detected in post-extraction solid (PES) was <10% of TRR except in seed kernels (28% TRR) and lint/hulls (15% TRR), although the absolute levels of radioactivity in these samples was very low (0.002–0.003 mg/kg). Radioactive distribution of sprayed test substance in/on cotton is summarised in Tables 22 and 23.

Fenpyroximate and M-1 (approximately equal amounts) were the major components in the immature cotton (forage), cotton seed, and the cotton gin trash. Immature cotton (forage) contained fenpyroximate (2.533 mg eq/kg; 70.3% of TRR) and M-1 (1.151 mg eq/kg; 32.0% of TRR). Metabolites corresponding to the pre-M-8 and M-8 region (see gin trash) accounted for 0.067 mg eq/kg (1.9% of TRR), while other metabolites were all < 0.05 mg eq/kg. These included M-13 region (0.014 mg eq/kg; 0.38% of TRR), M-5 region (0.012 mg eq/kg; 0.33% of TRR) and M-12 region (0.024 mg eq/kg; 0.66% of TRR).

Cottonseed (kernel) contained only metabolites at levels below 0.01 mg eq/kg. Of these, M-1 and fenpyroximate (each 0.003 mg eq/kg and 37.5% of TRR) constituted the major residues. Other metabolites were < 0.001 mg eq/kg. Cotton lint/hulls also contained metabolites at < 0.01 mg eq/kg. A metabolite (0.007 mg eq/kg, 33.3% of TRR) with similar retention time to M-5, and another (0.007 mg eq/kg, 33.3% of TRR) with similar retention time to N-desmethyl M-3, were the major extractable residues. Lesser amounts (0.001 mg eq/kg) of M-1, fenpyroximate, M-8 metabolite region, and a polar metabolite, (solvent front, RT~3.28 min) were also observed. A putative non-polar metabolite that eluted at 47.73 min (after fenpyroximate) contained 0.002 mg eq/kg. However, this metabolite was not detected during later analysis (storage stability) of cotton lint/hulls and it is considered a possible artefact.

Gin trash (subsample A) contained fenpyroximate (3.391 mg eq/kg; 36.8% of TRR) and M-1 (3.405 mg eq/kg; 37.0% of TRR). Many other metabolites were detected in gin trash extracts. These included M-8 (0.190 mg eq/kg; 2.1% of TRR) and a slightly more polar metabolite (pre- M-8, 0.315 mg eq/kg; 3.4% of TRR). Gin trash extracts were explored further in subsample C. Results confirmed a similar level of fenpyroximate (3.357 mg eq/kg; 36.5% of TRR) and M-1 (3.564 mg eq/kg; 38.7% of TRR). Many nonpolar metabolites were detected in the hexane extracts from gin trash subsample C. Of these, fractions designated as NPI - NP5 contained 0.091, 0.179, 0.155, 0.067 and 0.050 mg eq/kg. Other non-polar fractions contained < 0.05 mg eq/kg. NP 1 - NP5 all contained more than a single radiolabelled peak. Because of the high residues of M-I detected in gin trash it is likely that several of the unknown metabolites detected are metabolites of M-1 isomeric with fenpyroximate metabolites. This could account for apparent pairs of radiolabelled peaks observed during reversed phase analysis.

Methanol and MeOH: Water extracts of gin trash subsample C also contained fenpyroximate (0.578 mg eq/kg) and M-1 (0.453 mg eq/kg) as well a number of non-polar peaks (including NPI [M-8 region] and NP3). However, most of the unknown residues in the Methanol/MeOH: Water were relatively polar and eluted near the solvent front (RT 2.8 min., 0.925 mg eq/kg) during reversed phase HHC. The MeOH plus MeOH: H_2O fractions (2.079 mg eq/kg) were partitioned to extract M-1 and fenpyroximate (hexane phase), mid polar metabolites (EtOAc phase) to leave a polar (aqueous) fraction. The EtOAc fraction (1.210 mg eq/kg) was subjected to normal phase TLC and 10 individual radiolabelled zones (1a, 1b and 2-9) were scraped from the plate, dated and further analysed by reverse phase HPLC. A very large number of metabolites were detected following this procedure. However, only zone la/RT 24.2 min. (0.066 mg eq/kg), zone la/RT 47.3 min. (0.074 mg eq/kg), zone la/RT 48.4 min. (0.101 mg eq/kg), zone Ib/RT 48.6 min (0.056 mg eq/kg, more than one peak), zone 3/RT 39.8 min (0.198 mg eq/kg), zone 4/RT 40.3 min (0.063 mg eq/kg), zone 7/RT 41.3 min. (0.124 mg eq/kg; M-8 region) and zone 9/RT 56.0 min. (M-l, 0.052 mg eq/kg) contained > 0.05 mg eq/kg. Attempts to identify unknown metabolites by LC/MS were not successful. Zone 5 contained a number of metabolites < 0.05 mg eq/kg, one of which migrated with similar RT to M-14. The residual aqueous polar fraction also contained several metabolites as determined by C18 SPE fractionation and HPLC. Two polar metabolites (RT 3.2 min., 0.059 mg eq/kg; RT 12 2 min., 0.071 mg eq/kg) were above 0.05 mg eq/kg. Other residues were < 0.05 mg eq/kg. Separate analyses of MeOH and MeOH/water extracts of gin trash subsample C showed at least two components in addition to M-8 that could be methylated with diazomethane to form less polar products Additionally, polar (aqueous residue) extracts from gin trash could be hydrolysed (dilute acid) to produce less polar products. This is indicative of polar conjugates that are hydrolysed to less polar fenpyroximate/M-1 metabolites. Storage stability analysis of gin trash extracts showed the presence of a low level metabolite (< 0.05 mg eq/kg) corresponding to M-12.

Only 1.135 g of field gin trash was available for extraction and analysis. Low recovery of radiolabel was observed (68.5%) which was attributed to the very small sample available. Analysis of the field gin trash extracts by HPLC gave a very similar profile to the composite gin trash extract (from subsample A). This indicated that the composite sample was representative of field gin trash. The HPLC chromatogram of dried leaves extract also was very similar to that from composite and field gin trash. Dried leaves contained the highest TRR (14.629 mg eq/kg). Fenpyroximate (6.336 mg eq/kg) and its isomer M-1 (5.454 mg eq/kg) accounted for 80.6% of the RRR. In gin trash (subsample C) fenpyroximate plus M-1 accounted for 75.2% of the TRR.

The results of this study indicated that fenpyroximate and its Z stereoisomer (M-1) were the major components of the immature cotton (forage), cotton seed, and the cotton gin trash. Generally, fenpyroximate and M-1 were present in approximately equal amounts. Other metabolites such as M-5, M-12, M-14 and N-desmethyl-M-3 were identified by retention time on a single HPLC system at low level.

Table 22 Extraction of radioactivity from cotton matrices treated with ^{14}C-fenpyroximate

	Forage		Seed kernels		Lint/hulls		Leaves		Gin trash (field)		Gin Trash A	
Extract	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg
TRR	3.597 mg/kg		0.008 mg/kg		0.021 mg/kg		14.629 mg/kg		8.383 mg/kg		9.204 mg/kg	
MeOH:acetone 1:1 I	62.7	62.7	47.1	0.004	47.9	0.010	23.0	3.365	25.3	2.121	36.9	3.396
MeOH:acetone 1:1 II	30.0	30.0	18.8	0.002	21.3	0.004	31.0	4.435	10.5	0.880	26.7	2.457
MeOH:acetone 1:1 III	10.5	10.5	15.9	0.001	11.0	0.002	14.9	2.180	3.1	0.260	12.1	1.114
MeOH:acetone 1:1 IV	3.5	3.5	7.4	0.001	6.8	0.001	8.4	1.229	-	-	5.0	0.460
MeOH:acetone 1:1 V	-	-	4.8	0.000	3.7	0.001	5.8	0.848	-	-	2.9	0.267
Water I	0.3	0.3	4.2	0.000	8.6	0.002	4.8	0.702	7.9	0.662	3.7	0.341
Water II	-	-	-	-	-	-	2.5	0.366			1.0	0.092
MeOH I	1.2	1.2	2.0	0.000	1.9	0.000	2.4	0.351	10.6	0.889	1.0	0.092
MeOH II	-	-	-	-	-	-	-	-	3.6	0.302	-	-
0.5N HCl	0.2	0.2	3.6	0.000	1.5	0.000	-	-	-	-	0.6	0.055
0.5M NaOH	0.4	0.4	3.8	0.000	3.7	0.000	-	-	-	-	0.9	0.083
6N HCl	0.3	0.3	-	-	-	-	-	-	-	-	1.0	0.092
3M NaOH	0.5	0.5	-	-	-	-	-	-	-	-	1.4	0.129
PES combustions[a]	0.1	0.1	28.0	0.002	15.2	0.003	10.5	1.536	7.5	0.629	1.9	0.175
Total Extracted[b]	109.6		107.1		106.4		92.8		61.0		93.2	
Total Recovered[c]	109.7		126.3		114.5		103.3		68.5		95.1	

[a] Data obtained from triplicate combustion of the PES

[b] Sum of all extraction steps

[c] Sum of the extraction steps prior to PES combustion and the unextracted radiocarbon determined by PES combustion

Table 23 Extraction of radioactivity from cotton matrices treated with ^{14}C-fenpyroximate

	Gin Trash B		Gin Trash C	
Extract	%	mg/kg	%	mg/kg
TRR	9.204 mg/kg		9.204 mg/kg	
Hexane I	31.1	2.862	31.3	2.881
Hexane II	23.1	2.126	21.8	2.006
Hexane III	9.6	0.884	9.4	0.845
Hexane IV	5.3	0.488	5.7	0.525
Hexane V	2.9	0.267	3.2	0.295

Extract	Gin Trash B		Gin Trash C	
	%	mg/kg	%	mg/kg
TRR	9.204 mg/kg		9.204 mg/kg	
Hexane VI	3.4	0.313	0.4	0.037
MeOH wash	-	-	13.3	1.224
MeOH:water 1:1 I (B) or 9:1 II (C)	5.2	0.479	4.5	0.414
MeOH:water 1:1 II (B) or 9:1 III (C)	6.0	0.552	2.6	0.239
MeOH:water 1:1 III (B) or 9:1 IV (C)	5.8	0.534	1.5	0.138
MeOH:water 1:1 IV (B) or 9:1 V (C)	1.5	0.138	0.7	0.064
MeOH:water 1:1 V (B) or 9:1 VI (C)	0.8	0.074	-	-
PES combustions[a]	9.2	0.847	8.1	0.746
Total Extracted[b]	94.7		94.4	
Total Recovered[c]	103.9		102.5	

[a] Data obtained from triplicate combustion of the PES. [b] Sum of all extraction steps

[c] Sum of the extraction steps prior to PES combustion and the unextracted radiocarbon determined by PES combustion

Table 24 Residues in cotton after ^{14}C-fenpyroximate application

Matrix	% TRR (mg/kg)	Fenpyroximate (mg/kg)	M-1 (mg/kg)	M-8 region (mg/kg)	Other[a]
Forage	3.597	2.533(70.4%)	1.151(32.0%)	0.067(1.9%)	< 0.05
Seed (kernel)	0.008	0.003(37.5%)	0.003(37.5%)	ND	< 0.01
Seed (lint/hulls)	0.021	0.001(4.8%)	0.001(4.8%)	0.001(4.8%)	< 0.01
Composite gin trash A[b]	9.204	3.391(36.8%)	3.405(37.0%)	0.190(2.1%)	>0.05 (5)
Composite gin trash C[c]	9.204	3.357(36.5%)	3.564(38.7%)	0.156(1.7%)	>0.05 (12)[d]
Dried leaves	14.629	6.336(43.3%)	5.454(37.3%)	0.280(1.9%)	>0.05 (7)

[a] Refers to single unidentified metabolites. For example, < 0.05 mg/kg means individual metabolites not present above this level, or >0.05 mg/kg means at least one metabolite present above this level

[b] Based on HPLC analysis of composite sample of MeOH:acetone, water, plus MeOH extracts

[c] Based on HPLC analysis of composite sample of hexane extracts, plus HPLC of composite sample of MeOH and MeOH:water extracts, plus TLC of displaced hexane phase from MeOH extract

[d] 4 Non-polar, 6 mid-polar, 2 polar

Rotational crop studies

Confined rotational crop studies

The meeting received information on two confined rotational crop studies.

In a study, radish (*Raphanus sativus*, variety *Belle Glade*), lettuce (*Lactuca sativa*, variety *Green Towers*) and wheat (*Triticum aestivum L.*, variety RSI-5) were grown as rotational crops in a sandy loam soil treated with [pyrazole-3-^{14}C] fenpyroximate at a rate of 224 g/ha (Baker, F.C., Estigoy, L., Kimmel, E.C. 2001, report No.:R-4123). Crops were sown at an interval of 30, 120 and 365 days following treatment of soil with fenpyroximate. Lettuce and radish were harvested at maturity. Wheat was sampled at the forage and hay growth stages with the remainder being grown to maturity for collection of grain, straw and chaff. Residues in lettuce were ≤ 0.002 mg eq/kg. Residues in radish root and leaves ranged from 0.001 to 0.008 mg eq/kg. Residues wheat forage and hay ranged from 0.005 to 0.047 mg eq/kg. Residues in straw and chaff taken at maturity were 0.018 to 0.106 mg eq/kg and residues in grain were 0.004 to 0.01 mg eq/kg.

Samples of radish root, radish foliage and wheat forage were extracted with neutral organic solvents. Hay, straw and wheat chaff were extracted with an aqueous/solvent mixture and acid followed by base. Wheat grain radiolabel was only partially extracted. Analysis of extracts by HPLC or TLC showed that most individual metabolite residues were < 0.01 mg eq/kg. Fenpyroximate was detected at 0.001 mg eq/kg in radish root and a metabolite (0.001 mg eq/kg) with a retention close to M-1 was observed in radish foliage, otherwise parent or its isomer M-1 did not occur as residues. Wheat forage from the 120 day plant back group contained several metabolites (all < 0.01 mg eq/kg) two of which co-eluted in the region of M-5 and M-3 reference standards. Hay extracts from all plant

back groups each contained many metabolites (all < 0.01 mg eq/kg). In the 30 day plant back group hay neutral extract one of the metabolites migrated with a similar retention to M-21; other metabolites were unidentified. Hay extracts from 120 and 365 day plant back groups contained metabolites that eluted in the region of M-8, M-5 and M-3 reference standards, in addition to several other metabolites. It was not possible to observe mid-polar reference standards co-injected with sample extracts as they were overshadowed due to a high degree of UV absorption from matrix co-extractives. Wheat chaff neutral solvent extracts also contained many metabolites at a level of < 0.01 mg eq/kg. At least four metabolites (described as mid-polar, levels 0.001 mg eq/kg to 0.004 mg eq/kg) were in a group with a retention time of approximately 30 to 36 minutes.

Samples of wheat straw from the 30, 120 and 365 day plant back intervals contained residues > 0.01 mg eq/kg. Straw from the 30 day plant back contained a polar unknown although re-analysis of a stored extract and of a subsequent storage stability study sample suggested that this residue level is probably an overestimate due to solvent or matrix effects on the sample causing early elution of less polar metabolites. Straw from the 120 day plant back contained four metabolites at >0.01 mg eq/kg. These included a polar unknown, and three mid polarity metabolites of which one had a similar retention time to M-8. Results are shown in Tables 25, 26 and 27.

Table 25 Total radioactive residues in crops grown in soil treated with [pyrazole-3-^{14}C] fenpyroximate, expressed as mg eq/kg

Crop/part	Plant pack interval (days after treatment)		
	30	120	365
Mature lettuce	0.001	0.001	0.002
Radish root	0.008	0.006	0.001
Radish foliage	0.007	0.006	0.003
Wheat forage	0.005	0.021	0.004
Wheat hay	0.025	0.047	0.018
Wheat grain	0.010	0.010	0.004
Wheat straw	0.069	0.106	0.032
Wheat chaff	0.044	0.046	0.018

Table 26 Total extractability of radiocarbon expressed as %TRR and mg eq/kg

Crop/part	Plant pack interval (days after treatment)		
	30	120	365
Mature lettuce	NA	NA	NA
Radish root	78.7 (0.006)	NA	NA
Radish foliage	88.9 (0.005)	NA	NA
Wheat forage	117.2 (0.006)	106.2 (0.021)	NA
Wheat hay	96.1 (0.024)	93.9 (0.042)	100.1 (0.018)
Wheat grain	51.7 (0.004)	52.8 (0.005)	NA
Wheat straw	101.4 (0.070)	88.9 (0.094)	94.1 (0.030)
Wheat chaff	91.8 (0.04)	NA	NA

NA Not applicable–not extracted due to low residue

Table 27 Metabolite characterisation

DAT	Sample	TRR (mg equiv/kg)	Extract analysed	TRR in extract (mg equiv/kg)	Metabolite profile
30	Radish root	0.008	Neutral extract [a]	0.006	Fenpyroximate 0.001 mg eq/kg 2 Components each ≤ 0.002 mg eq/kg
	Radish foliage	0.007	Neutral extract [b]	0.005	3 Components each ≤ 0.002 mg eq/kg
120	Wheat forage	0.021	Neutral extract [a]	0.021	6 Components 0.001-0.005 mg eq/kg
30	Wheat hay	0.025	Neutral extract aqueous phase [b]	0.008	1 Component 0.007 mg eq/kg
			Acid extract aqueous phase [b]	0.006	2 Components 0.002 & 0.003 mg eq/kg

DAT	Sample	TRR (mg equiv/kg)	Extract analysed	TRR in extract (mg equiv/kg)	Metabolite profile
			Neutral extract organic phase [b]	0.002	4 Components 0.001-0.005 mg eq/kg
			Acid extract organic phase [b]	0.001	3 Components each ≤ 0.001 mg eq/kg
120	Wheat hay	0.047	Neutral extract [a]	0.010	M-8 0.001 mg eq/kg M-5 0.002 mg eq/kg 5 Components each ≤ 0.002 mg eq/kg
			Acid extract [a]	0.030	M-8 0.005 mg eq/kg M-5 0.002 mg eq/kg M-3 0.001 mg eq/kg 8 Components each ≤ 0.003 mg eq/kg
365	Wheat hay	0.018	Neutral extract [a]	0.010	M-8 0.001 mg eq/kg M-5 0.002 mg eq/kg M-3 0.001 mg eq/kg 4 Components each 0.001 mg eq/kg
			Acid extract [a]	0.008	2 Components 0.001 & 0.004 mg eq/kg
30	Wheat straw	0.069	Neutral extract [a]	0.052	M-8 0.008 mg eq/kg 6 Components 0.001-0.022 mg eq/kg
120	Wheat straw	0.106	Neutral extract [a]	0.081	M-8 0.011 mg eq/kg 7 Components 0.002-0.028 mg eq/kg
365	Wheat straw	0.032	Neutral extract [a]	0.025	10 Components 0.001-0.006 mg eq/kg
30	Wheat chaff	0.044	Neutral extract [a]	0.029	9 Components 0.001-0.006 mg eq/kg

[a] analysed by HPLC;

[b] analysed by TLC

In second study, the uptake and metabolism of [^{14}C]-fenpyroximate in rotational crops (radish, spinach and wheat) was studied at 30, 120 and 270 days after application of [benzyl-U-^{14}C]-fenpyroximate at application rate of 111 g ai/ha (Simmonds, M., Haynes, L., 2016, report No.: R-4513). The plants were harvested at immaturity and maturity. Total radioactive residues (TRR) in all crop matrices were very low. Only wheat hay, straw and grain from Day 31 (1st rotation) and wheat straw and grain from Day 118 (2nd rotation) had TRR values greater than 0.01 mg/kg. At 270 days (3rd rotation), TRRs in all crops were below 0.01 mg/kg.

Extractability of radioactivity from hay, grain and straw at Day 31 with neutral solvents ranged from 19.5–32.8% TRR (0.004–0.011 mg/kg). The remaining radioactivity was treated sequentially with a combined enzyme solution (cellulase/hemicellulase/ β-glucosidase) followed by dilute acid (1 M HCl) and dilute base (1 M NaOH) treatments which released a further 49.4–55.9% TRR (0.007–0.021 mg/kg). Unextracted radioactive residues accounted for 12.3–31.0% TRR (0.002–0.011 mg/kg).

Extractability of radioactivity from straw and grain at Day 118 with neutral solvents was between 17.9–23.6% TRR (0.002 mg/kg). Grain was subjected to sequential extraction with a combined enzyme solution (cellulase/hemicellulase/ β-glucosidase) followed by dilute acid (1 M HCl) and dilute base (1 M NaOH) treatments which released a further 20.8% TRR (0.002 mg.kg). The remaining non-extractable radioactivity accounted for 76.4% TRR (0.007 mg/kg) and 61.4% TRR (0.008 mg/kg) for straw and grain respectively.

Fenpyroximate, Metabolite M-1 (z-isomer) and Metabolite M-3 were tentatively identified in straw. As most radioactivity was extracted from crop matrices following enzyme, acid and base treatment, it was considered that the remaining extracted radioactivity was associated with natural products such as polysaccharides, lignin and starch. A proposed pathway of fenpyroximate metabolism in rotational crops is presented in Figure 2.

Table 28 TRR concentrations in crop after treatment of soil with [^{14}C]-fenpyroximate

Plant Commodity	Harvest	Matrix	TRR (mg/kg fenpyroximate equivalents)		
			Time after application (days)		
			31	118	270
Radish	Mature	Root	0.005	0.001	< 0.001
	Mature	Foliage	0.007	0.002	0.001
Spinach	Immature	Foliage	0.006	0.002	0.001
	Mature	Foliage	0.005	0.002	0.001
Wheat	Immature	Forage	0.009	0.002	0.001
	Immature	Hay	0.012[a]	0.004	0.002
	Mature	Straw	0.037[a]	0.009[a]	0.006
	Mature	Grain	0.037[a]	0.012[a]	0.005

[a] TRR results based on extraction/combustion

Table 29 Extraction of radioactivity from wheat grain, hay and straw after treatment of soil with [^{14}C]-fenpyroximate

Extraction	Matrix									
	31 Day rotation						118 Day rotation			
	Hay		Straw		Grain		Straw		Grain	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Total Radioactive Residue (TRR)	0.012	100.0	0.037	100.0	0.037	100.0	0.009	100.0	0.012	100.0
Extracted radioactivity E1–E8	0.004	32.8	0.011	30.1	0.007	19.5	0.002	23.6	0.002	17.9
NER after E1-E8	0.008	67.2	0.026	69.9	0.029	80.5	0.007	76.4	0.010	82.1
Enzyme treatment	0.004	29.2	0.010	28.4	0.006	16.0	nr	nr	0.001	6.85
1M HCl treatment	0.001	8.59	0.004	9.80	0.001	3.41	nr	nr	0.001	8.68
1M NaOH treatment	0.002	17.2	0.007	17.7	0.011	30.0	nr	nr	0.001	5.24
Total extracted	0.010	87.8	0.032	87.0	0.025	68.9	0002	23.6	0.005	38.7
Remaining NER	0.002	12.3	0.005	14.0	0.011	31.0	0.007	76.4	0.008	61.4
Total	0.012	100.0	0.037	100.0	0.037	100.0	0.009	100.0	0.012	100.0

Table 30: Partitioning of radioactivity from wheat hay and straw concentrates after treatment of soil with [^{14}C]-fenpyroximate

Extraction/Fraction	Matrix			
	Hay[a]		Straw [b]	
	% TRR	mg/kg	%TRR	mg/kg
Extracted radioactivity E1-E4	25.5	0.002	18.8	0.007
Ethyl acetate fraction	2.60	< 0.001	8.25	0.003
Aqueous fraction	22.7	0.002	10.5	0.004
TRR	-	0.009	-	0.038

[a] Overall recovery from partitioning was 85.6%, fractions not analysed further
[b] Overall recovery from partitioning was 83.4%, fractions analysed further

Table 31 Proportions of radioactive components in organosoluble and aqueous fractions of straw extract concentrates: 31 day rotation (TLC system 1)

Metabolite/reference standard		Rf value	Matrix=straw			
			Organic fraction		Aqueous fraction	
			% TRR	mg/kg	% TRR	mg/kg
Chromatographed radioactivity			8.25	0.003	10.5	0.004
1	(Polar fraction)	0.00	1.32	0.001	nd	nd
2	(Fenpyroximate)	0.07	1.58	0.001	nd	nd
3	(M-1/M-12)	0.11	1.63	< 0.001	nd	nd
4	(M-3)	0.23	0.73	< 0.001	nd	nd

Metabolite/reference standard		Rf value	Matrix=straw			
			Organic fraction		Aqueous fraction	
			% TRR	mg/kg	% TRR	mg/kg
5		0.37	1.44	0.001	nd	nd
6		0.51	1.44	0.001	nd	nd
7		0.65	1.11	< 0.001	nd	nd
8		0.79	nd	nd	10.5	0.004
TRR				0.038		0.038

nd: not detected

Table 32 Proportions of radioactive components in organosoluble and aqueous fractions of straw extract concentrates: 31 day rotation (TLC system 5)

Metabolite/reference standard		Rf value	Matrix=straw			
			Organic fraction		Aqueous fraction	
			%TRR	mg/kg	% TRR	mg/kg
Chromatographed radioactivity			8.25	0.003	10.5	0.004
M1		0.01	1.30	0.001	10.5	0.004
M2		0.17	0.26	< 0.001	nd	nd
M3		0.20	0.43	< 0.001	nd	nd
M4		0.40	0.48	< 0.001	nd	nd
M5	(M-3)	0.45	0.95	< 0.001	nd	nd
M6		0.59	0.74	< 0.001	nd	nd
M7	(Fenpyroximate, M-1)	0.87	4.02	0.002	nd	nd
TRR				0.038		0.038

nd: not detected

Table 33 Proportions of radioactive components in organosoluble and aqueous fractions of straw extract concentrates: 31 day rotation (TLC system 7/8)

Metabolite/reference standard		Rf value	Matrix=straw			
			Organic fraction		Aqueous fraction	
			%TRR	mg/kg	% TRR	mg/kg
Chromatographed radioactivity			8.25	0.003	10.5	0.004
Organic fraction						
M1		0.05	1.09	< 0.001	10.5	0.004
M2		0.26	2.49	0.001	nd	nd
M3		0.36	1.10	< 0.001	nd	nd
M4		0.39	1.16	< 0.001	nd	nd
M5		0.47	2.05	0.001	nd	nd
M6		0.55	0.35	< 0.001	nd	nd
Aqueous fraction						
Zone 1		0.20–0.43	-	-	1.48	0.001
Zone 2		0.46–0.76	-	-	5.54	0.002
Zone 3		0.77–0.99	-	-	3.47	0.001
TRR				0.038		0.038

nd: not detected

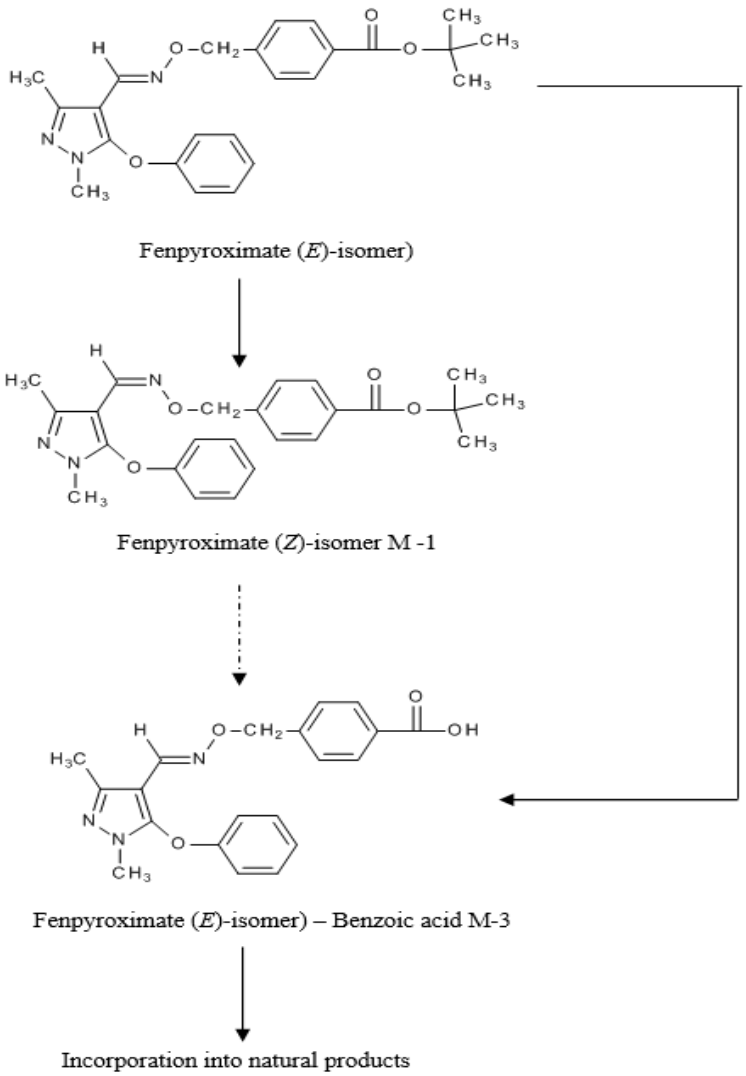


Figure 2 Proposed metabolic pathways of fenpyroximate in rotated crops after ^{14}C-fenpyroximate application

The confined rotational crop studies showed no potential residues of fenpyroximate and its metabolites are expected in rotational crops.

Field Rotational Crop Studies

No information on field rotational crop study was submitted

Animal metabolism

Lactating goats

Two studied on lactating goat metabolism were available for the meeting.

In a study conducted with [pyrazole-3-^{14}C]-fenpyroximate (Jalali K., Gibson N.A. (1999a, report No R-4114), a lactating goat (Nubian/Toggenburg cross bred, approximately 2 years old, 36 kg) was orally administered by capsule twice daily for 3 consecutive days at dose of 10 ppm, corresponding to 0.5 mg/kg bodyweight/day. Milk samples were collected twice daily during the dosing period. The treated goat was sacrificed approximately 22 hours after the final dose. Total radioactive residue (TRR) levels in selected matrices were determined.

TRR levels in liver, kidney, muscle, fat and blood were 1.209, 1.100, 0.021, 0.082 and 0.026 mg eq./kg, respectively. After estimating for the total mass of blood, muscle and fat in a typical goat, total radioactivity recovered in the tissues accounted for 3.3% of the dose. In milk, TRR levels ranged from 0.004 to 0.037 ppm and total radioactivity recovered in the milk accounted for 0.2% of the administered dose.

Urinary and faecal excretion was found to be notable routes of elimination, accounting for 32.1% and 33.3% of the dose, respectively. At sacrifice 10.6% of the dose remained unexcreted in the gastrointestinal tract. Based on the amounts of radioactivity recovered in all the samples analysed, material balance for the study was 79.5% of the administered dose. Results are show in Table 34.

The milk, kidney and liver samples were extracted and most of the radiocarbon in each sample was removed. Results are show in Table 35. Extracts were analysed by HPLC, TLC and LC-MS where possible. Metabolite identification was also carried out in urine and faeces to confirm the assignments made in the tissue and milk samples. The excretion of fenpyroximate in the goat was rapid. A small trace was found in milk, muscle, fat and kidney with faeces containing a large amount of parent. Seven metabolites were identified and/or characterised in milk and tissue samples. An eighth metabolite was the Z-isomer of parent fenpyroximate, referred to as M-1, and was detected in very low amounts in one of the milk samples (56 hour sacrifice). Results are show in Table 36.

Table 34 Residue levels in milk and tissues of goat administered [pyrazole-3-^{14}C]-fenpyroximate at a dose of 0.5 mg fenpyroximate/kg body weight/day for 3 consecutive days

Sample	Collection interval	Limit of quantification (mg eq./kg)	Residue level (mg eq./kg)	Percent of dose (%)	Cumulative percent of dose (%)
Liver	Sacrifice	0.0005	1.209	1.7	
Kidney	Sacrifice	0.0008	1.100	0.2	
Muscle	Sacrifice	0.0008	0.021	0.7	3.3
Fat	Sacrifice	0.0008	0.082	0.6	
Blood	Sacrifice	0.0008	0.026	0.1	
Milk	0-8 h	0.0006	0.004	0.00	
	8-24 h		0.020	0.04	
	24-32 h		0.026	0.03	0.2
	32-48 h		0.028	0.06	
	48-56 h		0.033	0.03	
	56h - sacrifice		0.037	0.07	
Urine	0-24 h	-	-	2.6	
	44-48 h	-	-	7.5	32.1
	48h - sacrifice	-	-	14.7	
Cage wash	Sacrifice	-	-	7.3	
Faeces	0-8 h	-	-	< 0.1	
	8-24 h	-	-	4.6	
	24-32 h	-	-	3.1	
	32-48 h	-	-	7.9	33.2
	48-56 h	-	-	4.0	
	56h - sacrifice	-	-	11.6	
Cage solids	Sacrifice	-	-	2.0	
Gastrointestinal tract		-	-	10.6	10.6
Total Recovery					79.5

Table 35 Extractability and Recovery of Radiocarbon in Goat Tissues and Milk

Sample	TRR(mg eq./kg)	Percent ^{14}C		Percent ^{14}C
		Extracted [a]	Unextracted [b]	Recovered [c]
Milk0-8 Hr	0.004	ND	ND	ND
Milk8-24 Hr	0.020	91.2	2.9	94.1
Milk24-32 Hr	0.026	95.6	2.8	98.4
Milk32-48 Hr	0.028	67.2	3.2	70.4
Milk48-56 Hr	0.033	94.8	1.9	96.7
Milk56 Hr-S[ac]	0.030	79.6	1.2	80.8
Kidney	1.100	100.7	7.0*	107.7

Sample	TRR(mg eq./kg)	Percent ^{14}C		Percent ^{14}C
		Extracted [a]	Unextracted [b]	Recovered [c]
Liver	1.209	93.4	11.5*	104.9
Muscle	0.024	91.9	20.0*	111.9
Fat	0.082	94.5	6.2	100.7

ND Not done due to low TRR.

a Refers to the amount extracted with hexane, ACN and ACN-water 1:1 (liver, kidney and 32-48 hour milk).

b Refers to the amount of unextractable radiocarbon as determined by combustion of the post extraction solids (PES).

c Sum of all extraction steps prior to combustion of the PES, plus the unextracted radiocarbon as determined by combustion of the PES

* TRR in PES was in < 0.001mg eq/kg

Table 36 Metabolite levels (ppm) detected in tissues and milk of goat administered [pyrazole-3-^{14}C]-fenpyroximate at a dose of 0.5 mg fenpyroximate/kg body weight/day for 3 consecutive days

Sample (times given in hours)	Distribution of metabolites (expressed as mg/kg and % of TRR in parenthesis)									
	Total radio-active residue	Fenp	(1)[a] M-21	(2) M-5	(3) N-desmethyl M-3	(4) M-5 Glucur-onide	(5) M-3	(6) Fen-OH	(7) M-22	(8) M-1
0-8 milk	0.004	NA	NA	NA	NA	NA	NA	NA	NA	NA
8-24 milk	0.020	0.001 (5)	0.011 (55)	0.000	0.000	0.000	0.000	0.001 (5)	0.000	0.000
24-32 milk	0.026	0.005 (19)	0.011 (42)	0.000	0.000	0.000	0.000	0.003 (12)	0.000	0.000
32-48 milk	0.028	0.003 (11)	0.010 (36)	0.000	0.000	0.000	0.000	0.002 (7)	0.000	0.000
48-56 milk	0.033	0.008 (24)	0.015 (46)	0.000	0.000	0.000	0.000	0.003 (9)	0.000	0.000
56-sac milk	0.030	0.001 (3)	0.011 (37)	0.000	0.000	0.000	0.000	0.001 (3)	0.000	0.001
Muscle	0.024	0.006 (25)	0.000	0.000	0.000	0.000	0.002 (8)	0.014 (58)	0.000	0.000
Fat	0.082	0.035 (43)	0.000	0.000	0.000	0.000	0.006 (7)	0.029 (35)	0.000	0.000
Kidney	1.100	0.005 (< 1)	0.023 (2)	0.044 (4)	0.305 (28)	0.050 (5)	0.462 [b] (42)	0.016 (2)	0.094 (9)	0.000
Liver	1.209	0.000	0.016 (1)	0.042 (4)	0.265 (22)	0.023 (2)	0.609 [b] (50)	0.042 (4)	0.053 (4)	0.000

Note: Values < 0.0005 ppm are reported as 0.000 ppm; NA –not analysed due to the low total radioactive residue level (0.004 ppm); values in parentheses represent % of total radioactive residues;

[a] Metabolites identified in the study were numbered based on their HPLC retention times (shortest to longest)

[b] These values were obtained from the composite kidney and liver extracts, respectively, minus the amount of Fen-OH observed for kidney and liver. The reason for this method of calculation is that the Fen-OH peak was masked by the M-3 peak

In another study conducted with [benzyl (U)-^{14}C]-fenpyroximate (Jalali K., Gibson N.A., 1999b, report No R-4115), a lactating goat (Alpine/Nubian cross bred, approximately 3.5 years old, 41 kg) was orally administered by capsule for 3 consecutive days, at a dose rate of 10 ppm, corresponding to 0.3 mg/kg bodyweight/day. Milk samples were collected twice daily during the dosing period. The treated goat was sacrificed approximately 22 hours after the final dose. Total radioactive residue (TRR) levels in selected matrices were determined.

TRR levels in liver, kidney, muscle, fat were 1.253, 2.082, 0.024, 0.138 mg eq/kg respectively. After estimating for the total mass of blood, muscle and fat in a typical goat, total radioactivity recovered in the tissues accounted for 6.0% of the dose. In milk, TRR levels ranged from 0.008 to 0.031 ppm and total radioactivity recovered in the milk accounted for 0.1% of the administered dose.

Excretion of administered radioactivity in faeces was significant accounting for 44.7% of the dose. Urinary excretion was also found to be a significant route of elimination, accounting for 12.1% of the dose. At sacrifice 21.8% of the dose remained unexcreted in the gastrointestinal tract. Based on the amounts of radioactivity recovered in all the samples analysed, material balance for the study was 84.7% of the administered dose. Results are shown in Table 37.

The milk, kidney and liver samples were extracted and most of the radiocarbon in each sample was removed. Results are shown in Table 38. Extracts were analysed by HPLC, TLC and LC-MS where possible. Metabolite identification was also carried out in urine and faeces to confirm the assignments made in the tissue and milk samples. The excretion of fenpyroximate in the goat was rapid. A small trace was found in milk, muscle, fat and kidney, with faeces containing a large amount of parent. Seven metabolites were identified and/or characterised in milk and tissue samples. An eighth metabolite was the Z-isomer of parent fenpyroximate, referred to as M-1. Results are shown in Table 39.

Table 37 Residue levels found in milk and tissue samples of goat administered [benzyl-U-^{14}C]-fenpyroximate at a dose of 0.3 mg fenpyroximate/kg body weight/day for 3 consecutive days

Sample	Collection interval	Limit of quantification (ppm)	Residue level (ppm)	Percent of dose (%)	Cumulative percent of dose (%)
Liver	Sacrifice	0.0011	1.253	2.7	
Kidney	Sacrifice	0.0012	2.082	0.7	
Muscle	Sacrifice	0.0010	0.024	1.1	6.0
Fat	Sacrifice	0.0011	0.138	1.4	
Blood	Sacrifice	0.0011	0.034	0.1	
Milk	0-8 h	0.0004	0.008	0.01	
	8-24 h		0.013	0.02	
	24-32 h		0.024	0.01	
	32-48 h		0.025	0.03	0.1
	48-56 h		0.031	0.02	
	56h - sacrifice		0.022	0.03	
Urine	0-24 h		0.398	1.5	
	44-48 h		0.942	3.8	
	48h - sacrifice		1.312	5.8	12.1
Cage wash	Sacrifice		0.035	1.0	
Faeces	0-8 h		0.000	0.0	
	8-24 h		1.805	3.7	
	24-32 h		4.077	4.3	
	32-48 h		6.429	7.5	44.7
	48-56 h		9.047	5.2	
	56h - sacrifice		11.287	19.7	
Cage solids	Sacrifice		4.284	4.3	
Gastrointestinal tract			0.866	21.8	21.8
Total Recovery					84.7

Table 38 Extractability and Recovery of Radiocarbon in Goat Tissues and Milk

Sample	TRR(ppm)	Percent ^{14}C		Percent^{14}C
		Extracted [a]	Unextracted [b]	Recovered [c]
Milk 0-8 Hr	0.008	ND	ND	ND
Milk 8-24 Hr	0.013	136.3	9.7	146.0
Milk 24-32 Hr	0.024	104.6	9.1	113.7
Milk 32-48 Hr	0.025	87.2	6.4	93.6
Milk 48-56 Hr	0.031	84.9	5.5	90.4
Milk 56 Hr-Sac	0.022	85.9	6.1	92.0
Kidney	2.082	101.4	2.8*	104.2
Liver	1.235	106.7	4.4*	111.1
Muscle	0.027	131.7	44.9*	176.6
Fat	0.138	81.0	15.5*	96.5

ND Not done due to low TRR.

NA Not analysed.

[a] Refers to the amount extracted with hexane, ACN and ACN-water 1:1 (liver and kidney).

[b] Refers to the amount of unextractable radiocarbon as determined by combustion of the post extraction solids (PES).

[c] Sum of all extraction steps prior to combustion of the PES, plus the unextracted radiocarbon as determined by combustion of the PES.

* TRR in PES was in < 0.001mg eq/kg.

Milk and tissue samples were extracted and analysed by HPLC, TLC and LC-MS for content of radioactivity and characterisation of metabolites. A total of 7 metabolites were identified with an eighth metabolite, the Z-isomer of fenpyroximate (M-1) detected in the milk sample taken at sacrifice only. A summary of metabolite levels detected in tissues and milk is given in Table 39 and a proposed metabolic pathway incorporating results from both radiolabelled forms is shown in Figure 3.

Table 39 Metabolite levels (ppm) detected in tissues and milk of goat administered [benzyl-U-^{14}C]-fenpyroximate at a dose of 0.3 mg fenpyroximate/kg body weight/day for 3 consecutive days

Sample (times given in hours)	Distribution of metabolites (expressed as mg/kg, %TRR in parenthesis)									
	Total radio-active residue	Fenp	(1)[a] N-desmethyl M-3 acid	(2) M-5	(3) N-desmethyl M-3	(4) M-5 Glucur-onide	(5) M-3	(6) Fen-OH	(7) M-22	(8) M-1
0-8 milk	0.008	NA	NA	NA	NA	NA	NA	NA	NA	NA
8-24 milk	0.013	0.003 (23)	0.005 (39)	0.000	0.000	0.000	0.000	0.000	0.003 (23)	0.000
24-32 milk	0.024	0.006 (25)	0.000	0.000	0.000	0.000	0.000	0.002 (8)	0.006 (25)	0.000
32-48 milk	0.025	0.004 (16)	0.000	0.000	0.000	0.000	0.000	0.000	0.004 (16)	0.000
48-56 milk	0.031	0.008 (26)	0.000	0.000	0.000	0.000	0.000	0.000	0.007 (23)	0.000
56-sac milk	0.022	0.003 (14)	0.000	0.000	0.000	0.000	0.000	0.000	0.006 (27)	0.000
Muscle	0.027	0.002 (7)	0.000	0.000	0.000	0.000	0.000	0.009 (33)	0.020 (74)	0.000
Fat	0.138	0.049 (36)	0.000	0.000	0.000	0.003 (2)	0.000	0.019 (14)	0.024 (17)	0.000
Kidney	2.082	0.022 (1)	0.000	0.060 (3)	0.111 (5)	0.550 (26)	0.102 (5)	0.984 (47)	0.054 (3)	0.140 (5)
Liver	1.253	0.000	0.070 (6)	0.014 (1)	0.036 (3)	0.250 (20)	0.000	0.741 (59)	0.068 (5)	0.073 (6)

Note: Values < 0.0005 ppm are reported as 0.000 ppm; values in parentheses represent % of total radioactive residues;

NA – not analysed due to the low total radioactive residue level (0.004 ppm)

[a] Metabolites identified in the study were numbered based on their HPLC retention times (shortest to longest)

Following 3 consecutive daily doses of 10 ppm fenpyroximate labelled in either the pyrazole or benzyl ring (equivalent to 0.5 and 0.3 mg/kg bodyweight per day, respectively) residues in examined tissues were low, accounting for a total 3.3–6.0% of the administered dose. Of these tissues the excretory organs, liver and kidney contained the highest amount of radioactivity. Radioactivity in milk was low, accounting for 0.1–0.2% of the administered dose. The majority of radioactivity was eliminated *via* faeces accounting for 33.2% and 44.7% of the dose for the pyrazole and benzyl labels, respectively. Elimination *via* urine accounted for 32.1% and 12.1% dose for the pyrazole and benzyl labels, respectively. At sacrifice (22 hours after the last dose), 10.6 and 21.8% dose remained in the gastrointestinal tract for the pyrazole and benzyl labels, respectively. The biotransformation of fenpyroximate in lactating goats was essentially independent of radiolabelled form dosed. Unchanged fenpyroximate was present at low levels in all samples investigated, and its isomer M-1, was detected at even lower levels and only in a single milk sample (pyrazole label) and liver and kidney (benzyl label). The major routes of metabolism were *via* oxidation and subsequent cleavage of the isobutyl side chain followed by hydroxylation of the phenyl ring or N-demethylation on the pyrazole moiety.

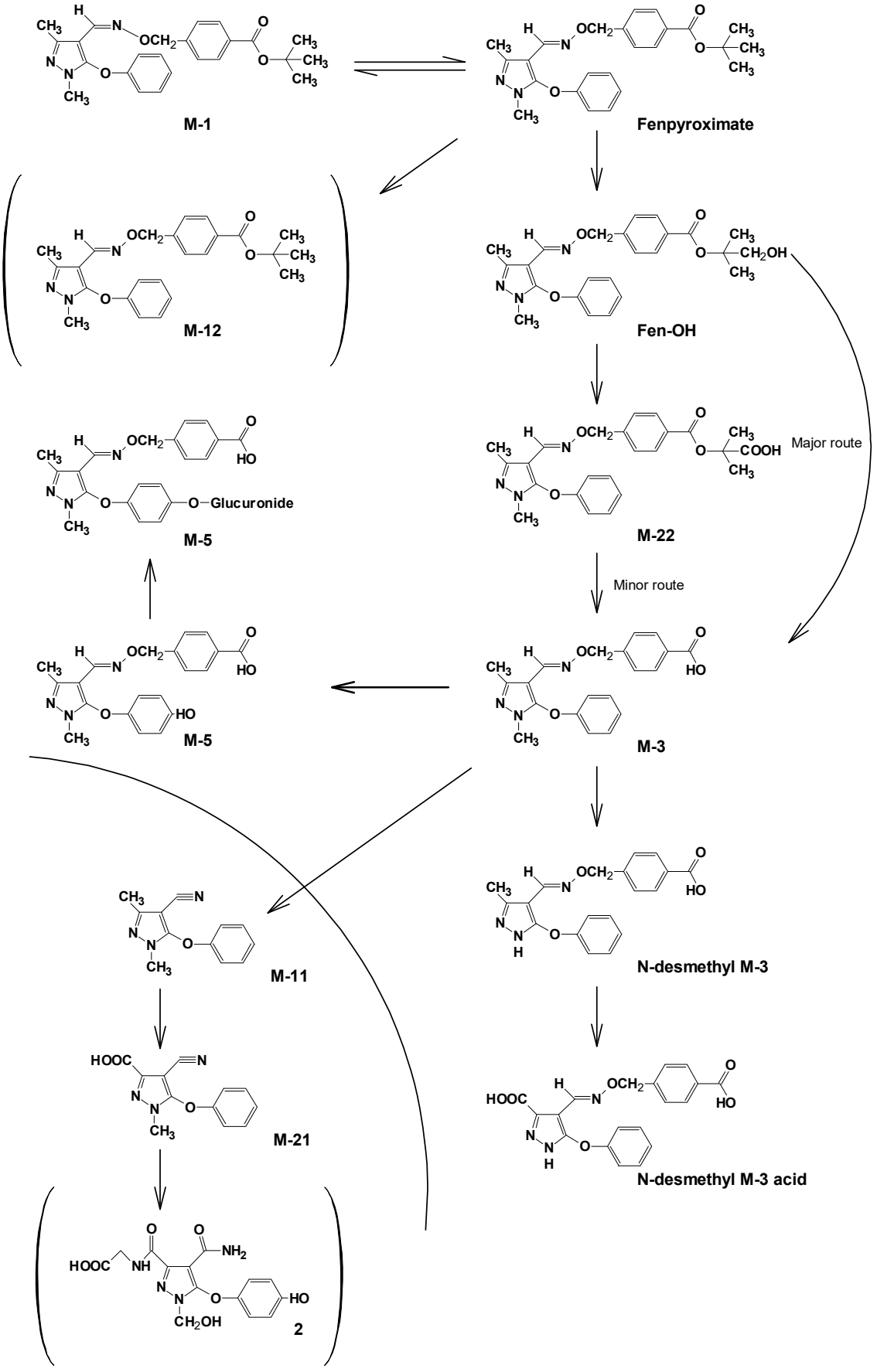


Figure 3 Proposed metabolic pathway for fenpyroximate in the lactating goat

FATE AND BEHAVIOUR IN THE ENVIRONMENT

Studies of Fenpyroximate on degradation under aerobic condition, field dissipation, hydrolysis and photolysis were received.

Fate and behaviour in soil

The degradation of fenpyroximate was investigated in two Japanese soils under aerobic conditions at 25 °C in the dark for up to 112 days (Funayama S., 1990, report No. E-4005). Soils were treated with [pyrazole-3-^{14}C] or [benzyl ring-^{14}C(U)]fenpyroximate at a rate of 1.30 and 2.12 mg/kg. The incubation unit was attached to traps containing ascarite and drierite and a polyurethane foam bung for the trapping of carbon dioxide and organic volatiles. Samples were removed for analysis at regular intervals, and were extracted with methanol:water (4:1, v/v), followed by two extractions with acetonitrile:1 M ammonium chloride (4:1, v/v), the resulting solution was used for radioassay and identification and analysis of metabolites by 2-dimensional thin-layer co-chromatography (2d-TLC) with authentic reference standards. The soil samples after extraction were combusted for quantification of unextracted residues. Bound residue fractionation was performed on samples from the final time point. The ascarite and foam bungs were extracted for quantification of carbon dioxide and organic volatiles.

Total recovery of applied radioactivity (AR) for the whole study ranged from 81.7% to 114.2%. The mean extractability of applied radioactivity from soil 0 days after treatment (DAT) for all samples ranged from 95.1% to 103.0% AR. Extractability decreased throughout the study with mean values of 13.6% to 31.1% AR recovered at 112 DAT across all samples. Bound residue and carbon dioxide increased steadily, reaching maximum mean values of 58.3% and 64.6% AR, respectively, at the study end. Fenpyroximate degraded in aerobic soil with DT_{50} values of 34.3–49.7 days in the Ehime soil and 26.3–35.6 days in the Kanagawa soil. The DT_{50} values for samples treated with the benzyl label should be treated with caution due to the limited number of sampling intervals. Fenpyroximate accounted for 92.2% to 101.8% AR across all samples at 0 DAT, and degraded steadily in both labels and soils to 6.6% to 20.1% AR at 112 DAT. Major metabolites of M-3 and M-11 were identified, reaching maximum mean values of 10.8% and 8.8% AR, respectively.

Table 40 Characteristics of soils used in aerobic soil study

Soil name	pH	OM (%)	Sand (%)	Silt (%)	Clay (%)	CEC (meq/ 100g)	Classification	MWHC (%)
Ehime	5.8	0.58	90.0	5.3	4.7	13.4	Sand	24.3
Kanagawa	5.5	2.52	61.2	28.8	10.0	45.8	Loam	61.8

OM=organic matter, CEC=cation exchange capacity

In the Ehime and Kanagawa soils, bound residues increased to maximum mean values of 46.0% and 58.3% AR, respectively. Carbon dioxide reached maximum mean values of 64.6% and 51.2% AR in Ehime and Kanagawa soils, respectively. Generally, pyrazole samples formed higher levels of bound residue than benzyl samples and the benzyl samples formed more carbon dioxide than the pyrazole samples. In the Ehime soil, fenpyroximate degraded steadily from a maximum mean value of 101.8% AR, to 10.7–20.1% AR. M-3 and M-11 were observed as major metabolites, reaching maximum mean values of 9.1% and 8.8% AR, respectively. In the Kanagawa soil, fenpyroximate degraded steadily from a maximum mean value of 101.1% AR, to 6.6–10.9% AR. M-3 and M-11 were observed as major metabolites, reaching maximum mean values of 10.8% and 8.2 AR, respectively.

Table 41 Mean percent recovery of AR after treatment of [pyrazole-^{14}C]fenpyroximate to Ehime soil

Sampling interval (DAT)	Extracts		Bound residue	Volatiles		Total
	Ethyl acetate layer	Aqueous layer		Organic	Carbon dioxide	
0	102.8	< 0.1	< 0.1	NA	NA	102.8
7	98.6	0.1	1.1	< 0.1	0.2	99.9
14	96.4	0.2	3.6	< 0.1	0.6	100.6
28	80.8	0.5	13.7	< 0.1	2.5	97.4
56	57.0	1.0	25.3	0.2	8.2	91.7
84	37.9	1.1	31.9	0.2	10.6	81.71
112	30.0	1.1	46.0	0.3	17.1	94.4
NA=Not applicable, [1] low recovery in both extracts (77.9 and 84.4%)						

Table 42 Mean percent recovery of AR after treatment of [benzyl-^{14}C]fenpyroximate to Ehime soil

Sampling interval (DAT)	Extracts		Bound residue	Volatiles		Total
	Ethyl acetate layer	Aqueous layer		Organic	Carbon dioxide	
0	97.8	< 0.1	< 0.1	NA	NA	97.8
7	88.7	< 0.1	2.2	< 0.1	2.4	93.3
28	93.2	0.2	8.0	< 0.1	11.2	112.5[a]
112	28.4	< 0.1	21.3	< 0.1	64.6	114.2[b]

[a] individual values were 111.5% and 113.5%, [2] individual values were 102.8% and 125.7%

Table 43 Mean percent recovery of AR after treatment of [pyrazole-^{14}C]fenpyroximate to Kanagawa soil

Sampling interval (DAT)	Extracts		Bound residue	Volatiles		Total
	Ethyl acetate layer	Aqueous layer		Organic	Carbon dioxide	
0	103.0	< 0.1	< 0.1	NA	NA	103.0
7	92.7	< 0.1	11.8	< 0.1	0.3	104.7
14	86.7	< 0.1	16.6	< 0.1	0.7	104.0
28	75.1	0.2	*	0.1	1.9	*
56	32.4[a]	0.4[a]	55.2[a]	0.1[a]	8.0[a]	96.1[a]
84	16.0	0.4	56.4	< 0.1	14.3	87.0[b]
112	13.4	0.2	58.3	0.1	16.8	88.8[c]

* no values available,

[a] values from one replicate only,

[b] individual values were 83.4% and 90.5%,

[c] individual values were 86.4% and 91.1%

Table 44 Mean percent recovery of AR after treatment of [benzyl-^{14}C]fenpyroximate to Kanagawa soil

Sampling interval (DAT)	Extracts		Bound residue	Volatiles		Total
	Ethyl acetate layer	Aqueous layer		Organic	Carbon dioxide	
0	95.1	< 0.1	3.5	NA	NA	98.6
7	93.2	< 0.1	10.0	< 0.1	1.7	104.9
28	60.7	0.8	30.7	< 0.1	6.6	98.7
112	13.5	0.6	41.5	< 0.1	51.2	106.7

The distribution of applied radioactivity as fenpyroximate and its metabolites in soil is provided in Tables 45-48.

Table 45 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [pyrazole-^{14}C]fenpyroximate to Ehime soil

Sampling interval (DAT)	Fenpyroximate	M-3	M-11	Minor metabolites*	Origin
0	101.8	ND	ND	0.3	< 0.1
7	88.7	2.6	0.6	6.5	0.2
14	77.4	5.4	4.9	8.4	0.3
28	51.5	9.1	8.8	9.8	1.6
56	33.5	5.1	4.9	12.1	1.4
84	17.5	3.9	5.0	9.4	1.9
112	10.7	2.4	6.8	8.2	1.9

ND=Not detected
*Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points. Includes metabolites M-1, M-8 and M-12 at maximum mean values of 3.0%, 3.5% and 0.6% AR, respectively

Table 46 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [benzyl-^{14}C]fenpyroximate to Ehime soil

Sampling interval (DAT)	Fenpyroximate	M-3	Minor metabolites*	Origin
0	95.5	ND	2.0	0.3
7	81.5	2.9	1.9	2.4
28	82.6	7.1	3.2	0.3
112	20.1	5.9	2.0	0.3

ND=Not detected
*Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points. Includes metabolites M-1 and M-12 at maximum mean values of 1.5% and 0.3% AR, respectively.

Table 47 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [pyrazole-^{14}C]fenpyroximate to Kanagawa soil

Sampling interval (DAT)	Fenpyroximate	M-3	M-11	Minor metabolites*	Origin
0	101.1	0.1	ND	1.8	< 0.1
7	81.0	0.7	6.3	4.6	0.1
14	64.5	10.8	7.1	3.9	0.2
28	51.4	5.7	8.2	9.5	0.3
56	16.9	1.9	5.2	8.0	0.5
84	7.9	0.5	3.5	3.8	0.3
112	6.6	0.1	3.1	3.3	0.3

ND=Not detected
*Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points. Includes metabolites M-1, M-8 and M-12 at maximum mean values of 1.6%, 3.4% and 1.5% AR, respectively.

Table 48 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [benzyl-^{14}C]fenpyroximate to Kanagawa soil

Sampling interval (DAT)	Fenpyroximate	M-3	Minor metabolites*	Origin
0	92.2	ND	2.8	0.1
7	89.8	1.1	2.1	0.1
28	54.5	2.6	3.4	0.2
112	10.9	1.0	1.4	0.2

ND=Not detected
*Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points. Includes metabolites M-1 and M-12, both at a maximum mean value of 1.3% AR.

The degradation of fenpyroximate was investigated in one soil under aerobic conditions at 10 °C in the dark for up to 120 days at 45% maximum water holding capacity (MWHC) (Lewis C.J.,

2002, report No. E-4041). Soils were treated with [pyrazole-3-^{14}C]-fenpyroximate at rate of 20 µg/100 g soil (equivalent to 0.15 kg ai/ha). The incubation unit was attached to a series of traps for the trapping of carbon dioxide and polar and non-polar volatiles. The soil samples at regular intervals were extracted three times with methanol, followed by one extraction with acetone, two extractions with methanol:water (1:1, v/v) and finally one extraction with methanol:0.1M HCl (1:1, v/v), and analysed by HPLC. Soil samples after extraction were air dried and combusted for quantification of unextracted residues.

The mean mass balance ranged from 99.2% to 103.5% AR. Extractability decreased throughout the study period from mean values of 102.8% AR at 0 DAT to 32.1% AR at 120 DAT. The maximum mean values of bound residue and carbon dioxide formed were 52.7% and 11.7% AR, respectively, both occurring at 120 DAT. Fenpyroximate degraded steadily from 101.9% at 0 DAT to 16.3% AR remaining at 120 DAT. Two major metabolites, M-3 and M-8, were observed, reaching maximum mean values of 9.2% and 7.0% AR, respectively. DT_{50} and DT_{90} values for fenpyroximate were calculated as 23 and 159 days, respectively.

Table 49 Soil characteristics of UK silt loam soil

Soil name	pH (CaCl2)	OC (%)	Sand (%)	Silt (%)	Clay (%)	Classification [a]	Water holding capacity (%)
SK108672	6.2	3.5	18	57	25	Silt loam	88.1 (at pF 0)

[a] USDA textural class, OC=organic carbon

Table 50 Mean percent recovery of AR after treatment of [^{14}C]fenpyroximate to SK108672 soil

Sampling interval (DAT)	Soil extracts		Bound residue	Carbon dioxide	Organic volatiles	Total
	Neutral	Acidic				
0	102.7	0.1	0.7	NA	NA	103.5
2	98.8	0.4	2.7	ND	ND	101.9
7	84.3	1.8	14.9	0.7	ND	101.7
14	81.6	1.2	15.9	1.1	ND	99.8
30	57.5	3.1	35.3	3.5	ND	99.4
59	47.2	1.9	43.1	7.0	ND	99.2
90	35.6	3.4	50.2	10.4	ND	99.6
120	31.8	3.0	52.7	11.7	ND	99.2

NA=not applicable, ND=not detected

Table 51 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [^{14}C]fenpyroximate to SK108672 soil

Sampling interval (DAT)	Fenpyroximate	M-3	M-8	M-11	Minor metabolites*
0	101.9	ND	ND	ND	ND
2	96.1	1.9	0.3	0.3	ND
7	70.1	9.2	2.4	1.5	0.8
14	68.5	9.1	2.1	0.7	0.7
30	41.6	7.6	3.8	0.7	3.5
59	31.5	5.1	5.8	0.9	3.4
90	20.4	3.8	6.2	1.1	3.9
120	16.3	2.6	7.0	1.0	4.8

ND=not detected, * Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points

The degradation of fenpyroximate was investigated in one US soil under aerobic conditions at 25 °C in the dark for up to 365 days (Shepler K., 2003 report No.: E-4039). Soil samples were treated with [pyrazole-^{14}C] or [benzyl-^{14}C]fenpyroximate at a rate of 0.44 mg/kg for both labels. Due to addition of too much water to the pyrazole treated samples after 2 months, a second application of

both [pyrazole-^{14}C] and [benzyl-^{14}C]fenpyroximate was made to additional units at a rate of 0.44 and 0.45 mg/kg, respectively.

Samples and their associated traps were removed for analysis at regular intervals. The soil samples were extracted with acetonitrile: 0.01 N aqueous HCl (3:1, v/v), and were analysed by HPLC. The soil samples after extraction were combusted for quantification of unextracted residues. The overall mean mass balance for ranged from 82.6% to 102.9% AR. The mean mass balance value of 82.6% came from two replicates of 71.4% and 93.8%. Extractability of fenpyroximate decreased throughout the study from a maximum mean value of 99.8% AR at 0 DAT, to mean values of 12.2% AR at the end of the study.

Bound residues reached a maximum mean value of 55.1% AR at 182 DAT, before decreasing for the remainder of the study. The amount of carbon dioxide generated increased steadily, reaching a maximum mean value of 57.8% AR at the study end. Fenpyroximate degraded from a mean value of 98.6% AR at 0 DAT to 1.9% AR at the end of the study. The half-lives for pyrazole and benzyl dosed samples were 33.2 and 35.5 days, respectively. Metabolite M-3 was identified as a major metabolite in both the pyrazole and benzyl samples, reaching maximum mean values of 15.1%. Metabolites M-8 and M-11 were identified as major metabolites in the pyrazole samples, reaching maximum mean values of 12.1% and 5.9% AR, respectively.

Table 52 Characteristics of soil used in aerobic soil study

Soil name	pH[a]	OM (%)	Sand (%)	Silt (%)	Clay (%)	CEC (meq/ 100g)	Classification	Moisture content at 1/3 bar (%)
Tulare County	8.6	0.8	64	30	6	10.9	Sandy loam	15.6

[a] measured in water, OM=organic matter, CEC=cation exchange capacity

Table 53 Percent recovery of AR after treatment of [pyrazole-^{14}C]fenpyroximate to Tulare County soil

Sampling interval (DAT)	Soil extracts	Bound residue	Carbon dioxide	Organic volatiles	Total
0[a]	101.8	2.5	NA	NA	104.3
0[1a]	98.1	2.5	NA	NA	100.6
0[b]	96.3	3.3	NA	NA	99.6
3[a]	99.2	4.1	0.0	0.0	103.3
3[a]	96.3	3.7	0.0	0.0	100.0
7[a]	95.5	4.5	0.0	0.0	100.0
7[a]	94.8	4.4	0.0	0.0	99.2
14[a]	94.2	11.5	0.1	0.0	105.8
14[a]	94.6	9.1	0.2	0.0	103.9
14[b]	93.6	6.8	0.1	0.0	100.5
14[b]	92.7	6.9	0.1	0.0	99.7
21[a]	87.1	14.6	0.6	0.0	102.3
21[a]	90.2	10.0	0.3	0.0	100.5
30[b]	84.4	14.9	0.6	0.0	99.9
30[b]	85.4	14.7	0.6	0.0	100.7
32[a]	81.1	21.1	1.3	0.0	103.5
32[a]	80.6	20.5	1.0	0.0	102.1
60[a]	56.5	38.2	3.6	0.0	98.3
60[a]	61.3	35.4	4.5	0.0	101.2
61[b]	72.7	26.2	1.7	0.0	100.6
61[b]	64.8	33.8	2.5	0.0	101.1
90[b]	49.8	44.6	5.9	0.0	100.3
90[b]	55.8	40.7	4.6	0.0	101.1
182[b]	27.1	55.2	15.7	0.0	98.0
182[b]	26.8	55.0	16.0	0.0	97.8

Sampling interval (DAT)	Soil extracts	Bound residue	Carbon dioxide	Organic volatiles	Total
273[b]	37.2	35.7	24.5	0.0	97.4
273[b]	42.6	34.2	19.2	0.0	96.0
365[b]	28.7	38.9	29.5	0.0	97.1
365[b]	28.9	37.7	29.0	0.0	95.2

NA=not applicable,

[a] first application,

[b] second application

Table 54 Percent recovery of AR after treatment of [benzyl-^{14}C]fenpyroximate to Tulare County soil

Sampling interval (DAT)	Soil extracts	Bound residue	Carbon dioxide	Organic volatiles	Total
0[a]	100.6	2.3	NA	NA	102.9
0[a]	100.5	2.5	NA	NA	103.0
0[b]	98.4	3.4	NA	NA	101.8
21[a]	82.5	13.6	4.6	0.0	100.7
21[a]	81.8	14.2	5.3	0.0	101.3
60[a]	39.0	31.8	17.9	0.0	88.7
60[a]	31.8	39.1	19.7	0.0	90.6
61[b]	64.3	23.8	12.7	0.1	100.9
61[b]	45.7	32.8	19.7	0.0	98.2
98[a]	19.2	39.1	38.7	0.0	97.0
98[a]	21.8	37.4	29.4	0.0	88.6
185[a]	11.6	37.9	44.3	0.0	93.8
185[a]	8.8	38.7	23.9	0.0	71.43
273[b]	17.8	26.0	52.8	0.0	96.6
273[b]	18.8	25.4	51.6	0.0	95.8
285[a]	6.1	32.5	58.2	0.0	96.8
285[a]	4.6	30.1	51.5	0.0	86.2
365[b]	13.3	25.2	56.6	0.0	95.1
365[b]	11.0	27.8	59.0	0.0	97.8

NA=not applicable,

[a] first application,

[b] second application,

Fenpyroximate degraded from a mean value of 98.6% AR at 0 DAT to 1.9% AR at the end of the study. Metabolite M-3 was identified as a major metabolite in both the pyrazole and benzyl samples, reaching maximum mean values of 15.1%. Metabolites M-8 and M-11 were identified as major metabolites in the pyrazole samples, reaching maximum mean values of 12.1% and 5.9% AR, respectively. The distribution of applied radioactivity as fenpyroximate and its metabolites in soil is provided in Tables 55 and 56.

Table 55 Distribution of AR as fenpyroximate and its metabolites after treatment of [pyrazole-^{14}C]fenpyroximate to Tulare County soil

Sampling interval (DAT)	Fenpyroximate	M-3	M-8	M-11	Minor metabolites*
0[a]	101.8	0.0	0.0	0.0	0.0
0[a]	98.1	0.0	0.0	0.0	0.0
0[b]	94.9	0.0	0.0	0.0	1.4
3[a]	97.4	1.8	0.0	0.0	0.0
3[a]	94.8	1.1	0.0	0.0	0.5
7[a]	95.5	0.0	0.0	0.0	0.0
7[a]	89.9	0.3	0.0	0.7	0.9
14[a]	83.6	5.7	0.8	1.4	2.8

Sampling interval (DAT)	Fenpyroximate	M-3	M-8	M-11	Minor metabolites*
14[a]	84.6	6.2	0.4	1.4	2.0
14[b]	87.0	3.7	0.0	0.0	2.9
14[b]	84.9	5.0	0.0	0.0	2.8
21[a]	73.2	7.7	2.1	1.6	2.6
21[a]	79.0	6.6	1.5	1.3	1.8
30[b]	73.2	6.2	2.2	1.6	1.3
30[b]	73.8	8.3	0.9	1.7	0.8
32[a]	61.0	11.0	3.8	2.7	1.6
32[a]	59.0	12.3	4.0	3.1	2.1
60[a]	23.1	14.7	10.5	5.1	0.5
60[a]	28.2	15.4	8.5	4.9	2.4
61[b]	49.8	10.1	5.5	3.7	1.2
61[b]	35.6	10.4	8.2	3.8	2.9
90[b]	13.2	13.9	11.7	5.8	2.1
90[b]	18.9	15.5	12.4	5.9	1.2
182[b]	2.5	2.2	9.3	3.1	1.5
182[b]	2.3	3.3	8.0	3.6	0.5
273[b]	2.7	4.0	4.6	4.0	7.3
273[b]	2.6	5.7	6.5	4.6	8.9
365[b]	2.1	3.9	2.0	1.7	7.9
365[b]	1.6	4.0	2.1	1.6	7.8

Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points.
[a] first application,
[b] second application

Table 56 Distribution of AR as fenpyroximate and its metabolites after treatment of [benzyl-^{14}C]fenpyroximate to Tulare County soil

Sampling interval (DAT)	Fenpyroximate	M-3	Minor metabolites*
0[a]	98.9	0.4	1.3
0[a]	100.0	0.0	0.5
0[b]	96.8	0.0	1.6
21[a]	70.7	9.9	1.9
21[a]	69.9	8.6	3.3
60[a]	30.3	7.7	0.5
60[a]	23.9	6.6	0.7
61[b]	48.4	14.8	0.3
61[b]	30.9	12.6	1.2
98[a]	3.8	11.5	3.6
98[a]	9.7	8.8	2.2
185[a]	4.3	5.2	1.4
185[a]	2.8	3.8	1.5
273[b]	2.4	7.8	1.2
273[b]	2.0	9.2	2.7
285[a]	1.8	2.8	0.2
285[a]	1.6	2.4	0.0
365[b]	2.9	5.9	2.6
365[b]	1.5	5.6	2.3

Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points.
[a] first application,
[b] second application

The degradation of fenpyroximate was investigated in three European soils and one US soil under aerobic conditions at 20 °C in the dark for up to 219 days (Roohi A., 2016, report No. E-4052).

Soils were treated with [^{14}C-benzyl] or [^{14}C-pyrazole]-fenpyroximate at a rate of 61.0 and 62.0 µg/100 g soil, respectively (equivalent to a nominal field rate of 0.224 kg as/ha). The vessels were attached to a series of traps and incubated at 20 ± 2 °C in the dark and moistened, carbon dioxide free air was passed over the samples.

Duplicate soil samples and their associated traps from each label were removed for analysis at regular intervals. The soil samples were extracted three times with acetonitrile:0.01 M HCl (3:1, v/v), followed by two extractions with acetonitrile:1 M ammonium chloride (4:1, v/v), and analysed by HPLC. Selected samples were also analysed by LC-MS for confirmation of fenpyroximate and metabolites. Once extracted, soil samples were combusted for quantification of unextracted residues. Soil samples from 70 DAT were further characterised by bound residue fractionation. The trapping of carbon dioxide in the KOH solutions was confirmed via barium chloride precipitation.

The overall mean recovery of applied radioactivity ranged from 75.59% to 101.86%. Where low mass balance occurred the recovery of volatiles was lower than at preceding time points, suggesting loss of applied radioactivity was due to carbon dioxide. Extractability of applied radioactivity varied significantly across the four soils with 9.78% to 91.93% AR in the extracts at the end of the study. Unextracted residue and total volatiles increased to maximum mean values of 53.61% and 49.23% AR, respectively.

Fenpyroximate degraded at varying rates across all soils. In the Speyer 2.2 soil, fenpyroximate degraded slowly with 66.92% to 69.63% AR remaining at 120 DAT. Degradation of fenpyroximate was quicker in the other soils with 6.95% to 30.31% AR remaining at 120 DAT and 5.50% to 29.17% AR remaining at 219 DAT. The DT_{50} and DT_{90} values for fenpyroximate ranged from 23.2 to 254 days and 134 to 844 days, respectively. Metabolite M-3 was identified as a major metabolite in both the pyrazole and benzyl samples, reaching a maximum mean value of 28.72% AR. Metabolites M-8 and M-11 were identified as major metabolites in the pyrazole samples, reaching maximum mean values of 15.90% and 9.55% AR, respectively.

Table 57 Characteristics of soils used in aerobic soil study

Soil name	pH [a]	OC (%)	Sand [b] (%)	Silt [b] (%)	Clay [b] (%)	CEC (meq/ 100g)	Classification [b]	MWHC (%)
Speyer 2.2	5.7	1.6	82	10	8	7.3	Loamy sand	55.9
Speyer 5M	7.3	1.1	56	28	16	10.3	Sandy loam	50.2
Speyer 6S	7.0	1.7	32	22	46	22.5	Clay	47.6
Iowa	6.0	1.7	14	71	15	12.1	Silt loam	64.4

[a] measured in $CaCl_2$,

[b] USDA textural classification,

OC=organic carbon,

CEC=cation exchange capacity

Table 58 Mean percent recovery of AR after treatment of [benzyl-^{14}C]fenpyroximate to Speyer 2.2 soil

Sampling interval (DAT)	Soil extracts	Unextracted residue	Total volatiles	Total
0	96.23[a]	1.87	NA	98.09
7	96.85	0.74	0.37	97.96
14	95.18	1.34	1.42	97.94
29	94.09	2.70	3.97	100.76
70	88.96	4.53	5.74	99.23
120	83.36	6.51	9.57	99.44

[a] Value in report is 98.50% which appears to be an error. Value calculated from the two replicates (96.69 and 95.76%),

NA=not applicable

Table 59 Mean percent recovery of AR after treatment of [benzyl-^{14}C]fenpyroximate to Speyer 5M soil

Sampling interval (DAT)	Soil extracts	Unextracted residue	Total volatiles	Total
0	98.24	3.32	NA	101.56
7	90.20	6.63	2.06	98.89
14	85.38	7.36	5.69	98.43
29	59.27	19.64	18.90	97.82
70	28.42	26.46	36.70	91.58
120	14.30	29.66	49.23	93.19
170	17.00	32.73	40.60	90.33
219	9.78	30.83	34.98	75.59*

NA=not applicable,

* low mass balance may be due to loss of volatiles

Table 60 Mean percent recovery of AR after treatment of [benzyl-^{14}C]fenpyroximate to Speyer 6S soil

Sampling interval (DAT)	Soil extracts	Unextracted residue	Total volatiles	Total
0	92.49	8.36	NA	100.85
7	82.43	11.59	0.72	94.75
14	74.98	19.48	1.79	96.25
29	70.43	18.87	6.53	95.83
70	59.63	17.14	16.65	93.42
120	44.58	22.10	28.32	95.00
170	29.04	25.37	37.03	91.44
219	23.07	28.86	40.79	92.72

NA=not applicable

Table 61 Mean percent recovery of AR after treatment of [benzyl-^{14}C]fenpyroximate to Iowa soil

Sampling interval (DAT)	Soil extracts	Unextracted residue	Total volatiles	Total
0	96.82	3.41	NA	100.23
7	87.13	6.60	2.68	96.41
14	82.68	8.94	4.84	96.46
29	60.61	22.48	14.85	97.94
70	55.65	21.41	20.15	97.21
120	48.25	18.91	29.60	96.76
170	33.77	27.38	27.97	89.12
219	48.65	28.63	23.53	100.81

NA=not applicable

Table 62 Mean percent recovery of AR after treatment of [pyrazole-^{14}C]fenpyroximate to Speyer 2.2 soil

Sampling interval (DAT)	Soil extracts	Unextracted residue	Total volatiles	Total
0	98.75	1.85	NA	100.59
7	99.20	0.51	0.04	99.76
14	96.90	1.16	0.09	98.15
29	99.22	1.65	0.23	101.10
70	94.29	4.77	1.16	100.22
120	91.93	6.71	2.07	100.72

NA=not applicable

Table 63 Mean percent recovery of AR after treatment of [pyrazole-^{14}C]fenpyroximate to Speyer 5M soil

Sampling interval (DAT)	Soil extracts	Unextracted residue	Total volatiles	Total
0	98.77	2.06	NA	100.82
7	96.25	5.46	0.15	101.86
14	92.71	6.67	0.40	99.79
29	83.58	14.73	1.33	99.65
70	52.35	34.80	8.81	95.95
120	29.96	46.65	18.69	95.30
170	17.64	53.61	23.11	94.36
219	15.57	51.16	28.89	95.62

NA=not applicable

Table 64 Mean percent recovery of AR after treatment of [pyrazole-^{14}C]fenpyroximate to Speyer 6S soil

Sampling interval (DAT)	Soil extracts	Unextracted residue	Total volatiles	Total
0	94.00	6.88	NA	100.88
7	91.93	4.74	0.04	96.70
14	89.08	6.09	0.17	95.34
29	81.22	15.40	0.79	97.41
70	68.36	24.12	4.67	97.15
120	48.36	35.45	13.75	97.55
170	36.37	42.21	12.01	90.59
219	35.77	45.52	17.14	98.43

NA=not applicable

Table 65 Mean percent recovery of AR after treatment of [pyrazole-^{14}C]fenpyroximate to Iowa soil

Sampling interval (DAT)	Soil extracts	Unextracted residue	Total volatiles	Total
0	94.93	5.28	NA	100.21
7	93.94	5.42	0.38	99.74
14	93.27	6.07	1.04	100.38
29	83.53	12.82	2.29	98.64
70	71.31	18.62	8.22	98.15
120	73.19	16.52	11.04	100.75
170	55.85	28.01	14.10	97.96
219	61.03	29.15	13.65	103.84

NA=not applicable

The distribution of applied radioactivity as fenpyroximate and its metabolites in soil is provided in Tables 66 to 73.

Table 66 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [benzyl-^{14}C]fenpyroximate to Speyer 2.2 soil

Sampling interval (DAT)	Fenpyroximate	M-3	Minor metabolites*
0	92.78	0.33	2.99
7	92.23	1.23	3.12
14	88.94	2.33	2.67
29	86.50	4.97	2.52
70	78.55	7.58	2.54
120	66.92	11.68	4.76

* Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points. Includes metabolite M-1 at a maximum mean value of 3.12% AR.

Table 67 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [benzyl-^{14}C]fenpyroximate to Speyer 5M soil

Sampling interval (DAT)	Fenpyroximate	M-3	Minor metabolites*
0	95.63	0.00	2.61
7	74.76	13.19	2.02
14	55.18	28.72	1.45
29	35.03	22.39	1.3
70	16.10	11.70	0.63
120	6.95	5.78	0.55
170	7.60	7.85	0.36
219	5.50	3.30	0.00

* Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points. Includes metabolite M-1 at a maximum mean value of 2.61% AR.

Table 68 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [benzyl-^{14}C]fenpyroximate to Speyer 6S soil

Sampling interval (DAT)	Fenpyroximate	M-3	Minor metabolites*
0	89.24	0.32	2.84
7	75.74	3.79	2.46
14	67.83	5.67	1.92
29	57.31	11.30	1.12
70	38.68	18.80	1.40
120	26.00	14.96	0.20
170	16.56	9.63	0.71
219	12.70	8.29	0.00

* Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points. Includes metabolite M-1 at a maximum mean value of 2.84% AR.

Table 69 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [benzyl-^{14}C]fenpyroximate to Iowa soil

Sampling interval (DAT)	Fenpyroximate	M-3	Minor metabolites*
0	94.19	0.11	2.54
7	77.76	8.06	2.58
14	68.04	13.46	1.45
29	46.84	12.43	1.05
70	37.11	16.54	1.55
120	27.18	19.58	1.50
170	18.43	12.03	3.32
219	29.17	15.22	4.25

* Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points. Includes metabolite M-1 at a maximum mean value of 2.54% AR.

Table 70 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [pyrazole-^{14}C]fenpyroximate to Speyer 2.2 soil

Sampling interval (DAT)	Fenpyroximate	M-3	M-8	M-11	Minor metabolites*
0	95.70	0.00	0.00	0.00	3.04
7	92.70	1.76	0.39	0.78	2.69
14	91.20	2.43	0.00	0.68	2.33

Sampling interval (DAT)	Fenpyroximate	M-3	M-8	M-11	Minor metabolites*
29	90.49	2.94	1.34	1.48	2.79
70	77.55	8.11	2.35	3.23	2.79
120	69.63	10.00	3.87	3.75	4.82

* Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points. Includes metabolite M-1 at a maximum mean value of 3.04% AR.

Table 71 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [pyrazole-^{14}C]fenpyroximate to Speyer 5M soil

Sampling interval (DAT)	Fenpyroximate	M-3	M-8	M-11	Minor metabolites*
0	94.78	0.00	0.00	0.00	3.07
7	77.38	13.45	0.76	1.39	2.44
14	68.05	16.67	2.35	2.37	1.91
29	51.62	10.11	11.03	5.44	4.92
70	19.41	2.71	15.26	8.32	6.07
120	10.13	1.36	7.73	5.78	4.73
170	7.73	1.05	1.10	4.95	2.81
219	7.06	0.32	1.20	3.32	2.25

* Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points. Includes metabolite M-1 at a maximum mean value of 3.07% AR.

Table 72 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [pyrazole-^{14}C]fenpyroximate to Speyer 6S soil

Sampling interval (DAT)	Fenpyroximate	M-3	M-8	M-11	Minor metabolites*
0	88.62	0.76	0.00	0.00	4.56
7	72.87	4.53	0.09	0.50	2.07
14	77.77	8.13	0.02	0.38	1.94
29	65.43	9.99	1.49	2.04	2.25
70	38.52	15.03	4.56	5.53	4.10
120	21.19	6.42	8.50	5.42	5.86
170	15.66	4.48	3.61	4.87	7.74
219	18.29	5.61	3.76	4.55	2.51

* Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points. Includes metabolite M-1 at a maximum mean value of 2.45% AR.

Table 73 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [pyrazole-^{14}C]fenpyroximate to Iowa soil

Sampling interval (DAT)	Fenpyroximate	M-3	M-8	M-11	Minor metabolites*
0	91.29	0.20	0.00	0.00	3.44
7	80.41	8.74	1.11	2.04	3.60
14	72.75	12.86	1.96	2.35	3.00
29	54.65	15.30	5.16	4.37	3.83
70	31.64	9.54	13.90	8.73	6.82
120	30.31	9.21	15.90	9.53	7.80
170	23.76	7.42	4.83	9.55	10.29
219	20.68	9.72	13.90	9.09	7.63

* Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points. Includes metabolite M-1 at a maximum mean value of 3.08% AR.

Soil photolysis

The photodegradation of fenpyroximate was investigated in one German soil under continuous irradiation with simulated natural sunlight and aerobic conditions at 20 °C (Doble M.L., 2016, report No.: E-4053). Soil samples in photolysis dishes were treated with [pyrazole-^{14}C] or [benzyl-^{14}C]fenpyroximate at a nominal rate of 13.8 µg/vessel (equivalent to 224 g ai/ha). The treated samples were incubated at 20 °C under continuous irradiation or in the dark. The soil samples at regular intervals were extracted twice with acetonitirile:0.01 M HCl (3:1, v/v) (extracts 1 and 2) and a further two times with acetonitrile:0.1M NH_4Cl (8:2, v/v), and analysed by LSC and HPLC. The soil samples after extraction were air-dried and combusted for quantification of unextracted residues. Degradation kinetics were investigated following SFO, FOMC and DFOP kinetics in the CAKE 2.0 software.

The mean total recovery of applied radioactivity ranged from 89.31% to 102.136% AR. Extractability remained high throughout the study, with mean values of >89% AR, in all samples with one exception (85.9% AR) where there was a loss on processing of one replicate, resulting in individual extraction recoveries of 97.96% and 72.84% AR. Unextracted residues and carbon dioxide accounted for maximum mean values of 5.34% and 3.55% AR, respectively.

Fenpyroximate degraded quickly under irradiated conditions, from mean values of 96.32–99.84% AR at 0 DAT, to 59.49–56.19% AR by the end of the study. Major metabolites M-1, M-12, M-12 isomer and MTBT were observed in irradiated samples, reaching maximum mean values of 17.22%, 5.46%, 5.10% and 8.26% AR, respectively.

In dark conditions fenpyroximate degraded more slowly, with 72.99–74.99% AR remaining at the end of the study. The only major metabolite observed in the dark samples was M-3, reaching a maximum mean value of 14.21% AR.

Table 74 Characteristics of soil used in aerobic soil study

Soil name	pH [a]	OC (%)	Sand [b] (%)	Silt [b] (%)	Clay [b] (%)	Classification [b]	MWHC (%)
Speyer 5M	7.3	1.1	55	29	16	Sandy loam	41.87

[a] measured in 0.01 M $CaCl_2$,

[b] USDA textural classification,

OC=organic carbon

Table 75 Mean percent recovery of AR after treatment of [benzyl-^{14}C]fenpyroximate to Speyer 5M soil (irradiated)

Incubation time (days)	Incubation time (hours[a])	Incubation time (days[b])	Soil extracts	Volatile traps	Unextracted residue	Total
0[c]	0	0	98.59	NA	0.00	98.59
2	45	2.4	96.01	0.00	0.21	96.22
6	138	7.4	97.65	0.04	0.46	98.15
13	303	16.2	92.51	1.09	1.30	94.90
20	468	25.1	94.01	1.38	1.76	97.14
24	568	30.4	95.34	0.82	1.58	97.74

[a] actual incubation time,

[b] days equivalent to natural sunlight conditions at 30–50 °N,

[c] zero day samples used for both irradiated and dark control,

NA=not applicable

Table 76 Mean percent recovery of AR after treatment of [benzyl-^{14}C]fenpyroximate to Speyer 5M soil (dark control)

Incubation time (days)	Incubation time (hours [a])	Soil extracts	Volatile traps	Unextracted residue	Total
0[b]	0	98.59	NA	0.00	98.59
2	45	91.02	0.00	0.07	91.09
6	138	97.00	0.23	0.41	97.64
13	303	93.26	1.22	1.29	95.77
20	468	90.19	2.51	2.63	95.32
24	568	89.25	3.55	2.83	95.63

[a] actual incubation time,

[b] zero day samples used for both irradiated and dark control,

NA=not applicable

Table 77 Mean percent recovery of AR after treatment of [pyrazole-^{14}C]fenpyroximate to Speyer 5M soil (irradiated)

Incubation time (days)	Incubation time (hours a)	Incubation time (days b)	Soil extracts	Volatile traps	Unextracted residue	Total
0c	0	0	102.16	NA	0.00	102.16
2	44	1.9	98.08	0.19	0.22	98.49
7	164	7.0	97.67	0.61	0.60	98.88
14	329	14.0	99.94	0.80	0.98	101.72
21	495	21.1	85.40	1.82	2.10	89.31*
30	710	30.2	95.40	1.27	5.34	102.00

[a] actual incubation time,

[b] days equivalent to natural sunlight conditions at 30-50 °N,

[c] zero day samples used for both irradiated and dark control,

* low mass balance in one replicate (76.48) due to partial loss of sample,

NA=not applicable

Table 78 Mean percent recovery of AR after treatment of [pyrazole-^{14}C]fenpyroximate to Speyer 5M soil (dark control)

Incubation time (days)	Incubation time (hours[a])	Soil extracts	Volatile traps	Unextracted residue	Total
0[b]	0	102.16	NA	0.00	102.16
2	44	99.18	0.00	0.17	99.34
7	164	101.88	0.02	0.70	102.59
14	329	99.48	0.06	0.93	100.48
21	495	96.27	0.12	2.60	98.99
30	710	97.98	0.62	5.08	103.67

[a] actual incubation time,

[b] zero day samples used for both irradiated and dark control,

NA=not applicable

The mean distribution of applied radioactivity as fenpyroximate and its metabolites in soil is provided in Tables 79-82.

Table 79 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [benzyl-^{14}C]fenpyroximate to Speyer 5M soil (irradiated)

DAT	Fenpyroximate	M-1	M-3	M-12	M-12 isomer	MTBT	Minor metabolites*
0	96.32	0.74	-	-	-	0.16	0.75
2	85.96	5.33	-	0.78	0.53	2.04	0.68
6	79.88	10.12	0.23	1.23	1.25	3.13	1.05
13	61.28	16.76	1.71	0.91	1.17	7.00	2.18
20	63.83	17.44	1.45	0.55	0.90	5.89	2.54
24	59.49	17.22	1.70	1.79	2.14	8.26	2.48

* Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points.

Table 80 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [benzyl-^{14}C]fenpyroximate to Speyer 5M soil (dark control)

DAT	Fenpyroximate	M-1	M-3	MTBT	Minor metabolites*
0	96.32	0.74	0.00	0.16	0.88
2	88.26	0.86	-	1.11	0.23
6	91.49	1.05	1.90	1.82	0.02
13	83.64	0.83	5.76	1.34	0.41
20	76.98	0.81	8.94	0.90	0.64
24	74.99	0.69	10.34	0.81	0.29

* Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points.

Table 81 Mean distribution of AR as fenpyroximate and its metabolites after treatment of [pyrazole-^{14}C]fenpyroximate to Speyer 5M soil (irradiated)

DAT	Fenpyroximate	M-1	M-3	M-12	M-12 isomer	Minor metabolites*
0	99.84	0.79	-	-	-	1.00
2	89.90	4.45	-	0.47	0.32	2.17
7	82.68	8.38	1.01	0.47	0.60	3.67
14	76.05	12.09	1.01	1.68	1.19	6.56
21	57.96	11.93	2.51	2.70	2.40	6.09
30	56.19	14.00	1.58	5.46	5.10	11.34

* Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points. . Includes metabolites M-8 and M-11 at a maximum mean values of 2.98% and 2.51% AR, respectively.

Table 82: Mean distribution of AR as fenpyroximate and its metabolites after treatment of [pyrazole-^{14}C]fenpyroximate to Speyer 5M soil (dark control)

DAT	Fenpyroximate	M-1	M-3	M-12	M-12 isomer	Minor metabolites*
0	99.84	0.79	-	-	-	1.00
2	95.30	1.07	0.04	0.26	0.14	1.62
7	95.35	0.74	2.67	0.17	0.03	1.91
14	88.10	0.64	5.60	-	0.45	3.39
21	80.15	0.84	9.90	0.53	-	3.64
30	72.79	0.93	14.21	0.22	-	7.77

* Sum of minor metabolites, no individual metabolite seen at ≥5% AR at consecutive time points. Includes metabolite M-8 at a maximum mean value of 3.93% AR.

A summary of the DT_{50} and DT_{90} values is provided in table 83.

Table 83 Summary of kinetic values after treatment of [benzyl-^{14}C] or [pyrazole-^{14}C]fenpyroximate to Speyer 5M soil

Sample type	Label	Kinetic model	χ2 err	Confidence measure	Visual assessment	DT_{50} (hours)	DT_{90} (hours)
Dark control	Benzyl	SFO	2.08	7.20×10^{-5}	Good	1690	5630
	Pyrazole	SFO	1.44	7.75×10^{-8}	Good	1620	5380
Irradiated	Benzyl	SFO	5.0	7.86×10^{-6}	Good	797	2650
	Pyrazole	SFO	3.6	4.05×10^{-6}	Good	816	2710

The DT_{50} and DT_{90} values were calculated in terms of actual hours under the suntest. For the irradiated samples, these values have been converted into equivalent days of natural sunlight at 30–50 °N, giving DT_{50} values of 42.7 and 34.7 days for the benzyl and pyrazole samples, respectively. The DT_{90} equivalent day values were 142 and 115 days for the benzyl and pyrazole samples, respectively. Fenpyroximate degraded quickly under irradiated conditions to major metabolites M-1, M-12, M-12 isomer and MTBT. In the dark, fenpyroximate degraded more slowly and M-3 was the only major metabolite observed.

The rate of degradation of fenpyroximate was investigated in three field soils under aerobic conditions at 20 °C in the dark for up to 100 days (Römbke, J., Möllerfeld J., 1992, report No.: E-4008). Soils were treated with [pyrazole-^{14}C]-fenpyroximate at a rate of 20 µg/100 g soil (equivalent to 150 g ai/ha). Samples were incubated under aerobic conditions at 20 °C in the dark. Samples were not connected to volatile traps. The soils samples at regular intervals were extracted once with methanol:water (1:1, v/v), once with methanol and once with acetone, and analysed by two-dimensional TLC. The soil samples after extraction were combusted for quantification of unextracted residues. The DT_{50} and DT_{90} values for each soil were calculated from these data, using a PC programme developed by V. Laska (Bayer AG).

The amount of applied radioactivity recovered in the soil extracts decreased from 84.0–89.7% at 0 DAT to 11.9–29.4% at 100 DAT. Bound residues increased from 9.1–16.2% AR at 0 DAT, to 13.7–34.5% AR at 100 DAT. There was no provision for the trapping of volatiles throughout the study, however significant amounts of carbon dioxide were generated in a previous study (up to 17.1% AR, Funayama (1990)), therefore it was concluded that the low mass balance is largely due to generation of carbon dioxide. Also the vapour pressure for fenpyroximate is low (0.75×10-5, taken from DAR 2005), indicating volatility of fenpyroximate from the soil surface is unlikely.

Extractability at 0 DAT was good (84.0–89.7%), indicating that the extraction method is reasonably robust for the extraction of fenpyroximate. Extractability throughout the study appears to follow a similar pattern as previously seen in Funayama (1990), therefore this study has been deemed acceptable for the calculation of the rate of degradation of fenpyroximate. Fenpyroximate degraded steadily, with 8.8–22.9% AR remaining at 100 DAT. M-3 was identified as a major metabolite, reaching a maximum of 8.9% AR in one soil The DT_{50} values were calculated as 16.9, 10.1 and 21.3 days for field soil I, II and III, respectively. The DT_{90} values were calculated as 186.4, 111.9 and >100 days for field soil I, II and III, respectively. These values have been superseded by a new full FOCUS kinetics assessment (Graham 2016, Report no. E-4058).

Table 84 Characteristics of soils used in aerobic soil study

Soil name	pH (KCl)	OC (%)	Sand (%)	Silt (%)	Clay (%)	Classification	MWHC (%)
Field Soil I	6.9	3.97	20.2	49.5	30.3	Clay loam	55.9
Field soil II	6.3	3.97	11.2	65.7	23.1	Silt loam	54.1
Field soil III	5.9	2.83	30.6	49.7	19.7	Sandy loam	52.8

OC=organic carbon, MWHC=maximum water holding capacity

Table 85 Mean distribution of AR after treatment of pyrazole-^{14}C-fenpyroximate to Field Soil I

Sampling interval (DAT)	Extract	Residue	Total
0	84.3	14.4	98.7
2	62.8	9.8	72.6
4	63.7	12.1	75.7
8	62.4	15.8	78.1
16	52.9	16.2	69.1
32	38.4	29.9	68.3
64	20.7	24.3	45.0
100	24.3	28.5	52.8

Table 86 Mean distribution of AR after treatment of pyrazole-^{14}C-fenpyroximate to Field Soil II

Sampling interval (DAT)	Extract	Residue	Total
0	88.5	9.6	98.3
2	61.3	8.3	69.6
4	59.9	6.9	66.8
8	62.7	10.8	73.4
16	54.6	12.6	67.1
32	30.8	21.0	51.7
64	16.6	22.0	38.6
100	12.3	23.5	35.8

Table 87 Mean distribution of AR after treatment of pyrazole-^{14}C-fenpyroximate to Field Soil III

Sampling interval (DAT)	Extract	Residue	Total
0	88.2	10.5	98.7
2	59.7	6.7	66.4
4	60.7	7.1	67.8
8	61.6	9.4	71.0
16	62.4	14.6	77.0
32	43.4	19.5	62.9
64	31.9	23.0	54.9
100	27.5	28.8	56.3

Fenpyroximate degraded steadily, with mean values of 9.9%–19.6% AR remaining at 100 DAT. M-3 was identified as a major metabolite, reaching a maximum mean value of 8.0% AR in Field soil I at 32 DAT. M-3 was also seen in Field soil II and III, however it did not reach levels >5% AR. Metabolites M-1, M-6, M-8 and M-11 were seen in each soil, but did not reach levels exceeding 5% AR.

Table 88 Mean distribution of AR as fenpyroximate and its metabolites after treatment of pyrazole-^{14}C-fenpyroximate to Field Soil I

Sampling interval (DAT)	Fenpyroximate	M-1	M-3	M-6	M-8	M-11
0	83.4	0.9	0.0	0.0	0.0	0.0
2	59.0	1.4	2.2	0.0	0.0	0.3
4	58.2	1.6	3.3	0.1	0.2	0.4
8	27.9	1.4	4.5	0.1	0.2	0.4
16	41.4	1.5	7.9	0.3	0.2	0.3
32	25.2	1.1	8.0	0.6	0.1	1.1
64	16.7	0.7	1.8	0.2	0.0	0.5
100	18.5	0.4	3.1	0.3	0.2	0.7

Table 89 Mean distribution of AR as fenpyroximate and its metabolites after treatment of pyrazole-^{14}C-fenpyroximate to Field Soil II

Sampling interval (DAT)	Fenpyroximate	M-1	M-3	M-6	M-8	M-11
0	86.9	1.7	0.0	0.0	0.0	0.0
2	58.9	1.0	0.7	0.0	0.1	0.6
4	56.2	1.9	1.3	0.0	0.1	0.3
8	57.1	1.5	2.8	0.2	0.3	0.6
16	46.4	0.8	5.3	0.3	0.4	0.8
32	23.9	1.0	3.0	0.4	0.2	1.6
64	*	*	*	*	*	*
100	9.9	0.4	0.5	0.0	0.1	0.7

* samples erroneously destroyed and could not be measured

Table 90 Mean distribution of AR as fenpyroximate and its metabolites after treatment of pyrazole-^{14}C-fenpyroximate to Field Soil III

Sampling interval (DAT)	Fenpyroximate	M-1	M-3	M-6	M-8	M-11
0	86.8	1.5	0.0	0.0	0.0	0.0
2	58.0	1.2	0.5	0.0	0.0	0.0
4	58.0	1.6	0.7	0.0	0.0	0.4
8	59.4	1.5	0.5	0.0	0.0	0.2
16	57.6	1.4	2.1	0.0	0.2	0.9
32	34.8	1.3	4.9	0.4	0.2	1.3
64	21.9[a]	1.0[a]	4.4[a]	0.2[a]	0.2[a]	2.4[a]
100	19.6	0.4	3.3	0.2	0.1	2.3

[a] one sample erroneously destroyed and could not be measured, values from one replicate

The rate of degradation of fenpyroximate was investigated in one German loamy sand soil (Römbke, J., Brodesser J., 1992, report No.: E-4020). 100 g Soil samples in glass containers were treated with fenpyroximate at a rate of 20 µg/100 g soil (equivalent to 150 g ai/ha), and incubated under aerobic conditions at 20 °C in the dark. The soil samples at 0, 2, 4, 8, 16, 32, 64 and 100 DAT were extracted three times by shaking with acetone:water (2:1, v/v), and were analysed by HPLC with UV detection using two reversed phase columns and a column switching procedure. The DT_{50} and DT_{90} values have been calculated as 159.0 and >200 days, respectively, according to methods recommended by the BBA.

Table 91 Characteristics of soil used in aerobic soil study

Soil name	pH (KCl)	OC (%)	Sand (%)	Silt (%)	Clay (%)	Classification	MWHC (%)
Standard 2.2*	5.6	2.29	82.0	13.0	5.1	Loamy sand	44.3

OC=organic carbon, MWHC=maximum water holding capacity

Table 92 Determination of fenpyroximate after application to Standard Soil 2.2

DAT	Analysed concentration (mg/kg)	Average concentration (mg/kg)	% nominal concentration (average)
0	0.123, 0.207, 0.150, 0.233	0.178	89
2	0.207, 0.212, 0.197, 0.197	0.203	102
4	0.201, 0.178, 0.166, 0.197	0.186	93
8	0.185, 0.193, 0.167, 0.179	0.181	91
16	0.139, 0.143, 0.193, 0.179	0.164	82
32	0.148, 0.303, 0.183, 0.220	0.214	107
64	0.153, 0.097, 0.093, 0.196	0.135	68

DAT	Analysed concentration (mg/kg)	Average concentration (mg/kg)	% nominal concentration (average)
100	0.131, 0.130, 0.100, 0.131	0.123	62

Kinetic assessment of data from six studies with 12 aerobic soils (eight European, two Japanese and two US soils) in the laboratory (report No. E-4005, E-4008, E-4020, E-4041, E-4039 and E-4052) were analysed using the CAKE version 3.2 (2016) software package according to guidance provided by FOCUS (2011) (Graham R., 2016, report No. E-4058). DT_{50} and DT_{90} values were calculated for comparison with relevant study triggers and persistence criteria. DT_{50} values were normalised to 20 °C and pF 2 soil moisture content for selection of suitable modelling endpoints. The fenpyroximate persistence/trigger DT_{50} values ranged from 6.64 to 238 days and DT_{90} values ranged from 88.1 to 863 days. The fenpyroximate modelling DT_{50} values (at 20 °C and pF2) ranged from 12.9 days to 238 days, with a geometric mean of 51.2 days).

Table 93 Evaluation of persistence/trigger endpoints for fenpyroximate from laboratory studies

Soil	Kinetic model	χ^2 err	Confidence measure	Visual assessment	DT_{50} (d)	DT_{90} (d)
Ehime	SFO	5.1	k: p=1.77×10^{-5}	Good	39.9	132
	FOMC	5.51	A^a β^a	Good	34.1	114
Kanagawa	SFO	6.73	k: p=1.62×10^{-7}	Good	26.5	88.1
	FOMC	7.27	α^{nd} β^{nd}	Good	22.5	74.7
Field soil I	SFO	19.7	k: p=4.70×10^{-4}	Poor	20.6	68.5
	FOMC	9.78	A^b β^a	Good	6.64	630
	DFOP	9.19	k1: p=0.2823 k2: p=5.68×10^{-5}	Good	10.4	93.2
Field soil II	SFO	16.9	k: p=7.58×10^{-4}	Poor	17.8	59
	FOMC	13.1	α^b β^a	Good	7.28	554
	DFOP	6.6	k1: p=nd k2: p=2.18×10^{-4}	Good	10.9	78.7
Field soil III	SFO	17.7	k: p=0.001133	Poor	35.5	118
	FOMC	13.7	α^b β^a	Intermediate	9.47	>1000
	DFOP	7.51	k1: p=0.493 k2: p=6.32×10^{-5}	Good	17.9	137
Standard 2.2	SFO	8.77	k: p=0.002488	Poor	171	568
	FOMC	9.49	α^a β^a	Poor	198	1.23×10^3
	SFO*	4.26	k: p=9.87×10^{-5}	Good	150	498
	FOMC*	4.1	α^a β^a	Good	250	>10 000
SK108672	SFO	10.7	k: p=4.44 x 10^{-4}	Good	33.3	111
	FOMC	5.59	α^b β^a	Excellent	21.6	236
	DFOP	6.01	k1: p=0.08685 k2: p=0.0259	Excellent	21.6	154
Tulare County	SFO	10.4	k: p=3.16×10^{-26}	Good	38.2	127
	FOMC	10.7	α^2 β^2	Good	30.1	100
Speyer 2.2	SFO	1.62	k: p=1.33×10^{-13}	Excellent	238	789
	FOMC	1.6	α^a β^a	Excellent	372	>10 000
Speyer 5M	SFO	9.39	k: p=3.71×10^{-14}	Good	26.1	86.6
	FOMC	2.1	α^b β^b	Excellent	21.1	126
	DFOP	3.56	k1: p=5.73×10^{-6} k2: p=0.04769	Excellent	21.6	131

Soil	Kinetic model	χ^2 err	Confidence measure	Visual assessment	DT_{50} (d)	DT_{90} (d)
Speyer 6S	SFO	9.67	k: p=7.06 × 10^{-15}	Intermediate	63.2	210
	FOMC	6.55	α^b β^b	Excellent	46.2	371
	DFOP	4.36	k1: p=0.4768 k2: p=9.25 × 10^{-18}	Excellent	49.6	221
Iowa	SFO	15.1	k: p=8.55 x 10^{-11}	Intermediate	70.2	233
	FOMC	3.82	α^{a1} β^b	Excellent	33	863
	DFOP	3.63	k1: p=4.96 × 10^{-8} k2: p=0.01478	Excellent	30.7	603

[a] 95th percentile confidence interval includes zero,

[b] 95th percentile confidence interval does not include zero

nd=not determined;

DFOP: distribution-free one-pass model;

FOMC: first-order multi-compartment model;

SFO: single first order.

Table 94 Evaluation of modelling endpoints for fenpyroximate from laboratory studies

Soil	Kinetic model	χ^2 err	Confidence measure	Visual assessment	DT_{50} (d)	DT_{90} (d)	$DT_{50\ MOD}$ (d)
Ehime	SFO	5.1	k: p=1.77 × 10^{-5}	Good	39.9	132	39.9
Kanagawa	SFO	6.73	k: p=1.62 × 10^{-7}	Good	26.5	88.1	26.5
Field soil I	SFO	19.7	k: p=4.70 × 10^{-4}	Poor	20.6	68.5	-
	DFOP	9.19	k1: p=0.2823 k2: p=5.68 × 10^{-5}	Good	10.4	93.2	35.7 [c]
	HS	16.5	k1: p=0.001373 k2: p=0.02998	Good	8.48	131	-
Field soil II	SFO	16.9	k: p=7.58 × 10^{-4}	Poor	17.8	59	-
	FOMC	13.1	α^b β^a	Good	7.28	554	167 [d]
Field soil III	SFO	17.7	k: p=0.001133	Poor	35.5	118	-
	DFOP	7.51	k1: p=0.493 k2: p=6.32 × 10^{-5}	Good	17.9	137	51.4 [c]
	HS	7.5	k1: p=0.4999 k2: p=1.26 × 10^{-4}	Intermediate	17.8	137	-
Standard 2.2	SFO	8.77	k: p=0.002488	Poor	171	568	-
	SFO*	4.26	k: p=9.87 × 10^{-5}	Good	150	498	150
SK108672	SFO	10.7	k: p=4.44 × 10^{-4}	Good	33.3	111	33.3
Tulare County	SFO	10.4	k: p=3.16 × 10^{-26}	Good	38.2	127	38.2
Speyer 2.2	SFO	1.62	k: p=1.33 × 10^{-13}	Excellent	238	789	238
Speyer 5M	SFO	9.39	k: p=3.71 × 10^{-14}	Good	26.1	86.6	26.1
Speyer 6S	SFO	9.67	k: p=7.06 × 10^{-15}	Intermediate	63.2	210	63.2
Iowa	SFO	15.1	k: p=8.55 × 10^{-11}	Intermediate	70.2	233	70.2

[a] 95th percentile confidence interval includes zero,

[b] 95th percentile confidence interval does not include zero,

[c] Overall days,

[d] DT_{90} / 3.32;

DFOP: distribution-free one-pass model;

FOMC: first-order multi-compartment model;

SFO: single first order;

HS: hockey-stick.

A summary of the best-fit persistence/trigger endpoints is shown in Table 95. The DT_{50} values of fenpyroximate ranged from 6.64 to 238 days and DT_{90} values ranged from 88.1 to 863 days.

A summary of the modelling endpoints is shown in Table 96. The DT_{50} values (at 20 °C and pF2) ranged from 12.9 days to 238 days, with a geometric mean of 51.2 days.

Table 95 Summary of persistence/trigger endpoints for fenpyroximate from laboratory studies

Soil (USDA)	pH	Temp. (°C)	Moisture content (% MWHC)	DT_{50} (days)	DT_{90} (days)	χ^2 error (%)	Kinetics
Ehime (sand [a])	5.8[c]	25	15.1[f]	39.9	132	5.1	SFO
Kanagawa (sandy loam [a])	5.5[c]	25	50.7[f]	26.5	88.1	6.73	SFO
Field soil I (clay loam[b])	6.9[d]	20	40	6.64	630	9.78	FOMC
Field soil II (silt loam[b])	6.3[d]	20	40	7.28	554	13.1	FOMC
Field soil III (loam[b])	5.9[d]	20	40	17.9[g]	137	7.51	DFOP
Standard 2.2 (loamy sand[b])	5.6[d]	20	40	150	498	4.26	SFO
SK 108672 (silt loam)	6.8[e]	10	45	21.6	236	5.59	FOMC
Tulare County (sandy loam)	8.6[e]	25	11.7[f]	38.2	127	10.4	SFO
Speyer 2.2 (loamy sand)	5.4[e]	20	pF2	238	789	1.62	SFO
Speyer 5M (sandy loam)	7.1[e]	20	pF2	21.1	126	2.1	FOMC
Speyer 6S (clay)	6.5[e]	20	pF2	46.2	371	6.55	FOMC
Iowa (silt loam)	5.7[e]	20	pF2	33	863	3.82	FOMC
		Worst-case		238	863		
		pH dependence		No			

[a] Classification estimated from values for sand, silt and clay stated in report, assuming particle size, according to USDA guidance,

[b] converted to USDA classification,

[c] medium measured in not stated,

[d] measured in KCl,

[e] measured in H_2O,

[f] actual soil moisture content (%),

[g] overall days,

MWHC=maximum water holding capacity

Table 96 Summary of modelling endpoints (corrected to pF2 and 20 °C) for fenpyroximate from laboratory studies

Soil (USDA)	pH (water)	Temp (°C)	Moisture content (% MWHC)	DT_{50} (days)	DT_{90} (days)	Modelling DT_{50} at pF2 and 20 °C (days)	χ^2 error (%)	Kinetics
Ehime (sand[a])	5.8[c]	25	15.1[f]	39.9	132	62.6	5.1	SFO
Kanagawa (sandy loam[a])	5.5[c]	25	50.7[f]	26.5	88.1	41.6	6.73	SFO
Field soil I (clay loam[b])	6.9[d]	20	40	35.7[g]	93.2	30.5	9.19	DFOP
Field soil II (silt loam[b])	6.3[d]	20	40	167[h]	554	146.7	13.3	FOMC
Field soil III (loam[b])	5.9[d]	20	40	51.4[g]	137	45.6	7.51	DFOP
Standard 2.2 (loamy sand[b])	5.6[d]	20	40	150	498	150.0	4.26	SFO
SK 108672 (silt loam)	6.8[e]	10	45	33.3	111	12.9	10.7	SFO
Tulare County (sandy loam)	8.6[e]	25	11.7[f]	38.2	127	42.7	10.4	SFO
Speyer 2.2 (loamy sand)	5.4[d]	20	pF2	238	789	238	1.62	SFO
Speyer 5M (sandy loam)	7.1[d]	20	pF2	26.1	86.6	26.1	9.39	SFO
Speyer 6S (clay)	6.5[4]	20	pF2	63.2	210	63.2	9.67	SFO

Soil (USDA)	pH (water)	Temp (°C)	Moisture content (% MWHC)	DT_{50} (days)	DT_{90} (days)	Modelling DT_{50} at pF2 and 20 °C (days)	χ^2 error (%)	Kinetics
Iowa (silt loam)	5.7[d]	20	pF2	70.2	233	70.2	15.1	SFO
		Geometric mean				51.2[i]		
		pH dependence		No				

[a] Classification estimated from values for sand, silt and clay stated in report, assuming particle size according to USDA guidance,

[b] converted to USDA classification,

[c] medium measured in not stated,

[d] measured in KCl,

[e] measured in H_2O,

[f] actual soil moisture content (%),

[g] slow phase,

[h] DT_{90} / 3.32,

[i] Standard 2.2 and Speyer 2.2 are the same soil. The geometric mean DT_{50} value for these soils was calculated first and included as a single value in the overall geometric mean calculation,

MWHC=maximum water holding capacity

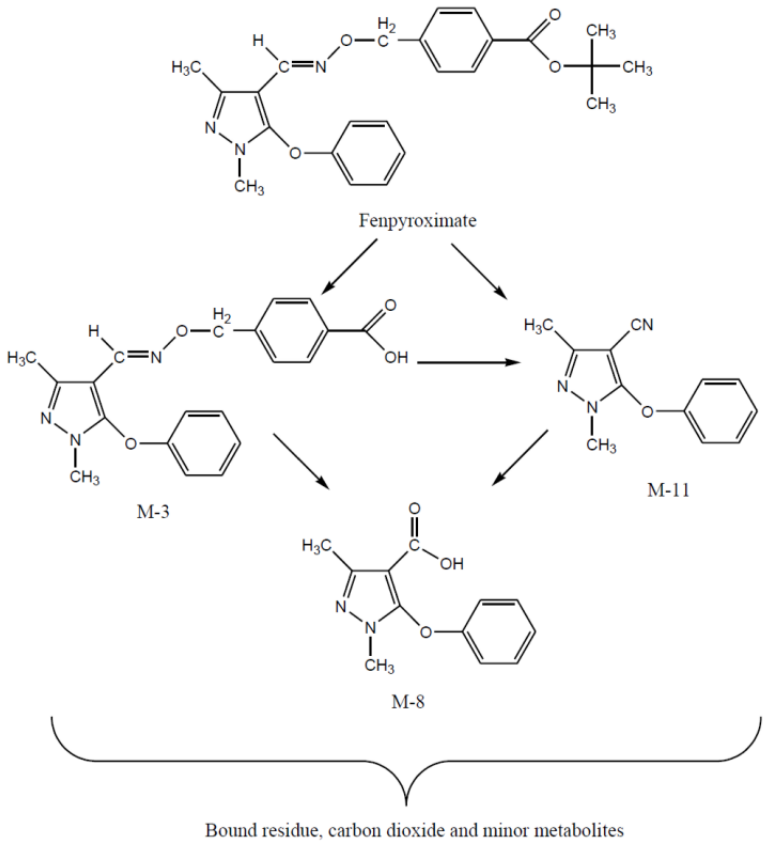


Figure 4 Proposed metabolic pathway for Fenpyroximate in soil

Environmental fate in water

The hydrolysis of fenpyroximate was studied in sterile aqueous solution buffered at pH 5, 7 and 9 for thirty days at 25 °C (Saxena A., McCann D., 1992, E-4013). Sterilised aqueous solutions buffered at pH 5, 7 and 9 were fortified with [pyrazole-^{14}C]-fenpyroximate in acetonitrile at concentrations of 9.5 ng/mL (ppb) buffer solution. The sample containers were maintained in a dark at 25 °C ± 1 °C.

Duplicate samples for each pH were collected at 0, 1, 2, 4, 7, 14, 21 and 30 DAT, and were extracted by partitioning with ethyl acetate (the pH 9 samples were acidified prior to extraction) and analysed by HPLC with selected samples analysed by 2d-TLC.

The total mean radioactivity recovered by extraction relative to the applied radioactivity ranged from 90.4% to 99.7% AR, with one exception (pH 7, day 30) where a mean value of 116.0% AR was reported. Fenpyroximate degraded from mean relative values of 100% at 0 DAT, to 88.5%, 92.5% and 88.6% at pH 5, 7 and 9, respectively. The principal hydrolysis product observed at each pH was M-3 which did not exceed 10.1% of the total radioactivity in any of the samples. M-1 was only observed at pH 5 and pH 9 and represented a maximum of approximately 7% of the injected radioactivity. Radiolabelled fenpyroximate degradation was very slow at each pH studied. The half-life for the hydrolytic degradation of [pyrazole-^{14}C]-fenpyroximate was calculated to be 180 days for pH 5 (correlation coefficient r^2=0.9671), 226 days for pH 7 (r^2=0.9344) and 221 days for pH 9 (r^2=0.6586).

Table 97 Mean recovery of AR after treatment of [^{14}C]fenpyroximate to sterile buffer solutions

Day	Mean mass balance (%)[a]		
	pH 5 buffer solutions	pH 7 buffer solutions	pH 9 buffer solutions
0	95.8	99.2	98.9
1	97.8	98.0	97.3
2	98.0	94.3	98.3
4	99.7	97.5	99.4
7	95.3	90.4	95.8
14	96.8	95.0	94.9
21	96.6	95.1	98.3
30	94.4	116.0	98.8

[a] Mean of two samples; mass balance calculated relative to the applied radioactivity on day 0

The principal hydrolysis product observed at each pH was M-3 and did not exceed 10.1% of the total radioactivity on any of the samples. M-1 was only observed at pH 5 and pH 9 and represented a maximum of approximately 7% of the injected radioactivity. The composition of hydrolysis samples during the course of the study are summarised in Tables 98–100.

Table 98 Percentage composition of fenpyroximate and its hydrolysis products for pH 5 buffer solution

Day	pH 5 buffer		
	Fenpyroximate [a] (%)	M-1[a] (%)	M-3[a] (%)
0	100.0	-	-
1	100.0	-	-
2	100.0	-	-
4	98.46	1.55	-
7	97.79	2.22	-
14	95.85	1.67	2.69
21	93.91	1.76	7.02
30	88.47	4.54	8.49

[a] Mean of two samples; percent of injected radioactivity

Table 99 Percentage composition of fenpyroximate and its hydrolysis products for pH 7 buffer solution

Day	pH 7 buffer		
	Fenpyroximate [a] (%)	M-1[a] (%)	M-3[a] (%)
0	100	-	-
1	100	-	-
2	100	-	-
4	100	-	-
7	100	-	-
14	95.67	-	4.33
21	93.33	-	6.67
30	92.45	-	7.56

[a] Mean of two samples; percent of injected radioactivity

Table 100 Percentage composition of fenpyroximate and its hydrolysis products for pH 9 buffer solution

Day	pH 9 buffer		
	Fenpyroximate [a] (%)	M-1 [a] (%)	M-3[a] (%)
0	100	-	-
1	97.1	2.90	-
2	100	-	-
4	93.75	4.51	1.75
7	96.66	3.35	-
14	95.14	2.18	2.69
21	88.63	4.35	7.02
30	91.52	-	8.49

[a] Mean of two samples; percent of injected radioactivity

RESIDUE ANALYSIS

The meeting received the information on validation of analysis method for determination of residues of fenpyroximate and its metabolites in plant matrices, animal matrices and soil.

Analytical methods

The commonly used methods are summarised below:

Method No.	Matrix	Analyte	LOQ (mg/kg)	Method principle
Plant matrices				
RES/RAM/004 RES-0029	Apple Rape seed Bean Wheat Orange Sugar beet hops	fenpyroximate M-1	0.01 0.01	Extracted with acetonitrile Purified with SPE, PSA LC-MS/MS analysis

Method No.	Matrix	Analyte	LOQ (mg/kg)	Method principle
DFG S19	Apple (mash, cidar) grapes (cidar, wine) orange cucumber/courgette tomato strawberry eggplant pear melon hops (dregs, yeast, beer) bean plum peach paprika cotton	fenpyroximate M-1	0.01 0.01	Extracted with acetone cleaned with GPC GC-N-FID analysis
Method P-14.045.02 Hoechst method AL 015/90-0	tomato	fenpyroximate M-1	0.05 0.05	Extracted with acetonitrile Partitioned with n-hexane and acetronitrile cleaned with silica gel column HPLC-UV analysis
RES/RAM/005 EN15662:2008	Apple (juice, pomace, sauce) Grape (raisin, juice, wine) Tomato (juice, puree) Strawberry (jam) Bean Malt Hop (beer)	fenpyroximate M-1 M-3	0.01 0.01 0.01	Extracted with acetonitrile Purified with PSE, PSAwith n-hexane and acetronitrile HPLC-MS/MS analysis
Animal matrices				
QuEChERS method	musle kidney liver fat milk egg	fenpyroximate M-3	0.005 0.005	Extracted with acetonitrile Partitioned with n-hexane HPLC-MS/MS analysis
Soil				
RES-000601	soil	fenpyroximate	0.01	Extracted with methanol, acetone HPLC-MS/MS analysis

Method for plant matrices

Monitoring Method RES/RAM/004 (RES-00029)

The QuEChERS method (RES-00029, RES/RAM/004) for the determination of residues of fenpyroximate and M-1 in crop matrices by LC-MS/MS was validated (Watson, G, 2016a, report No. A-4088). 10 g sample of high moisture crops was extracted with acetonitrile (10 mL acetonitrile, 2 minutes vortex extraction), or 5 g sample of low moisture crops was extracted with acetonitrile (6.5 mL water and 10 mL acetonitrile, 2 minutes vortex extraction), or 1 g sample of difficult matrices such as hops was extracted with water and acetonitrile (approximately 9 mL water and 10 mL acetonitrile, 2 minutes vortex extraction), followed by liquid-liquid partition with magnesium sulphate, sodium chloride and sodium citrate. After centrifugation, an aliquot of the acetonitrile phase (8 mL) was transferred to a centrifuge tube and stored overnight in a freezer. Extracts were removed from frozen storage and immediately centrifuged at 3000 rpm for 1 minute. Purification of an aliquot of the acetonitrile extract by dispersive SPE with primary/secondary amine (PSA) is prior to dilution and then determination.

All samples were measured using LC-MS/MS employing an Ascentis Express C18 column, 2.7 µm particle size and monitoring at two MRM transitions for each analyte (all matrices except hops). For hops a second column was used for confirmation, Phenomenex, Kinetex Biphenyl column,

2.6 µm particle size. Quantification of extracts was performed using external calibration standards over the range 0.1 to 10.0 ng/mL for all crops except 0.1 to 5.0 ng/mL in sugar beet roots and 0.15 to 10 ng/mL for hops. For all crops, matrix matched standards were used. Results are determined as fenpyroximate or M-1 and expressed as mg/kg. The method was validated for linearity, specificity precision and accuracy. The lowest level tested (LOQ) was 0.01 mg/kg fenpyroximate or M-1 in all crop matrices.

Table 101 Recovery results from method validation of fenpyroximate using the analytical method RES/RAM/004 in crops

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Apple (high water)	fenpyroximate	0.01 (n=5)	103 to 106% (Mean 104%)	1.6%	MRM1 422→366
Apple (high water)	fenpyroximate	0.1 (n=5)	101 to 104% (Mean 102%)	1.2%	MRM1 422→366
Apple (high water)	fenpyroximate	Overall (n=10)	101 to 106% (Mean 103%)	1.8%	MRM1 422→366
Apple (high water)	fenpyroximate	0.01 (n=5)	100 to 107% (Mean 103%)	3.0%	MRM2 422→135
Apple (high water)	fenpyroximate	0.1 (n=5)	100 to 106% (Mean 102%)	2.1%	MRM2 422→135
Apple (high water)	fenpyroximate	Overall (n=10)	100 to 107% (Mean 103%)	2.5%	MRM2 422→135
Oilseed rape seed (high oil)	fenpyroximate	0.01 (n=5)	78 to 80% (Mean 79%)	0.8%	MRM1 422→366
Oilseed rape seed (high oil)	fenpyroximate	0.1 (n=5)	76 to 79% (Mean 77%)	1.5%	MRM1 422→366
Oilseed rape seed (high oil)	fenpyroximate	Overall (n=10)	76 to 80% (Mean 78%	1.7%	MRM1 422→366
Oilseed rape seed (high oil)	fenpyroximate	0.01 (n=5)	78 to 87% (Mean 82%)	4.9%	MRM2 422→135
Oilseed rape seed (high oil)	fenpyroximate	0.1 (n=5)	76 to 79% (Mean 78%)	1.7%	MRM2 422→135
Oilseed rape seed (high oil)	fenpyroximate	Overall (n=10)	76 to 87% (Mean 80%)	4.5%	MRM2 422→135
Dried beans (dry matrix)	fenpyroximate	0.01 (n=5)	94 to 98% (Mean 96%)	1.5%	MRM1 422→366
Dried beans (dry matrix)	fenpyroximate	0.1 (n=5)	95 to 98% (Mean 96%)	1.5%	MRM1 422→366
Dried beans (dry matrix)	fenpyroximate	Overall (n=10)	94 to 98% (Mean 96%)	1.4%	MRM1 422→366
Dried beans (dry matrix)	fenpyroximate	0.01 (n=5)	97 to 101% (Mean 99%)	1.5%	MRM2 422→135
Dried beans (dry matrix)	fenpyroximate	0.1 (n=5)	96 to 99% (Mean 103%)	1.4%	MRM2 422→135
Dried beans (dry matrix)	fenpyroximate	Overall (n=10)	96 to 101% (Mean 98%)	1.7%	MRM2 422→135
Wheat grain (dry, high starch)	fenpyroximate	0.01 (n=5)	96 to 99% (Mean 98%)	2.0%	MRM1 422→366
Wheat grain (dry, high starch)	fenpyroximate	0.1 (n=5)	95 to 98% (Mean 96%)	1.1%	MRM1 422→366
Wheat grain (dry, high starch)	fenpyroximate	Overall (n=10)	95 to 99% (Mean 97%)	1.8%	MRM1 422→366
Wheat grain (dry, high starch)	fenpyroximate	0.01 (n=5)	93 to 102% (Mean 99%)	4.1%	MRM2 422→135
Wheat grain (dry, high starch)	fenpyroximate	0.1 (n=5)	95 to 97% (Mean 96%)	0.7%	MRM2 422→135
Wheat grain (dry, high starch)	fenpyroximate	Overall (n=10)	93 to 102% (Mean 97%)	3.2%	MRM2 422→135
Oranges (high acid)	fenpyroximate	0.01 (n=5)	107 to 110% (Mean 109%)	1.2%	MRM1 422→366
Oranges (high acid)	fenpyroximate	0.1 (n=5)	104 to 107% (Mean 106%)	1.4%	MRM1 422→366

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Oranges (high acid)	fenpyroximate	Overall (n=10)	104 to 110% (Mean 107%)	1.8%	MRM1 422→366
Oranges (high acid)	fenpyroximate	0.01 (n=5)	106 to 108% (Mean 107%)	0.4%	MRM2 422→135
Oranges (high acid)	fenpyroximate	0.1 (n=5)	104 to 108% (Mean 106%)	1.6%	MRM2 422→135
Oranges (high acid)	fenpyroximate	Overall (n=10)	104 to 108% (Mean 107%)	1.1%	MRM2 422→135
Sugar beet roots (high water)	fenpyroximate	0.01 (n=5)	104 to 108% (Mean 106%)	2.0%	MRM1 422→366
Sugar beet roots (high water)	fenpyroximate	0.1 (n=5)	104 to 107% (Mean 105%)	1.4%	MRM1 422→366
Sugar beet roots (high water)	fenpyroximate	Overall (n=10)	104 to 108% (Mean 105%)	1.6%	MRM1 422→366
Sugar beet roots (high water)	fenpyroximate	0.01 (n=5)	104 to 109% (Mean 107%)	1.9%	MRM2 422→135
Sugar beet roots (high water)	fenpyroximate	0.1 (n=5)	102 to 108% (Mean 105%)	2.3%	MRM2 422→135
Sugar beet roots (high water)	fenpyroximate	Overall (n=10)	102 to 109% (Mean 106%)	2.2%	MRM2 422→135
Sugar beet tops (high water)	fenpyroximate	0.01 (n=5)	100 to 104% (Mean 101%)	1.4%	MRM1 422→366
Sugar beet tops (high water)	fenpyroximate	0.1 (n=5)	99 to 102% (Mean 100%)	1.5%	MRM1 422→366
Sugar beet tops (high water)	fenpyroximate	Overall (n=10)	99 to 104% (Mean 101%)	1.5%	MRM1 422→366
Sugar beet tops (high water)	fenpyroximate	0.01 (n=5)	99 to 111% (Mean 106%)	5.1%	MRM2 422→135
Sugar beet tops (high water)	fenpyroximate	0.1 (n=5)	98 to 103% (Mean 101%)	1.8%	MRM2 422→135
Sugar beet tops (high water)	fenpyroximate	Overall (n=10)	98 to 103% (Mean 101%)	1.8%	MRM2 422→135
Hops (difficult matrix)	fenpyroximate	0.01 (n=5)	82 to 96% (Mean 90%)	6.3%	MRM 422→366 Column 1
Hops (difficult matrix)	fenpyroximate	0.1 (n=5)	88 to 97% (Mean 92%)	4.8%	MRM 422→366 Column 1
Hops (difficult matrix)	fenpyroximate	Overall (n=10)	82 to 97% (Mean 91%)	5.4%	MRM 422→366 Column 1
Hops (difficult matrix)	fenpyroximate	0.01 (n=5)	87 to 95% (Mean 92%)	3.5%	MRM 422→366 Column 2
Hops (difficult matrix)	fenpyroximate	0.1 (n=5)	88 to 95% (Mean 92%)	2.9%	MRM 422→366 Column 2
Hops (difficult matrix)	fenpyroximate	Overall (n=10)	87 to 95% (Mean 92%)	3.0%	MRM 422→366 Column 2

Table 102 Recovery results from method validation of M-1 using the analytical method RES/RAM/004 in crops

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Apple (high water)	M-1	0.01 (n=5)	99 to 104% (Mean 102%)	2.0%	MRM1 422→366
Apple (high water)	M-1	0.1 (n=5)	99 to 103% (Mean 101%)	1.3%	MRM1 422→366
Apple (high water)	M-1	Overall (n=10)	99 to 104% (Mean 102%)	1.8%	MRM1 422→366
Apple (high water)	M-1	0.01 (n=5)	98 to 107% (Mean 104%)	3.7%	MRM2 422→135
Apple (high water)	M-1	0.1 (n=5)	100 to 104% (Mean 101%)	1.5%	MRM2 422→135
Apple (high water)	M-1	Overall (n=10)	98 to 107% (Mean 102%)	3.1%	MRM2 422→135
Oilseed rape seed (high oil)	M-1	0.01 (n=5)	79 to 83% (Mean 82%)	2.1%	MRM1 422→366
Oilseed rape seed (high oil)	M-1	0.1 (n=5)	77 to 80% (Mean 79%)	1.5%	MRM1 422→366

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Oilseed rape seed (high oil)	M-1	Overall (n=10)	77 to 83% (Mean 80%	2.5%	MRM1 422→366
Oilseed rape seed (high oil)	M-1	0.01 (n=5)	82 to 91% (Mean 86%)	4.1%	MRM2 422→135
Oilseed rape seed (high oil)	M-1	0.1 (n=5)	78 to 81% (Mean 79%)	1.8%	MRM2 422→135
Oilseed rape seed (high oil)	M-1	Overall (n=10)	78 to 91% (Mean 83%)	5.7%	MRM2 422→135
Dried beans (dry matrix)	M-1	0.01 (n=5)	93 to 97% (Mean 96%)	1.5%	MRM1 422→366
Dried beans (dry matrix)	M-1	0.1 (n=5)	94 to 98% (Mean 96%)	1.6%	MRM1 422→366
Dried beans (dry matrix)	M-1	Overall (n=10)	93 to 98% (Mean 96%)	1.5%	MRM1 422→366
Dried beans (dry matrix)	M-1	0.01 (n=5)	88 to 100% (Mean 94%)	5.6%	MRM2 422→135
Dried beans (dry matrix)	M-1	0.1 (n=5)	94 to 96% (Mean 95%)	1.0%	MRM2 422→135
Dried beans (dry matrix)	M-1	Overall (n=10)	88 to 100% (Mean 95%)	3.8%	MRM2 422→135
Wheat grain (dry, high starch)	M-1	0.01 (n=5)	99 to 103% (Mean 101%)	1.7%	MRM1 422→366
Wheat grain (dry, high starch)	M-1	0.1 (n=5)	95 to 101% (Mean 98%)	2.4%	MRM1 422→366
Wheat grain (dry, high starch)	M-1	Overall (n=10)	95 to 103% (Mean 99%)	2.5%	MRM1 422→366
Wheat grain (dry, high starch)	M-1	0.01 (n=5)	98 to 105% (Mean 101%)	2.5%	MRM2 422→135
Wheat grain (dry, high starch)	M-1	0.1 (n=5)	93 to 97% (Mean 95%)	1.9%	MRM2 422→135
Wheat grain (dry, high starch)	M-1	Overall (n=10)	93 to 105% (Mean 98%)	3.7%	MRM2 422→135
Oranges (high acid)	M-1	0.01 (n=5)	108 to 112% (Mean 110%)	1.3%	MRM1 422→366
Oranges (high acid)	M-1	0.1 (n=5)	106 to 111% (Mean 108%)	1.8%	MRM1 422→366
Oranges (high acid)	M-1	Overall (n=10)	106 to 112% (Mean 109%)	1.7%	MRM1 422→366
Oranges (high acid)	M-1	0.01 (n=5)	107 to 113% (Mean 110%)	1.9%	MRM2 422→135
Oranges (high acid)	M-1	0.1 (n=5)	105 to 111% (Mean 108%)	2.0%	MRM2 422→135
Oranges (high acid)	M-1	Overall (n=10)	105 to 113% (Mean 109%)	2.2%	MRM2 422→135
Sugar beet roots (high water)	M-1	0.01 (n=5)	103 to 107% (Mean 105%)	1.3%	MRM1 422→366
Sugar beet roots (high water)	M-1	0.1 (n=5)	104 to 106% (Mean 105%)	0.9%	MRM1 422→366
Sugar beet roots (high water)	M-1	Overall (n=10)	103 to 107% (Mean 105%)	1.1%	MRM1 422→366
Sugar beet roots (high water)	M-1	0.01 (n=5)	103 to 111% (Mean 105%)	3.9%	MRM2 422→135
Sugar beet roots (high water)	M-1	0.1 (n=5)	106 to 108% (Mean 106%)	0.8%	MRM2 422→135
Sugar beet roots (high water)	M-1	Overall (n=10)	103 to 111% (Mean 106%)	2.7%	MRM2 422→135
Sugar beet tops (high water)	M-1	0.01 (n=5)	100 to 102% (Mean 101%)	1.1%	MRM1 422→366
Sugar beet tops (high water)	M-1	0.1 (n=5)	99 to 103% (Mean 101%)	1.7%	MRM1 422→366
Sugar beet tops (high water)	M-1	Overall (n=10)	99 to 103% (Mean 101%)	1.3%	MRM1 422→366
Sugar beet tops (high	M-1	0.01 (n=5)	104 to 118%	5.5%	MRM2 422→135

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
water)			(Mean 108%)		
Sugar beet tops (high water)	M-1	0.1 (n=5)	98 to 105% (Mean 101%)	2.4%	MRM2 422→135
Sugar beet tops (high water)	M-1	Overall (n=10)	98 to 118% (Mean 105%)	5.7%	MRM2 422→135
Hops (difficult matrix)	M-1	0.01 (n=5)	88 to 100% (Mean 94%)	5.0%	MRM 422→366 Column 1
Hops (difficult matrix)	M-1	0.1 (n=5)	96 to 100% (Mean 98%)	2.3%	MRM 422→366 Column 1
Hops (difficult matrix)	M-1	Overall (n=10)	88 to 100% (Mean 96%)	4.2%	MRM 422→366 Column 1
Hops (difficult matrix)	M-1	0.01 (n=5)	93 to 103% (Mean 102%)	1.5%	MRM 422→366 Column 2
Hops (difficult matrix)	M-1	0.1 (n=5)	95 to 99% (Mean 96%)	1.9%	MRM 422→366 Column 2
Hops (difficult matrix)	M-1	Overall (n=10)	93 to 103% (Mean 99%)	3.3%	MRM 422→366 Column 2

Table 103 Characteristics for the analytical method used for validation of fenpyroximate and M-1 residues in crops

	Fenpyroximate	M-1
Specificity (all crops)	blank value < 30% LOQ Product ion MS scan presented	blank value < 30% LOQ Product ion MS scan presented
Calibration (type, number of data points)	typical calibration data presented typical calibration line equation presented linear, no weighting applied r=≥ 0.999 for all crops except r=> 0.997 for hops on C18 column 7 data points Matrix matched used Both MRM1 and MRM2 were reported for all crops except hops. For hops linearity for both columns was reported	typical calibration data presented| typical calibration line equation presented linear, no weighting applied r=>0.998 7 data points in all crops except 6 data points in sugar beet roots Matrix matched used Both MRM1 and MRM2 were reported for all crops except hops. For hops linearity for both columns was reported
Calibration range	0.1 to 10.0 ng/mL for all crops except 0.15 to 10 ng/mL for hops Corresponding calibration range 0.0025 to 0.25 mg/kg fenpyroximate in high moisture, high acid, dried and high oil matrices. Corresponding calibration range 0.003 to 0.2 mg/kg fenpyroximate in difficult matrices (hops).	0.1 to 10.0 ng/mL all crops except 0.1 to 5.0 ng/mL in sugar beet roots and 0.15 to 10 ng/mL for hops Corresponding calibration range 0.0025 to 0.25 mg/kg M-1 in high moisture (except sugar beet roots), high acid, dried and high oil matrices. Corresponding calibration range 0.0025 to 0.125 mg/kg M-1 in sugar beet roots. Corresponding calibration range 0.003 to 0.2 mg/kg M-1 in difficult matrices (hops).
Assessment of matrix effects is presented	Yes, significant only in hops but matrix matched standards used for all crops	Yes, significant only in hops but matrix matched standards used for all crops
Limit of determination/quantification	LOQ=0.01 mg/kg in all crops	LOQ=0.01 mg/kg in all crops
Comment	Acceptable against SANCO/825/00 rev 8.1 criteria. Method suitable for monitoring in all matrix types.	Acceptable against SANCO/825/00 rev 8.1 criteria. Method suitable for monitoring in all matrix types.

The method (RES/RAM/004 based on QuEChERS) was further validated by Independent Laboratory successfully in different matrices of plant origin (Gasso-Brown, D., 2016a, report No. A-4089; 2016b, report No. S16-06628) for linearity, specificity precision and accuracy. The LOQ was 0.01 mg/kg for fenpyroximate or M-1 in all crop matrices

Table 104 Recovery results from independent method validation of fenpyroximate using the analytical method RES/RAM/004 in crops

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Apple (high water)	fenpyroximate	0.01 (n=5)	105 to 119% (Mean 114%)	4.6%	MRM1 422→366
Apple (high water)	fenpyroximate	0.1 (n=5)	94 to 103% (Mean 99%)	3.4%	MRM1 422→366
Apple (high water)	fenpyroximate	Overall (n=10)	94 to 119% (Mean 106%)	8.3%	MRM1 422→366
Apple (high water)	fenpyroximate	0.01 (n=5)	104 to 120% (Mean 114%)	5.3%	MRM2 422→135
Apple (high water)	fenpyroximate	0.1 (n=5)	94 to 102% (Mean 98%)	2.9%	MRM2 422→135
Apple (high water)	fenpyroximate	Overall (n=10)	94 to 120% (Mean 106%)	9.1%	MRM2 422→135
Oilseed rape seed (high oil)	fenpyroximate	0.01 (n=5)	59 to 91% (Mean 75%)	18.7%	MRM1 422→366
Oilseed rape seed (high oil)	fenpyroximate	0.1 (n=5)	78 to 98% (Mean 86%)	8.8%	MRM1 422→366
Oilseed rape seed (high oil)	fenpyroximate	Overall (n=10)	59 to 98% (Mean 80%	14.9%	MRM1 422→366
Oilseed rape seed (high oil)	fenpyroximate	0.01 (n=5)	56 to 114% (Mean 80%)	28.7%	MRM2 422→135
Oilseed rape seed (high oil)	fenpyroximate	0.1 (n=5)	78 to 103% (Mean 87%)	11.0%	MRM2 422→135
Oilseed rape seed (high oil)	fenpyroximate	Overall (n=10)	56 to 114% (Mean 84%)	20.3%	MRM2 422→135
Dried beans (dry matrix)	fenpyroximate	0.01 (n=5)	85 to 97% (Mean 92%)	5.3%	MRM1 422→366
Dried beans (dry matrix)	fenpyroximate	0.1 (n=5)	91 to 108% (Mean 102%)	6.8%	MRM1 422→366
Dried beans (dry matrix)	fenpyroximate	Overall (n=10)	85 to 108% (Mean 97%)	7.9%	MRM1 422→366
Dried beans (dry matrix)	fenpyroximate	0.01 (n=5)	84 to 100% (Mean 95%)	6.9%	MRM2 422→135
Dried beans (dry matrix)	fenpyroximate	0.1 (n=5)	89 to 106% (Mean 101%)	7.0%	MRM2 422→135
Dried beans (dry matrix)	fenpyroximate	Overall (n=10)	84 to 106% (Mean 98%)	7.2%	MRM2 422→135
Sugar beet roots (high water)	fenpyroximate	0.01 (n=5)	101 to 110% (Mean 104%)	3.8%	MRM1 422→366
Sugar beet roots (high water)	fenpyroximate	0.1 (n=5)	105 to 117% (Mean 109%)	4.8%	MRM1 422→366
Sugar beet roots (high water)	fenpyroximate	Overall (n=10)	101 to 117% (Mean 107%)	4.8%	MRM1 422→366
Sugar beet roots (high water)	fenpyroximate	0.01 (n=5)	97 to 116% (Mean 108%)	7.0%	MRM2 422→135
Sugar beet roots (high water)	fenpyroximate	0.1 (n=5)	102 to 122% (Mean 110%)	6.8%	MRM2 422→135
Sugar beet roots (high water)	fenpyroximate	Overall (n=10)	97 to 122% (Mean 109%)	6.5%	MRM2 422→135

Table 105 Recovery results from independent method validation of M-1 using the analytical method RES/RAM/004 in crops

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Apple (high water)	M-1	0.01 (n=5)	109 to 122% (Mean 117%)	4.3%	MRM1 422→366
Apple (high water)	M-1	0.1 (n=5)	96 to 106% (Mean 100%)	4.4%	MRM1 422→366
Apple (high water)	M-1	Overall (n=10)	96 to 122%	8.9%	MRM1 422→366

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
			(Mean 109%)		
Apple (high water)	M-1	0.01 (n=5)	113 to 123% (Mean 117%)	3.3%	MRM2 422→135
Apple (high water)	M-1	0.1 (n=5)	96 to 106% (Mean 100%)	4.9%	MRM2 422→135
Apple (high water)	M-1	Overall (n=10)	96 to 123% (Mean 109%)	8.9%	MRM2 422→135
Oilseed rape seed (high oil)	M-1	0.01 (n=5)	67 to 95% (Mean 79%)	15.6%	MRM1 422→366
Oilseed rape seed (high oil)	M-1	0.1 (n=5)	89 to 101% (Mean 94%)	5.9%	MRM1 422→366
Oilseed rape seed (high oil)	M-1	Overall (n=10)	67 to 101% (Mean 86%	13.7%	MRM1 422→366
Oilseed rape seed (high oil)	M-1	0.01 (n=5)	48 to 99% (Mean 82%)	25.4%	MRM2 422→135
Oilseed rape seed (high oil)	M-1	0.1 (n=5)	89 to 103% (Mean 94%)	6.5%	MRM2 422→135
Oilseed rape seed (high oil)	M-1	Overall (n=10)	48 to 103% (Mean 88%	18.0%	MRM2 422→135
Dried beans (dry matrix)	M-1	0.01 (n=5)	92 to 109% (Mean 100%)	7.0%	MRM1 422→366
Dried beans (dry matrix)	M-1	0.1 (n=5)	93 to 112% (Mean 105%)	7.1%	MRM1 422→366
Dried beans (dry matrix)	M-1	Overall (n=10)	92 to 112% (Mean 103%)	7.1%	MRM1 422→366
Dried beans (dry matrix)	M-1	0.01 (n=5)	86 to 100% (Mean 94%)	5.8%	MRM2 422→135
Dried beans (dry matrix)	M-1	0.1 (n=5)	91 to 111% (Mean 105%)	7.8%	MRM2 422→135
Dried beans (dry matrix)	M-1	Overall (n=10)	86 to 111% (Mean 9%)	8.9%	MRM2 422→135
Sugar beet roots (high water)	M-1	0.01 (n=5)	100 to 107% (Mean 105%)	2.8%	MRM1 422→366
Sugar beet roots (high water)	M-1	0.1 (n=5)[a]	106 to 117% (Mean 111%)	4.1%	MRM1 422→366
Sugar beet roots (high water)	M-1	Overall (n=10)	100 to 117% (Mean 108%)	4.5%	MRM1 422→366
Sugar beet roots (high water)	M-1	0.01 (n=5)	95 to 104% (Mean 99%)	3.4%	MRM2 422→135
Sugar beet roots (high water)	M-1	0.1 (n=5)[b]	105 to 119% (Mean 112%)	5.2%	MRM2 422→135
Sugar beet roots (high water)	M-1	Overall (n=10)	95 to 119% (Mean 99%)	8.9%	MRM2 422→135

[a] Includes one result from a mean of three injections (110%, 109%, 107%) after rejection via Dixon test of the original recovery at 135% obtained at first injection

[b] Includes one result from a mean of three injections (110%, 109%, 104%) after rejection via Dixon test of the original recovery at 142% obtained at first injection

Table 106 Characteristics for the analytical method used for independent validation of fenpyroximate and M-1 residues in crops

	Fenpyroximate	M-1
Specificity (all crops)	blank value < 30% LOQ Product ion MS scan presented	blank value < 30% LOQ Product ion MS scan presented
Calibration (type, number of data points)	typical calibration data presented| typical calibration line equation presented linear, 1/x weighting applied r=> 0.994 (1 run), generally >0.995 7 data points Matrix matched used	typical calibration data presented| typical calibration line equation presented linear, 1/x weighting applied r=>0.996 7 data points Matrix matched used

	Fenpyroximate	M-1
	Both MRM1 and MRM2 were reported for all crops	Both MRM1 and MRM2 were reported for all crops
Calibration range	0.1 to 10.0 ng/mL for all crops Corresponding calibration range 0.0025 to 0.25 mg/kg fenpyroximate	0.1 to 10.0 ng/mL for all crops Corresponding calibration range 0.0025 to 0.25 mg/kg M-1
Assessment of matrix effects is presented	Yes, not significant but matrix matched standards used for all crops	Yes, not significant but matrix matched standards used for all crops
Limit of determination/quantification	LOQ=0.01 mg/kg in all crops	LOQ=0.01 mg/kg in all crops
Comment	Acceptable against SANCO/825/00 rev 8.1 criteria. Method suitable for monitoring in all matrix types.	Acceptable against SANCO/825/00 rev 8.1 criteria. Method suitable for monitoring in all matrix types.

Table 107 Recovery results from independent method validation of fenpyroximate using the analytical method RES/RAM/004 in hops

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Dried hops	fenpyroximate	0.01 (n=4)	80 to 83% (Mean 81%)	1.8 %	MRM1 422→366 Column 1
Dried hops	fenpyroximate	0.1 (n=5)	79 to 84% (Mean 81%)	2.4%	MRM1 422→366 Column 1
Dried hops	fenpyroximate	Overall (n=9)	79 to 84% (Mean 81%)	2.4%	MRM1 422→366 Column 1
Dried hops	fenpyroximate	0.01 (n=4)	87 to 102% (Mean 93%)	7.9%	MRM2 422→366 Column 2
Dried hops	fenpyroximate	0.1 (n=5)	78 to 87% (Mean 81%)	4.4%	MRM2 422→366 Column 2
Dried hops	fenpyroximate	Overall (n=9)	78 to 102% (Mean 86%)	9.3%	MRM2 422→366 Column 2

Table 108 Recovery results from independent method validation of M-1 using the analytical method RES/RAM/004 in hops

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Dried hops	M-1	0.01 (n=5)	74 to 93% (Mean 83%)	10.5 %	MRM1 422→366 Column 1
Dried hops	M-1	0.1 (n=5)	84 to 89% (Mean 85%)	2.4%	MRM1 422→366 Column 1
Dried hops	M-1	Overall (n=10)	74 to 93% (Mean 84%)	7.3%	MRM1 422→366 Column 1
Dried hops	M-1	0.01 (n=5)	80 to 102% (Mean 87%)	10.2%	MRM2 422→366 Column 2
Dried hops	M-1	0.1 (n=5)	85 to 93% (Mean 88%)	3.5%	MRM2 422→366 Column 2
Dried hops	M-1	Overall (n=10)	80 to 102% (Mean 87%)	7.2%	MRM2 422→366 Column 2

Table 109 Characteristics for the analytical method used for independent validation of fenpyroximate and M-1 residues in hops

	Fenpyroximate	M-1
Specificity	blank value < 30% LOQ Product ion MS scan presented	blank value < 30% LOQ Product ion MS scan presented
Calibration (type, number of data points)	typical calibration data presented| typical calibration line equation presented linear, 1/x weighting applied	typical calibration data presented| typical calibration line equation presented linear, 1/x weighting applied

	Fenpyroximate	M-1
	r=>0.99 7 data points Matrix matched used Both columns data were reported	r=>0.99 7 data points Matrix matched used Both columns data were reported
Calibration range	0.15 to 10.0 ng/mL Corresponding calibration range 0.003 to 0.20 mg/kg fenpyroximate	0.15 to 10.0 ng/mL Corresponding calibration range 0.003 to 0.20 mg/kg M-1
Assessment of matrix effects is presented	Yes, not significant on the C18 column but significant on the Biphenyl column, matrix matched standards used	Yes, significant on both columns, matrix matched standards used
Limit of determination/quantification	LOQ=0.01 mg/kg in all crops	LOQ=0.01 mg/kg in all crops
Comment	Acceptable against SANCO/825/00 rev 8.1 criteria. Method suitable for monitoring in all matrix types.	Acceptable against SANCO/825/00 rev 8.1 criteria. Method suitable for monitoring in all matrix types.

2. Analytical methods for data generation

GC method for fenpyroximate and M-1

GLC method for determination of fenpyroximate and its metabolite in fruit was validated (Anonymous, 1989a, report No. A-4001). Fenpyroximate and its Z-isomer were extracted with methanol. The extract was filtered and concentrated by rotary evaporated at below 40 °C. The evaporation residue was diluted with 10% sodium chloride aqueous solution and then partitioned three times with n-hexane. The n-hexane phase was partitioned three times with acetonitrile. The acetonitrile phase was rotary evaporated to dryness at below 40 °C and then reconstituted in n-hexane: ethyl acetate (9:1) for column chromatography. The extract was cleaned up on a silica gel/alumina column followed by analysis using GC-FTD with a fused silica capillary column. The method was validated for linearity and accuracy with three 0.2 mg/kg fortifications for orange peel and tea, and three 0.1 mg/kg fortifications for orange pulp, apple and strawberry.

Table 110 Summary of results for fenpyroximate and its Z-isomer (M-1)

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Number of replicates
Fenpyroximate			
Orange peel	0.2	93	3
Orange pulp	0.1	92	3
Apple	0.1	105	3
Strawberry	0.1	91	3
Tea	0.2	78	3
Z-Isomer (M-1)			
Orange peel	0.2	87	3
Orange pulp	0.1	81	3
Apple	0.1	88	3
Strawberry	0.1	78	3
Tea	0.2	85	3

Table 111 Characteristics for the analytical method used for fenpyroximate and M-1 residues in crops

	Fenpyroximate	M-1
Calibration range	0.1 to 1.0 µg/mL	0.1 to 1.0 µg/mL
Limit of determination/quantification	LOQ=0.2 mg/kg orange peel and tea, 0.1 mg/kg orange pulp, apple and strawberry	LOQ=0.2 mg/kg orange peel and tea, 0.1 mg/kg orange pulp, apple and strawberry
Comment	Acceptable with mean recoveries between 70-120%	Acceptable with mean recoveries between 70-120%

HPLC method for fenpyroximate and M-1

HPLC method for determination of residues of fenpyroximate and its metabolites in fruit was developed and validated (Anonymous, 1989b, report No. A-4002). Fenpyroximate, its Z-isomer and desmethyl fenpyroximate were extracted with acetonitrile. The extract was filtered and concentrated by rotary evaporated at below 40 °C. The evaporation residue was diluted with 10% sodium chloride aqueous solution and then partitioned three times with n-hexane. The n-hexane phase was partitioned three times with acetonitrile. The acetonitrile phase was rotary evaporated to dryness at below 40 °C and then reconstituted in n-hexane:ethyl acetate (9:1) for column chromatography. The extract was cleaned up on a silica gel/alumina column and then a C_{18} SPE cartridge, followed by analysis using by reverse-phase HPLC-UV with a C_{18} column. The method was validated for linearity and accuracy with three 0.1 mg/kg fortifications for orange pulp and grapes.

Table 112 Summary of results for fenpyroximate, its Z-isomer (M-1) and demethyl fenpyroximate

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Number of replicates
Fenpyroximate			
Orange pulp	0.1	96	3
Grapes	0.1	103	3
Z-Isomer (M-1)			
Orange pulp	0.1	98	3
Grapes	0.1	90	3
Demethyl fenpyroximate			
Orange pulp	0.1	88	3
Grapes	0.1	78	3

Table 113 Characteristics for the analytical method used for fenpyroximate, M-1 and desmethyl fenpyroximate residues in crops

	Fenpyroximate	M-1	Demethyl fenpyroximate
Calibration range	0.05 to 1 µg/mL	0.05 to 1 µg/mL	0.05 to 1 µg/mL
Limit of determination/quantification	LOQ=0.1 mg/kg in orange pulp and grapes	LOQ=0.1 mg/kg in orange pulp and grapes	LOQ=0.1 mg/kg in orange pulp and grapes
Comment	Acceptable giving mean recoveries of between 70-120%.	Acceptable giving mean recoveries of between 70-120%.	Acceptable giving mean recoveries of between 70-120%.

GC/HPLC method for fenpyroximate, M-1and desmethyl fenpyroximate

GLC/HPLC method for determination of residues of fenpyroximate and its metabolites in fruits and vegetables was developed and validated (Anonymous, 1989c, report No. A-4003). Fenpyroximate, its Z-isomer and demethyl fenpyroximate were extracted with acetonitrile. The extract was filtered and concentrated by rotary evaporated at below 40 °C. The extract was cleaned-up on a C_{18} SPE cartridge and then a silica gel/alumina column. For fenpyroximate and its Z isomer, further clean-up was performed on a silica SPE cartridge and, if necessary, a second C_{18} SPE cartridge, followed by analysis using GC-NPD with a fused silica capillary column. For demethyl fenpyroximate, further clean-up was performed on a silica SPE cartridge and, if necessary, a second C_{18} SPE cartridge, followed by analysis using normal-phase HPLC-UV with a silica column. The method was validated for linearity and accuracy with green pepper.

Table 114 Summary of results for fenpyroximate, its Z-isomer and desmethyl fenpyroximate

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Number of replicates
Fenpyroximate			
Green pepper	0.1	109	3
Z-Isomer			
Green pepper	0.1	99	3

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Number of replicates
Demethyl fenpyroximate			
Green pepper	0.1	80	3

Table 115 Characteristics for the analytical method used for fenpyroximate, M-1 and desmethyl fenpyroximate residues in crops

	Fenpyroximate	M-1	Demethyl fenpyroximate
Calibration range	0.05 to 1 µg/mL	0.05 to 1 µg/mL	0.05 to 1 µg/mL
Limit of determination/quantification	LOQ=0.1 mg/kg in peppers	LOQ=0.1 mg/kg in peppers	LOQ=0.1 mg/kg in peppers
Comment	Acceptable giving mean recoveries of between 70-120%.	Acceptable giving mean recoveries of between 70-120%.	Acceptable giving mean recoveries of between 70-120%.

Method DFG S 19 for fenpyroximate and M-1

The method DFG S 19 for determination of residues of fenpyroximate (HOE 094552) and metabolite M-1 (HOE 112573) was validated with vine (grape, cider, wine) (Specht W., 1991a, report No. A-4007), apple (fruit, mash, cider) (Specht W., 1991b, report No. A-4008), orange (pulp and peel) (Specht W., 1992a, report No. A-4010), cucumber (Specht W., 1992b, report No. A-4011), tomato (Weber H., 1992c, report No. A-4012), strawberry (Specht W., 1992d, report No. A-4013), eggplant (Specht W., 1992e, report No. A-4014), pear (Specht W., 1992f, report No. A-4015), paprika (Specht W., 1992g, report No.: A-4016), peach (Specht W., 1992h, report No. A-4017), melon (pulp and peel) (Specht W., 1992i, report No.: A-4018), hop (dregs, yeast, beer) (Specht W., 1992j, report No. A-4019), dwarf bean (leaves) (Weber H., 1993a, report No. A-4020), apple (fruit) (Weber H., 1993b, report No. A-4026), plum (fruit) (Specht W., 1992k, report No. A-4046), and matric with high water content (apple) (Klimmek S. and Klimmek A. 2007, report No. A-4068). Fenpyroximate and its metabolite M-1 were extracted with acetone. Water is added beforehand in an amount that takes full account of the natural water content of the sample so that during extraction the acetone: water ratio remains constant at 2:1 v/v. The extract is saturated with sodium chloride and diluted with dichloromethane. The evaporation residue of the organic phase is cleaned up by GPC using a mixture of cyclohexane and ethyl acetate as eluant. Further clean up on a silica gel column is followed by analysis using GC-N-FID.

Modification to the original method is necessary for the analysis of wine samples. To 100 g wine, 200 mL acetone and 100 mL dichloromethane are added. After mixing 10 mL of a saturated sodium potassium solution and 600 mL water are added and completely mixed. After separation of the organic phase the extraction stop is repeated with 50 mL dichloromethane. The combined organic phases are dried, concentrated and cleaned up as described above.

Modification to the original method is necessary for the analysis of beer samples. Beer is diluted with an aqueous solution of sodium chloride and extracted three times with dichloromethane. The organic phases are combined and cleaned up as described above.

Table 116 Summary of results for fenpyroximate and M-1

Report	Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range (%)	RSD (%)	Number of replicates
Fenpyroximate						
A-4007	Grapes	0.050	116	114-117	NA	2
		0.50	93	84-102	NA	2
		1.98	74	70-77	NA	2
	Cider	0.050	84	81-86	NA	2
		0.50	71	70-71	NA	2

Report	Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range (%)	RSD (%)	Number of replicates
	Wine	0.050	97	87-106	NA	2
		0.50	81	74-88	NA	2
A-4008	Apple (fruit)	0.050	89	88-89	NA	2
		0.50	81	81-81	NA	2
		4.80	85	83-87	NA	2
	Mash	0.050	110	102-118	NA	2
		0.5	91	87-94	NA	2
	Cider	0.050	94	88-99	NA	2
		0.50	82	76-88	NA	2
A-4010	Orange (pulp)	0.05	120	119-121	NA	2
		0.50	114	107-121	NA	2
		5.0	119	118-120	NA	2
	Orange (peel)	0.05	116	114-117	NA	2
		0.48	108	100-116	NA	2
		5.0	108	95-120	NA	2
A-4011	Cucumber	0.01	120	117-122	NA	2
		0.05	116	113-119	NA	2
		0.50	108	104-111	NA	2
		5.0	108	106-109	NA	2
A-4012	Tomato	0.01	114	103-125	NA	2
		0.05	117	113-120	NA	2
		0.50	106	98-114	NA	2
		5.0	91	81-101	NA	2
A-4013	Strawberry	0.01	112	109-115	NA	2
		0.10	99	96-102	NA	2
		1.0	109	107-111	NA	2
A-4014	Egg plant	0.01	111	109-112	NA	2
		0.10	90	90-90	NA	2
		1.0	108	107-109	NA	2
A-4015	Pear	0.01	111	107-115	NA	2
		0.10	117	113-120	NA	2
		1.0	118	114-121	NA	2
A-4016	Paprika	0.01	108	107-109	NA	2
		0.10	117	115-118	NA	2
		1.0	107	95-119	NA	2
A-4017	Peach	0.01	118	116-120	NA	2
		0.10	105	102-107	NA	2
		1.0	104	101-106	NA	2
A-4018	Melon (pulp)	0.01	104	103-104	NA	2
		0.10	104	95-112	NA	2
		1.0	107	104-109	NA	2
	Melon (peel)	0.01	95	93-97	NA	2
		0.10	89	86-92	NA	2
		1.0	111	109-112	NA	2
A-4019	Hops (dregs)	0.10	119	117-120	NA	2
		1.0	112	103-121	NA	2
	Hops (yeast)	0.10	105	95-114	NA	2
		1.0	89	85-93	NA	2
	Hops (beer)	0.01	94	86-102	NA	2
		0.10	89	77-101	NA	2
A-4020	Dwarf beans (leaves)	0.10	117	115-118	NA	2
		1.0	100	92-107	NA	2
		20	99	92-105	NA	2
A-4026	Apple	0.01	100	100-100	NA	2
A-4046	Plum	0.050	85	83-87	NA	2
		0.50	91	88-93	NA	2

Report	Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range (%)	RSD (%)	Number of replicates
		1.98	84	83-84	NA	2
A-4068	Apple	0.005	104	86-126	11.7	10
		0.050	102	93-110	6.7	10
M-1						
A-4007	Grapes	0.049	80	70-90	NA	2
		0.49	88	82-93	NA	2
		1.97	86	84-87	NA	2
	Cider	0.049	89	87-91	NA	2
		0.49	85	83-86	NA	2
	Wine	0.049	93	90-95	NA	2
		0.49	95	91-98	NA	2
A-4008	Apple (fruit)	0.050	91	87-95	NA	2
		0.50	87	84-90	NA	2
		4.75	87	82-91	NA	2
	Mash	0.050	83	76-89	NA	2
		0.50	87	86-87	NA	2
	Cider	0.050	86	83-88	NA	2
		0.50	70	68-72	NA	2
A-4010	Orange (pulp)	0.05	88	74-102	NA	2
		0.48	87	80-93	NA	2
		5.0	91	90-92	NA	2
	Orange (peel)	0.05	84	74-98	NA	2
		0.47	92	89-94	NA	2
		5.0	100	90-109	NA	2
A-4011	Cucumber	0.01	90	87-92	NA	2
		0.05	79	77-80	NA	2
		0.50	78	77-89	NA	2
		5.0	96	93-99	NA	2
A-4012	Tomato	0.01	94	87-101	NA	2
		0.05	86	85-87	NA	2
		0.50	81	75-87	NA	2
		5.0	84	77-91	NA	2
A-4013	Strawberry	0.01	83	78-88	NA	2
		0.10	83	82-84	NA	2
		1.0	84	79-88	NA	2
A-4014	Egg plant	0.01	104	104-104	NA	2
		0.10	75	74-75	NA	2
		1.0	106	106-106	NA	2
A-4015	Pear	0.01	85	82-87	NA	2
		0.10	84	80-88	NA	2
		1.0	98	98-98	NA	2
A-4016	Paprika	0.01	108	106-110	NA	2
		0.10	116	115-116	NA	2
		1.0	100	91-109	NA	2
A-4017	Peach	0.01	107	107-107	NA	2
		0.10	85	84-85	NA	2
		1.0	101	98-103	NA	2
A-4018	Melon (pulp)	0.01	96	95-97	NA	2
		0.10	104	98-109	NA	2
		1.0	95	92-98	NA	2
	Melon (peel)	0.01	91	89-92	NA	2
		0.10	89	88-90	NA	2
		1.0	99	98-99	NA	2
A-4019	Hops (dregs)	0.10	79	76-82	NA	2
		1.0	79	70-87	NA	2
	Hops	0.10	83	80-85	NA	2

Report	Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range (%)	RSD (%)	Number of replicates
	(yeast)	1.0	76	75-76	NA	2
	Hops (beer)	0.01	96	90-102	NA	2
		0.10	94	81-106	NA	2
A-4020	Dwarf beans (leaves)	0.10	97	96-98	NA	2
		1.0	93	82-103	NA	2
		20	89	81-96	NA	2
A-4026	Apple	0.01	99	95-102	NA	2
A-4046	Plum	0.049	86	82-90	NA	2
		0.49	84	83-85	NA	2
		1.97	85	81-88	NA	2
A-4068	Apple	0.005	103	90-118	8.1	10
		0.05	108	101-115	3.8	10

Table 117 Characteristics for the analytical method used for fenpyroximate and M-1 residues in crops

	Fenpyroximate	M-1
Specificity	Control chromatograms show no interferences	Control chromatograms show no interferences
Calibration range	Nominal calibration ranges are Report A-4007: 0.24-2.4 µg/mL Report A-4008, A-4013, A-4014, A-4015, A-4016, A-4017, A-4018, A-4020: 0.12-1.2 µg/mL Report A-4010: 0.07-2.9 µg/mL Report A-4011, A-4012: 0.13-2.9 µg/mL Report A-4019: 0.06-0.68 µg/mL Report A-4026: 0.12 µg/mL Report A-4046: 0.12-0.24 µg/mL Report A4068: 0.5-100 ng/mL, caibration curve presented R≥0.9996, assessment of matrix effects presented	Nominal calibration ranges are Report A-4007: 0.24-2.4 µg/mL Report A-4008, A-4013, A-4014, A-4015, A-4016, A-4017, A-4018, A-4020: 0.12-1.2 µg/mL Report A-4010: 0.07-2.9 µg/mL Report A-4011, A-4012: 0.13-2.9 µg/mL Report A-4019: 0.06-0.55 µg/mL Report A-4026: 0.12 µg/mL Report A-4046: 0.12-0.24 µg/mL Report A4068: 0.5-100 ng/mL, caibration curve presented R≥0.9996, assessment of matrix effects presented
Limit of determination/quantification	LOQ grapes, cider, wine, apple mash, cider, orange pulp and plum= 0.05 mg/kg LOQ in cucumber, tomato. Strawberry, egg plant, pear, paprika, peach, melon pulp, melon peel, hops beer=0.01 mg/kg LOQ in hops dregs, hops yeast and dwarf beans=0.10 mg/kg LOQ in apples 0.005 mg/kg	LOQ grapes, cider, wine, apple mash, cider, orange pulp and plum= 0.05 mg/kg LOQ in cucumber, tomato. Strawberry, egg plant, pear, paprika, peach, melon pulp, melon peel, hops beer =0.01 mg/kg LOQ in hops dregs, hops yeast and dwarf beans=0.10 mg/kg LOQ in apples 0.005 mg/kg
Comment	Acceptable giving mean recoveries of between 70-120%	Acceptable against giving mean recoveries of between 70-120%

The method for determination of residues of fenpyroximate in/on apple raw agricultural commodities was validated in apple for linearity, specificity, precision and accuracy (Rose J.E., 2001, report No.: A-4119). Fenpyroximate and it metabolites M-1 were extracted with acetone. Water is added beforehand in an amount that takes full account of the natural water content of the sample so that during extraction the acetone: water ratio remains constant at 2:1 v/v. The extract is saturated with sodium chloride and diluted with dichloromethane. The evaporation residue of the organic phase is cleaned up by GPC. Further clean up on a silica gel column is followed by analysis using GC-NPD.

Table 118 Summary of results for fenpyroximate and M1

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range(%)	RSD (%)	Number of replicates
Fenpyroximate/M1					
Apple	0.01	116	112-120	3	3

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range(%)	RSD (%)	Number of replicates
	0.1	77	66-83	12	3
	0.5	75	74-77	3	3

Table 119 Characteristics for the analytical method used for fenpyroximate and M-1residues in crops

	Fenpyroximate/M-1
Specificity	Control chromatograms show no significant interferences
Calibration range	0.1-2 µg/mL, r^2 value typically 0.99 or better
Limit of determination/quantification	LOQ 0.01 mg/kg
Comment	Acceptable giving mean recoveries of between 70-120%

The DFG S19 method for the Determination of Fenpyroximate and its Metabolite (M-1) was further validated for linearity, specificity, precision and accuracy by Independent Laboratory in Apple, Grape and Cotton Samples / Matrices (Kretschmer **S.,** 2001 report No. A-4040).

Table 120 Summary of results for fenpyroximate and M-1

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range (%)	RSD (%)	Number of replicates
Apple	0.01	90	72-105	17	5
	0.10	84	78-95	9	5
Grapes	0.05	82	70-94	14	5
	0.50	99	87-107	8	5
Cotton seed	0.01	95	90-100	5	5
	0.10	94	85-103	9	4
Cotton seed oil	0.05	92	84-100	7	5
	0.5	93	85-116	6	5

Table 121 Characteristics for the analytical method used for fenpyroximate and M-1 residues in crops

	Fenpyroximate and M1
Specificity	Control chromatograms show no significant interferences
Calibration range	0.02-2.0 or 5.0 ng/µL depending on matrix with r^2 > 0.99
Limit of determination/quantification	LOQ 0.01 mg/kg for apple and 0.05 mg/kg for grapes, cotton seed and cotton seed oil.
Comment	Acceptable giving mean recoveries of between 70-120%

Another Independent Laboratory Validation of the DFG S19 multi-residue method for the analysis of fenpyroximate and its M-1 metabolite was conducted in pears (Brown D., 2006b, report No.: A-4065) for linearity, specificity, precision and accuracy. Fenpyroximate and it metabolites M-1 were extracted with acetone. Water is added beforehand in an amount that takes full account of the natural water content of the sample so that during extraction the acetone:water ratio remains constant at 2:1 v/v. The extract is saturated with sodium chloride and partitioned with dichloromethane. The evaporation residue of the organic phase is cleaned up by GPC using a mixture of cyclohexane and ethyl acetate as eluant. Further clean up on a silica gel column is followed by analysis using GC-NPD.

Table 122 Summary of results for fenpyroximate and M-1

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range(%)	RSD (%)	Number of replicates
Fenpyroximate					
Pears	0.05	83	76-87	5.6	5

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range(%)	RSD (%)	Number of replicates
	0.5	100	98-101	1.5	5
M-1					
Pears	0.05	100	99-100	0.6	5
	0.5	103	100-108	2.9	5

Table 123 Characteristics for the analytical method used for fenpyroximate residues in crops

	Fenpyroximate	M-1
Specificity	Control chromatograms show no significant interferences	Control chromatograms show no significant interferences
Calibration range	0-5.0 µg/mL, r^2 value typically 0.99 or better	0-5.0 µg/mL, r^2 value typically 0.99 or better
Limit of determination/quantification	LOQ 0.05 mg/kg	LOQ 0.05 mg/kg
Comment	Acceptable giving mean recoveries of between 70-120%	Acceptable giving mean recoveries of between 70-120%

Method P-14.045.02 for fenpyroximate and M-1

Method P-14.045.02 based on Hoechst method AL 015/90-0 for the determination of the residues of fenpyroximate (HOE 094552) and metabolite M-1 (HOE 112573) was validated in tomato (fruit) (Specht W., 1991c, report No. A-4009). Fenpyroximate and it metabolites M-1 were extracted with acetonitrile. The extract is cleaned up by partitioning between n-hexane and water, followed by partitioning between n-hexane and acetonitrile. Further clean up on a silica gel column is followed by analysis using HPLC with UV photometric analysis. The method was validated for linearity, specificity and accuracy in tomatoes. Results are summarised below.

Table 124 Summary of results for fenpyroximate and M-1

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range (%)	RSD (%)	Number of replicates
Fenpyroximate					
Tomato	0.053	62.6	60.9-64.3	NA	2
	0.51	76.5	72.0-81.0	NA	2
	5.10	69.0	67.1-70.9	NA	2
M-1					
Tomato	0.049	74.8	73.3-76.2	NA	2
	0.53	83.8	81.0-86.5	NA	2
	5.32	77.9	74.8-81.0	NA	2

Table 125 Characteristics for the analytical method used for fenpyroximate and M-1 residues in tomatoes

	Fenpyroximate	M-1
Specificity	Control chromatograms show no interferences	Control chromatograms show no interferences
Calibration range	Nominal calibration ranges are 0.5-5 µg/mL	Nominal calibration ranges are 0.5-5 µg/mL
Limit of determination/quantification	LOQ 0.05 mg/kg	LOQ 0.05 mg/kg
Comment	Acceptable giving mean recoveries of between 70-120%	Acceptable against giving mean recoveries of between 70-120%

HPLC-MS method for fenpyroximate and M-1

HPLC-MS method for the determination of fenpyroximate residues in dried hops, grapes and oranges (pulp and peel) was validated (Todd M.A., 1999, report No.: A-4036)**.** Fenpyroximate was extracted

from grapes, orange pulp and orange peel with acetone: water followed by liquid/liquid partition and clean-up by SPE. The method for hops involved extraction into ethyl acetate, a solvent change to methanol, and liquid/liquid partition into 2,2,4-trimethyl pentane prior to further clean-up using a C18 SPE cartridge. For all matrices quantitation was using HPLC-MS. The method was validated for linearity, specificity, precision and accuracy.

Table 126 Summary of results for fenpyroximate (m/z 422)

Matrix	Fortification level (ng/g)	Mean recovery (%)	Recovery range(%)	RSD (%)	Number of replicates
Fenpyroximate					
Dried hops	50	91	82-96	8.4	3
	250	91	87-94	4.0	3
	1000	90	89-91	1.1	3
Grapes	10	81	78-87	6.1	3
	50	86	80-94	8.6	3
	500	102	97-106	4.4	3
Orange pulp	10	92	80-100	11.7	3
	50	98	93-100	4.1	3
	500	92	86-97	6.0	3
Orange peel	50	84	81-88	4.2	3
	250	86	85-86	0.7	3
	1000	90	88-92	2.3	3

Table 127 Characteristics for the analytical method used for fenpyroximate and M-1 residues in tomatoes

	Fenpyroximate
Specificity	Control chromatograms show no significant interferences
Calibration range	1-100 ng/mL with $r^2 > 0.99$
Limit of determination/quantification	LOQ 50 ng/g for orange peel and dried hops and 10 ng/g for grapes and orange pulp
Comment	Acceptable giving mean recoveries of between 70-120%

HPLC-MS/MS method for fenpyroximate and M-1

HPLC-MS/MS method for the determination of fenpyroximate in plants was validated for linearity, specificity, precision and accuracy in apple, grape, orange and cotton (Bacher R., 2005, report No. A-4062). The method was based on the modular multi-residue enforcement method L 00.00-34 of the Official Collection of test methods (DFG S19) with some modifications. Extraction module C1 was used for two plant materials (apple, grape). The extraction module E 3 was applied to acidic material (whole orange). Adjustment of total water content (100 g), pH adjustment using sodium hydrogen carbonate (for oranges only), subsequent extraction with water/acetone (1/2 v/v); partition into organic phase by addition of sodium chloride and ethyl acetate/cyclohexane (1/1 v/v). Extraction module E 7 for cotton seed (oily/fatty material: Extraction with acetonitrile/acetone (225:25 v/v), addition of synthetic calcium silicate (Calflo E). Clean-up using gel permeation chromatography module GPC (for cotton seeds only), LC/MS/MS determination.

Table 128 Summary of results for fenpyroximate m/z 422→366

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range(%)	RSD (%)	Number of replicates
Apple	0.025	98	96-101	2	5
	0.25	99	90-108	6	5
Grapes	0.025	102	95-108	5	5
	0.25	99	93-107	5	5
Cotton seed	0.025	93	86-101	8	5

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range(%)	RSD (%)	Number of replicates
	0.25	92	89-101	6	5
Whole orange	0.025	91	86-97	6	5
	0.25	91	86-92	4	5

Table 129 Summary of results for fenpyroximate m/z 422→135

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range(%)	RSD (%)	Number of replicates
Apple	0.025	97	91-101	4	5
	0.25	99	88-112	9	5
Grapes	0.025	102	94-106	5	5
	0.25	100	93-110	7	5
Cotton seed	0.025	95	88-102	7	5
	0.25	95	90-104	6	5
Whole orange	0.025	93	86-101	7	5
	0.25	92	86-98	5	5

Table 130 Characteristics for the analytical method used for fenpyroximate residues in crops

	Fenpyroximate
Specificity	Control chromatograms show no significant interferences
Calibration range	0.20-20.0 ng/mL with $r^2 > 0.99$ for both transitions
Limit of determination/quantification	LOQ 0.0025 mg/kg
Comment	Acceptable giving mean recoveries of between 70-120%

Another LC-MS/MS analytical method for determination of residues of fenpyroximate and its M-1 metabolite was validated for linearity, specificity, precision and accuracy in apples, strawberries, peaches, pears, plums, beans, cucumbers, peppers and tomatoes (Brown D., 2006a, report No. A-4064). Fenpyroximate and it metabolite M-1 were extracted by maceration with ethyl acetate. Following centrifugation of the mixture, an aliquot was taken together with a drop of octanol and evaporated to dryness. The residue was then re-dissolved in methanol and analysed using LC-MS/MS.

Table 131 Summary of results for fenpyroximate and M-1

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range (%)	RSD (%)	Number of replicates
Fenpyroximate					
Apple	0.01	93	91-96	2.1	5
	0.1	92	83-97	5.9	5
Strawberry	0.01	94	89-97	3.4	5
	0.1	93	91-96	2.1	5
Peaches	0.01	103	100-104	2.2	3
	0.1	97	93-101	4.1	3
Pear	0.01	95	92-98	3.2	3
	0.1	93	90-96	3.2	3
Plum	0.01	103	100-106	3.0	3
	0.1	97	97-98	0.6	3
Bean	0.01	84	83-85	1.4	3
	0.1	85	84-87	1.8	3
Cucumber	0.01	93	92-94	1.2	3
	0.1	90	89-91	1.3	3
Pepper	0.01	88	86-91	3.0	3
	0.1	91	88-93	3.2	3
Tomato	0.01	93	87-99	6.5	3

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range (%)	RSD (%)	Number of replicates
	0.1	92	90-95	3.1	3
M-1					
Apple	0.01	96	94-100	2.4	5
	0.1	94	88-98	4.2	5
Strawberry	0.01	98	96-100	1.9	5
	0.1	90	88-93	2.6	5
Peaches	0.01	89	88-90	1.1	3
	0.1	82	77-85	5.1	3
Pear	0.01	94	92-95	1.6	3
	0.1	90	86-94	4.4	3
Plum	0.01	92	89-97	4.9	3
	0.1	85	84-86	1.2	3
Bean	0.01	86	82-89	4.1	3
	0.1	86	84-88	2.3	3
Cucumber	0.01	90	90-91	0.6	3
	0.1	88	87-88	0.7	3
Pepper	0.01	84	83-85	1.4	3
	0.1	88	84-90	3.7	3
Tomato	0.01	90	87-94	4.2	3
	0.1	86	85-87	1.2	3

Table 132 Characteristics for the analytical method used for fenpyroximate residues in crops

	Fenpyroximate	M-1
Specificity	Control chromatograms show no significant interferences	Control chromatograms show no significant interferences
Calibration range	0.01 to 100 µg/mL, r^2 value typically 0.99 or better	0.01 to 100 µg/mL, r^2 value typically 0.99 or better
Limit of determination/quantification	LOQ 0.01 mg/kg	LOQ 0.01 mg/kg
Comment	Acceptable giving mean recoveries of between 70-120%	Acceptable giving mean recoveries of between 70-120%

The Independent laboratory validation of methodology for the determination of residues of fenpyroximate and its metabolite M1 was conducted for linearity, specificity, precision and accuracy in apples and tomatoes (Todd M.A., 2003, report No.: A-4050). A sub-sample (20 g) of matrix is weighed into a polyethylene bottle. Control samples are fortified at this stage, if required. Samples are extracted with ethyl acetate (50 to 100 mL) and anhydrous sodium sulphate (20 g) by homogenisation for 2 minutes. All samples are then centrifuged for 2 minutes to separate phases. An aliquot (5 mL) of the supernatant is transferred to a clean glass scintillation vial with 1 drop of octanol and taken to dryness in a water bath at approximately 40 °C under a stream of air or nitrogen. The residue is re-constituted in methanol (1 mL) with the aid of sonication and vortex mixing. Quantification is performed by LC-MS/MS.

Table 133: Summary of results for fenpyroximate

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range(%)	RSD (%)	Number of replicates
Fenpyroximate					
Apples	0.01	96	94-99	2.3	5
	0.1	95	93-98	2.4	5
	1.0	96	92-99	3.5	5
Tomatoes	0.01	95	91-98	3.0	5
	0.1	98	96-101	1.8	5
	1.0	101	97-105	3.2	5

Table 134 Summary of results for M-1

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range(%)	RSD (%)	Number of replicates
M-1					
Apples	0.01	91	80-98	7.3	5
	0.1	92	89-94	2.1	5
	1.0	95	90-100	3.9	5
Tomatoes	0.01	95	92-97	2.2	5
	0.1	94	93-96	1.5	5
	1.0	94	90-97	2.9	5

Table 135 Characteristics for the analytical method used for fenpyroximate and M-1 residues in crops

	Fenpyroximate	M-1
Specificity	Control chromatograms show no significant interferences	Control chromatograms show no significant interferences
Calibration range	0-0.5 µg/mL with $r^2 > 0.99$	0-0.5 µg/mL with $r^2 > 0.99$
Limit of determination/quantification	LOQ 0.01 mg/kg	LOQ 0.01 mg/kg
Comment	Acceptable giving mean recoveries of between 70-120%	Acceptable giving mean recoveries of between 70-120%

HPLC-MS/MS method for determination of residues of fenpyroximate in sugar beet was validated for linearity, specificity, precision and accuracy (Ihara T., 2013, report No. A-4081). Specimens obtained were combined with 50 mL acetone/methanol (1/1, v/v) and vigorously shaken for 10 min and then supernatant was decanted. Remained precipitate was then extracted again with 20 mL acetone/methanol (1/1 v/v). Combined supernatant was filtrated under suctioning. Using the glass wool filter paper, the extract was filtrated and obtained filter cake was washed with acetone/methanol (1/1, v/v). Combined filtrates were filled up to 100 mL with acetone/methanol (1/1, v/v). An aliquot (10 mL, 2 g sample equivalent) of extract was evaporated after adding 2 mL of distilled water. The aqueous residue was loaded to a SPE cartridge column (Bond Elut C18(500 mg/3 mL)), which was conditioned by 3 mL of methanol and distilled water. The inner side of the flask was rinsed with 3+2 mL of methanol/distilled water (7/3, v/v). The column was applied with this rinsate and eluted with 2.5 mL of acetonitrile (twice). The eluate was evaporated after addition of 2 mL of distilled water. The residue was filled up to 10 mL with acetonitrile /distilled water (1/1, v/v). The samples were analysed with LC-MS/MS.

Table 136 Summary of results for fenpyroximate and M-1

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range(%)	RSD (%)	Number of replicates
Fenpyroximate m/z 422.2→366.20					
Sugar beet leaf	0.01	81.2	77.00-83.24	3.06	5
	0.1	89.8	87.09-92.78	2.29	5
Sugar beet root	0.01	89.0	87.37-92.30	2.19	
	0.1	96.7	94.55-98.17	1.49	
Fenpyroximate m/z 422.2→135.10					
Sugar beet leaf	0.01	73.4	70.73-79.22	4.55	5
	0.1	81.6	79.59-85.15	2.67	5
Sugar beet root	0.01	78.8	70.60-90.57	9.23	5
	0.1	94.6	93.39-96.56	1.53	5
M-1 m/z 422.2→366.20					
Sugar beet leaf	0.01	87.0	83.55-88.86	2.79	5
	0.1	90.5	86.52-93.27	2.82	5
Sugar beet root	0.01	88.5	87.01-91.25	2.16	5
	0.1	103.4	99.93-106.33	2.30	5

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range(%)	RSD (%)	Number of replicates
M-1 m/z 422.2→135.10					
Sugar beet leaf	0.01	89.6	83.48-96.26	5.10	5
	0.1	89.3	85.57-93.30	3.43	5
Sugar beet root	0.01	88.0	81.01-97.98	8.52	5
	0.1	98.5	97.43-99.55	0.80	5

Table 137 Characteristics for the analytical method used for fenpyroximate and M-1 residues in crops

	Fenpyroximate	M-1
Specificity	Control chromatograms show no significant interferences	Control chromatograms show no significant interferences
Calibration range	0.30-30 ng/mL, r^2 value typically 0.95 or better	0.30-30 ng/mL, r^2 value typically 0.95 or better
Limit of determination/quantification	LOQ 0.01 mg/kg	LOQ 0.01 mg/kg
Comment	Acceptable giving mean recoveries of between 70-120%	Acceptable giving mean recoveries of between 70-120%

The HPLC-MS/MS method for Determination of fenpyroximate residue on coffee beans was validated for linearity, specificity, precision and accuracy (Matos D., 2015, report No.: A-4084). Homogenise the sample with methanol in vortex and centrifuge it. Transfer an aliquot to a vial, filtering it. Analyse using LC-MS/MS.

Table 138: Summary of results for fenpyroximate

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range (%)	RSD (%)	Number of replicates
Fenpyroximate					
Coffee beans	0.01	107	94-115	6	6
	0.1	99	95-107	4	6

Table 139 Characteristics for the analytical method used for fenpyroximate and M-1residues in crops

	Fenpyroximate	M-1
Specificity	Control chromatograms show no significant interferences	Control chromatograms show no significant interferences
Calibration range	0.5-12.5 ng/mL, r^2 value typically 0.99 or better	0.5-12.5 ng/mL, r^2 value typically 0.99 or better
Limit of determination/quantification	LOQ 0.01 mg/kg	LOQ 0.01 mg/kg
Comment	Acceptable giving mean recoveries of between 70-120%	Acceptable giving mean recoveries of between 70-120%

RES/RAM/005 method (QuEChERS, EN 15662:2008) for fenpyroximate, M-1 and M-3

QuEChERS (EN 15662:2008) method with LC-MS/MS (RES/RAM/005) for the determination of residues of fenpyroximate, M-1 and M-3 in processing matrices were validated in for linearity, specificity precision and accuracy (Watson G., 2016b, report No. A-4094).

Residues of fenpyroximate, M-1 or M-3 in high moisture and high acid matrices (10 g sample weight) are determined by an initial direct extraction with acetonitrile (10 mL acetonitrile, 1 minute vortex extraction). Extracts were centrifuged for 5 minutes and the supernatant transferred to a glass vial. The remaining solids are extracted with 1% formic acid in acetonitrile and 1% formic acid in water (5 mL of each, 1 minute vortex extraction) and the two extracts are combined. [Note: for liquid matrices such as fruit juices, wine and beer the extractions steps are omitted]. Addition of magnesium

sulphate, sodium chloride and sodium citrate allows for liquid-liquid phase partition followed by subsequent centrifugation. Samples are transferred to a centrifuge tube and stored a freezer (minimum 2 hours). Extracts were removed from frozen storage and immediately centrifuged at 3000 rpm for 1 minute. Purification of an aliquot of the acetonitrile extract by dispersive SPE with primary/secondary amine (PSA) is prior to dilution and then determination (extract concentration 0.04 g/mL).

Residues of fenpyroximate, M-1 or M-3 in low moisture matrices (5 g sample weight) are determined after addition of water by an initial direct extraction with acetonitrile (water to adjust total to 10 mL and 10 mL acetonitrile, 1 minute vortex extraction). Extracts were centrifuged for 5 minutes and the supernatant transferred to a glass vial. The remaining solids are extracted with 1% formic acid in acetonitrile and 1% formic acid in water (5 mL of each, 1 minute vortex extraction) and the two extracts are combined. Addition of magnesium sulphate, sodium chloride and sodium citrate allows for liquid-liquid phase partition followed by subsequent centrifugation. Samples are transferred to a centrifuge tube and stored a freezer (minimum 2 hours). Extracts were removed from frozen storage and immediately centrifuged at 3000 rpm for 1 minute. Purification of an aliquot of the acetonitrile extract by dispersive SPE with primary/secondary amine (PSA) is prior to dilution and then determination (extract concentration 0.04 g/mL).

Residues of fenpyroximate, M-1 or M-3 in difficult matrices such as apple dry pomace (1 g sample weight) are determined after addition of water by an initial direct extraction with acetonitrile (water to adjust total to 10 mL and 10 mL acetonitrile, 1 minute vortex extraction). Extracts were centrifuged for 5 minutes and the supernatant transferred to a glass vial. The remaining solids are extracted with 1% formic acid in acetonitrile and 1% formic acid in water (5 mL of each, 1 minute vortex extraction) and the two extracts are combined. Addition of magnesium sulphate, sodium chloride and sodium citrate allows for liquid-liquid phase partition followed by subsequent centrifugation. Samples are transferred to a centrifuge tube and stored a freezer (minimum 2 hours). Extracts were removed from frozen storage and immediately centrifuged at 3000 rpm for 1 minute. Purification of an aliquot of the acetonitrile extract by dispersive SPE with primary/secondary amine (PSA) is prior to dilution and then determination (extract concentration 0.04 g/mL).

Residues of fenpyroximate, M-1 or M-3 in difficult matrices such as hops (2 g sample weight) are determined after addition of water by an initial direct extraction with acetonitrile (water to adjust total to 20 mL and 20 mL acetonitrile, 1 minute manual shaking). Extracts were centrifuged for 5 minutes and the supernatant transferred to a glass vial. The remaining solids are extracted with 1% formic acid in acetonitrile and 1% formic acid in water (10 mL of each, 1 minute manual shaking) and the two extracts are combined. Extracts were centrifuged for 5 minutes and an aliquot (30 mL) transferred to a tube with hexane (15 mL). The remaining extract in the tube is retained for determination of fenpyroximate and M-1. After manual shaking the hexane layer is removed and discarded. For both sets of extracts, addition of magnesium sulphate, sodium chloride and sodium citrate allows for liquid-liquid phase partition followed by subsequent centrifugation. Samples are transferred to a centrifuge tube and stored a freezer (minimum 2 hours). Extracts were removed from frozen storage and immediately centrifuged at 3000 rpm for 1 minute. Purification of an aliquot of the acetonitrile extracts by dispersive SPE with primary/secondary amine (PSA) is prior to dilution and then determination (extract concentration 0.04 g/mL). The extracts without the hexane partition purification step were quantified for fenpyroximate and M1. The extracts with the hexane partition purification step were quantified for M3.

All samples were measured using LC-MS/MS employing an Ascentis Express C18 column, 2.7 µm particle size or a Develosil, RP Aqueous column 3 µm particle size. Analysis employed monitoring at two MRM transitions for all analytes (all matrices except hops). For hops a second column was used for confirmation, Phenomenex, Kinetex Biphenyl column, 2.6 µm particle size. Quantification of extracts was performed using external calibration standards over the range 0.1 to 10.0 ng/mL or 0.1 to 5.0 ng/mL. For all processed crop commodities, matrix matched standards were used.

The lowest level tested (LOQ) was 0.01 mg/kg fenpyroximate, M-1 or M-3 in all processed crop matrices.

Table 140 Recovery results from method validation of fenpyroximate using the analytical method RES/RAM/005 in processed crop commodities

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Apple juice	fenpyroximate	0.01 (n=5)	106 to 112% (Mean 108%)	2.3%	MRM1 422→366
Apple juice	fenpyroximate	0.1 (n=5)	102 to 106% (Mean 105%)	1.8%	MRM1 422→366
Apple juice	fenpyroximate	Overall (n=10)	102 to 112% (Mean 106%)	2.7%	MRM1 422→366
Apple juice	fenpyroximate	0.01 (n=5)	106 to 113% (Mean 110%)	2.5%	MRM2 422→135
Apple juice	fenpyroximate	0.1 (n=5)	105 to 108% (Mean 106%)	1.1%	MRM2 422→135
Apple juice	fenpyroximate	Overall (n=10)	105 to 113% (Mean 108%)	2.4%	MRM2 422→135
Apple dry pomace	fenpyroximate	0.01 (n=5)	87 to 105% (Mean 93%)	7.7%	MRM1 422→366
Apple dry pomace	fenpyroximate	0.1 (n=5)	93 to 100% (Mean 96%)	3.0%	MRM1 422→366
Apple dry pomace	fenpyroximate	Overall (n=10)	87 to 105% (Mean 95%	5.6%	MRM1 422→366
Apple dry pomace	fenpyroximate	0.01 (n=5)	94 to 101% (Mean 97%)	2.8%	MRM2 422→135
Apple dry pomace	fenpyroximate	0.1 (n=5)	96 to 98% (Mean 97%)	0.6%	MRM2 422→135
Apple dry pomace	fenpyroximate	Overall (n=10)	94 to 101% (Mean 97%)	1.9%	MRM2 422→135
Apple sauce	fenpyroximate	0.01 (n=5)	101 to 110% (Mean 105%)	3.2%	MRM1 422→366
Apple sauce	fenpyroximate	0.1 (n=5)	102 to 107% (Mean 105%)	2.1%	MRM1 422→366
Apple sauce	fenpyroximate	Overall (n=10)	101 to 110% (Mean 105%)	2.6%	MRM1 422→366
Apple sauce	fenpyroximate	0.01 (n=5)	104 to 109% (Mean 106%)	1.7%	MRM2 422→135
Apple sauce	fenpyroximate	0.1 (n=5)	105 to 107% (Mean 106%)	0.9%	MRM2 422→135
Apple sauce	fenpyroximate	Overall (n=10)	104 to 109% (Mean 106%)	1.2%	MRM2 422→135
Dried apples	fenpyroximate	0.01 (n=5)	101 to 107% (Mean 102%)	2.5%	MRM1 422→366
Dried apples	fenpyroximate	0.1 (n=5)	98 to 103% (Mean 100%)	2.1%	MRM1 422→366
Dried apples	fenpyroximate	Overall (n=10)	98 to 107% (Mean 101%)	2.5%	MRM1 422→366
Dried apples	fenpyroximate	0.01 (n=5)	77 to 95% (Mean 89%)	7.5%	MRM2 422→135
Dried apples	fenpyroximate	0.1 (n=5)	100 to 107% (Mean 103%)	2.8%	MRM2 422→135
Dried apples	fenpyroximate	Overall (n=10)	77 to 107% (Mean 96%)	9.1%	MRM2 422→135
Raisins	fenpyroximate	0.01 (n=5)	97 to 100% (Mean 98%)	1.0%	MRM1 422→366
Raisins	fenpyroximate	0.1 (n=5)	96 to 103% (Mean 100%)	2.4%	MRM1 422→366
Raisins	fenpyroximate	Overall (n=10)	96 to 103% (Mean 99%)	1.9%	MRM1 422→366
Raisins	fenpyroximate	0.01 (n=5)	97 to 101% (Mean 100%)	3.4%	MRM2 422→135
Raisins	fenpyroximate	0.1 (n=5)	95 to 103% (Mean 100%)	3.1%	MRM2 422→135
Raisins	fenpyroximate	Overall (n=10)	95 to 103% (Mean 100%)	3.1%	MRM2 422→135

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Grape juice	fenpyroximate	0.01 (n=5)	108 to 113% (Mean 110%)	1.6%	MRM1 422→366
Grape juice	fenpyroximate	0.1 (n=5)	102 to 106% (Mean 104%)	1.9%	MRM1 422→366
Grape juice	fenpyroximate	Overall (n=10)	102 to 113% (Mean 107%)	3.3%	MRM1 422→366
Grape juice	fenpyroximate	0.01 (n=5)	105 to 110% (Mean 107%)	1.7%	MRM2 422→135
Grape juice	fenpyroximate	0.1 (n=5)	101 to 109% (Mean 106%)	2.9%	MRM2 422→135
Grape juice	fenpyroximate	Overall (n=10)	101 to 110% (Mean 107%)	2.3%	MRM2 422→135
Wine	fenpyroximate	0.01 (n=5)	97 to 101% (Mean 99%)	1.5%	MRM1 422→366
Wine	fenpyroximate	0.1 (n=5)	93 to 99% (Mean 97%)	2.5%	MRM1 422→366
Wine	fenpyroximate	Overall (n=10)	93 to 101% (Mean 98%)	2.3%	MRM1 422→366
Wine	fenpyroximate	0.01 (n=5)	100 to 105% (Mean 101%)	2.2%	MRM2 422→135
Wine	fenpyroximate	0.1 (n=5)	95 to 100% (Mean 97%)	2.1%	MRM2 422→135
Wine	fenpyroximate	Overall (n=10)	95 to 105% (Mean 99%)	2.8%	MRM2 422→135
Tomato juice	fenpyroximate	0.01 (n=5)	94 to 100% (Mean 96%)	2.8%	MRM1 422→366
Tomato juice	fenpyroximate	0.1 (n=5)	88 to 98% (Mean 93%)	3.7%	MRM1 422→366
Tomato juice	fenpyroximate	Overall (n=10)	88 to 100% (Mean 95%)	3.4%	MRM1 422→366
Tomato juice	fenpyroximate	0.01 (n=5)	96 to 105% (Mean 100%)	3.6%	MRM2 422→135
Tomato juice	fenpyroximate	0.1 (n=5)	91 to 97% (Mean 95%)	2.2%	MRM2 422→135
Tomato juice	fenpyroximate	Overall (n=10)	91 to 105% (Mean 97%)	3.9%	MRM2 422→135
Canned tomatoes	fenpyroximate	0.01 (n=5)	99 to 107% (Mean 103%)	3.0%	MRM1 422→366
Canned tomatoes	fenpyroximate	0.1 (n=5)	100 to 105% (Mean 102%)	1.9%	MRM1 422→366
Canned tomatoes	fenpyroximate	Overall (n=10)	99 to 107% (Mean 103%)	2.4%	MRM1 422→366
Canned tomatoes	fenpyroximate	0.01 (n=5)	100 to 115% (Mean 108%)	5.6%	MRM2 422→135
Canned tomatoes	fenpyroximate	0.1 (n=5)	100 to 106% (Mean 103%)	2.4%	MRM2 422→135
Canned tomatoes	fenpyroximate	Overall (n=10)	100 to 115% (Mean 105%)	4.9%	MRM2 422→135
Tomato puree	fenpyroximate	0.01 (n=5)	108 to 111% (Mean 110%)	1.5%	MRM1 422→366
Tomato puree	fenpyroximate	0.1 (n=5)	102 to 108% (Mean 106%)	2.1%	MRM1 422→366
Tomato puree	fenpyroximate	Overall (n=10)	102 to 111% (Mean 108%)	2.4%	MRM1 422→366
Tomato puree	fenpyroximate	0.01 (n=5)	103 to 113% (Mean 106%)	4.0%	MRM2 422→135
Tomato puree	fenpyroximate	0.1 (n=5)	102 to 111% (Mean 107%)	3.7%	MRM2 422→135
Tomato puree	fenpyroximate	Overall (n=10)	102 to 113% (Mean 106%)	3.7%	MRM2 422→135
Canned strawberry	fenpyroximate	0.01 (n=5)	101 to 107% (Mean 104%)	2.1%	MRM1 422→366
Canned strawberry	fenpyroximate	0.1 (n=5)	96 to 106%	3.8%	MRM1 422→366

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
			(Mean 101%)		
Canned strawberry	fenpyroximate	Overall (n=10)	96 to 107% (Mean 103%)	3.5%	MRM1 422→366
Canned strawberry	fenpyroximate	0.01 (n=5)	94 to 110% (Mean 105%)	6.4%	MRM2 422→135
Canned strawberry	fenpyroximate	0.1 (n=5)	99 to 106% (Mean 102%)	3.1%	MRM2 422→135
Canned strawberry	fenpyroximate	Overall (n=10)	94 to 110% (Mean 103%)	5.0%	MRM2 422→135
Strawberry jam	fenpyroximate	0.01 (n=5)	96 to 102% (Mean 99%)	2.2%	MRM1 422→366
Strawberry jam	fenpyroximate	0.1 (n=5)	93 to 99% (Mean 95%)	2.5%	MRM1 422→366
Strawberry jam	fenpyroximate	Overall (n=10)	93 to 102% (Mean 97%)	3.2%	MRM1 422→366
Strawberry jam	fenpyroximate	0.01 (n=5)	95 to 105% (Mean 100%)	3.7%	MRM2 422→135
Strawberry jam	fenpyroximate	0.1 (n=5)	92 to 97% (Mean 94%)	2.6%	MRM2 422→135
Strawberry jam	fenpyroximate	Overall (n=10)	92 to 105% (Mean 97%)	4.3%	MRM2 422→135
Cooked beans	fenpyroximate	0.01 (n=5)	98 to 104% (Mean 101%)	2.5%	MRM1 422→366
Cooked beans	fenpyroximate	0.1 (n=5)	94 to 101% (Mean 97%)	3.2%	MRM1 422→366
Cooked beans	fenpyroximate	Overall (n=10)	94 to 104% (Mean 99%)	3.5%	MRM1 422→366
Cooked beans	fenpyroximate	0.01 (n=5)	104 to 106% (Mean 105%)	0.8%	MRM2 422→135
Cooked beans	fenpyroximate	0.1 (n=5)	91 to 100% (Mean 96%)	3.5%	MRM2 422→135
Cooked beans	fenpyroximate	Overall (n=10)	91 to 106% (Mean 101%)	5.3%	MRM2 422→135
Canned beans	fenpyroximate	0.01 (n=5)	93 to 103% (Mean 98%)	4.1%	MRM1 422→366
Canned beans	fenpyroximate	0.1 (n=5)	91 to 98% (Mean 96%)	3.0%	MRM1 422→366
Canned beans	fenpyroximate	Overall (n=10)	91 to 103% (Mean 97%)	3.5%	MRM1 422→366
Canned beans	fenpyroximate	0.01 (n=5)	97 to 106% (Mean 100%)	3.8%	MRM2 422→135
Canned beans	fenpyroximate	0.1 (n=5)	92 to 102% (Mean 98%)	3.9%	MRM2 422→135
Canned beans	fenpyroximate	Overall (n=10)	92 to 106% (Mean 99%)	3.8%	MRM2 422→135
Malt	fenpyroximate	0.01 (n=5)	94 to 97% (Mean 95%)	1.3%	MRM1 422→366
Malt	fenpyroximate	0.1 (n=5)	85 to 96% (Mean 92%)	4.9%	MRM1 422→366
Malt	fenpyroximate	Overall (n=10)	85 to 97% (Mean 93%)	3.7%	MRM1 422→366
Malt	fenpyroximate	0.01 (n=5)	91 to 97% (Mean 94%)	2.7%	MRM2 422→135
Malt	fenpyroximate	0.1 (n=5)	85 to 93% (Mean 91%)	3.7%	MRM2 422→135
Malt	fenpyroximate	Overall (n=10)	85 to 97% (Mean 93%)	3.4%	MRM2 422→135
Cleaned hops	fenpyroximate	0.01 (n=5)	99 to 114% (Mean 109%)	5.8%	MRM 422→366 Column 1
Cleaned hops	fenpyroximate	0.1 (n=5)	91 to 107% (Mean 99%)	6.1%	MRM 422→366 Column 1
Cleaned hops	fenpyroximate	Overall (n=10)	91 to 114% (Mean 104%)	7.7%	MRM 422→366 Column 1

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Cleaned hops	fenpyroximate	0.01 (n=5)	94 to 113% (Mean 106%)	7.1%	MRM 422→366 Column 2
Cleaned hops	fenpyroximate	0.1 (n=5)	97 to 108% (Mean 101%)	4.3%	MRM 422→366 Column 2
Cleaned hops	fenpyroximate	Overall (n=10)	94 to 113% (Mean 103%)	6.1%	MRM 422→366 Column 2
Beer	fenpyroximate	0.01 (n=5)	95 to 104% (Mean 100%)	3.3%	MRM1 422→366
Beer	fenpyroximate	0.1 (n=5)	97 to 101% (Mean 99%)	1.5%	MRM1 422→366
Beer	fenpyroximate	Overall (n=10)	95 to 104% (Mean 99%)	2.5%	MRM1 422→366
Beer	fenpyroximate	0.01 (n=5)	101 to 105% (Mean 103%)	1.5%	MRM2 422→135
Beer	fenpyroximate	0.1 (n=5)	102 to 104% (Mean 103%)	0.7%	MRM2 422→135
Beer	fenpyroximate	Overall (n=10)	101 to 105% (Mean 103%)	1.1%	MRM2 422→135

Table 141 Recovery results from method validation of M-1 using the analytical method RES/RAM/005 in processed crop commodities

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Apple juice	M-1	0.01 (n=5)	101 to 104% (Mean 103%)	1.2%	MRM1 422→366
Apple juice	M-1	0.1 (n=5)	99 to 103% (Mean 101%)	1.6%	MRM1 422→366
Apple juice	M-1	Overall (n=10)	99 to 104% (Mean 102%)	1.5%	MRM1 422→366
Apple juice	M-1	0.01 (n=5)	99 to 107% (Mean 104%)	3.1%	MRM2 422→135
Apple juice	M-1	0.1 (n=5)	101 to 103% (Mean 101%)	0.9%	MRM2 422→135
Apple juice	M-1	Overall (n=10)	99 to 107% (Mean 103%)	2.7%	MRM2 422→135
Apple dry pomace	M-1	0.01 (n=5)	94 to 109% (Mean 100%)	6.0%	MRM1 422→366
Apple dry pomace	M-1	0.1 (n=5)	99 to 103% (Mean 101%)	1.9%	MRM1 422→366
Apple dry pomace	M-1	Overall (n=10)	94 to 109% (Mean 100%	4.2%	MRM1 422→366
Apple dry pomace	M-1	0.01 (n=5)	95 to 113% (Mean 103%)	7.9%	MRM2 422→135
Apple dry pomace	M-1	0.1 (n=5)	101 to 104% (Mean 102%)	1.6%	MRM2 422→135
Apple dry pomace	M-1	Overall (n=10)	95 to 113% (Mean 103%)	5.4%	MRM2 422→135
Apple sauce	M-1	0.01 (n=5)	103 to 110% (Mean 106%)	3.0%	MRM1 422→366
Apple sauce	M-1	0.1 (n=5)	103 to 107% (Mean 106%)	1.4%	MRM1 422→366
Apple sauce	M-1	Overall (n=10)	103 to 110% (Mean 106%)	2.6%	MRM1 422→366
Apple sauce	M-1	0.01 (n=5)	107 to 109% (Mean 108%)	0.8%	MRM2 422→135
Apple sauce	M-1	0.1 (n=5)	102 to 105% (Mean 103%)	1.1%	MRM2 422→135
Apple sauce	M-1	Overall (n=10)	102 to 109% (Mean 106%)	2.6%	MRM2 422→135
Dried apples	M-1	0.01 (n=5)	97 to 106% (Mean 101%)	3.2%	MRM1 422→366

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Dried apples	M-1	0.1 (n=5)	101 to 104% (Mean 102%)	1.5%	MRM1 422→366
Dried apples	M-1	Overall (n=10)	97 to 106% (Mean 102%)	2.5%	MRM1 422→366
Dried apples	M-1	0.01 (n=5)	86 to 104% (Mean 96%)	7.7%	MRM2 422→135
Dried apples	M-1	0.1 (n=5)	98 to 105% (Mean 101%)	2.5%	MRM2 422→135
Dried apples	M-1	Overall (n=10)	86 to 104% (Mean 98%)	6.0%	MRM2 422→135
Raisins	M-1	0.01 (n=5)	92 to 101% (Mean 98%)	4.1%	MRM1 422→366
Raisins	M-1	0.1 (n=5)	97 to 103% (Mean 100%)	2.3%	MRM1 422→366
Raisins	M-1	Overall (n=10)	92 to 103% (Mean 99%)	3.5%	MRM1 422→366
Raisins	M-1	0.01 (n=5)	95 to 102% (Mean 98%)	3.2%	MRM2 422→135
Raisins	M-1	0.1 (n=5)	96 to 102% (Mean 100%)	2.1%	MRM2 422→135
Raisins	M-1	Overall (n=10)	95 to 102% (Mean 99%)	2.6%	MRM2 422→135
Grape juice	M-1	0.01 (n=5)	107 to 112% (Mean 109%)	1.9%	MRM1 422→366
Grape juice	M-1	0.1 (n=5)	103 to 107% (Mean 105%)	1.5%	MRM1 422→366
Grape juice	M-1	Overall (n=10)	103 to 112% (Mean 107%)	2.6%	MRM1 422→366
Grape juice	M-1	0.01 (n=5)	103 to 112% (Mean 109%)	4.2%	MRM2 422→135
Grape juice	M-1	0.1 (n=5)	106 to 107% (Mean 106%)	0.5%	MRM2 422→135
Grape juice	M-1	Overall (n=10)	103 to 112% (Mean 108%)	3.1%	MRM2 422→135
Wine	M-1	0.01 (n=5)	98 to 100% (Mean 99%)	1.0%	MRM1 422→366
Wine	M-1	0.1 (n=5)	92 to 99% (Mean 96%)	3.1%	MRM1 422→366
Wine	M-1	Overall (n=10)	92 to 100% (Mean 97%)	2.6%	MRM1 422→366
Wine	M-1	0.01 (n=5)	98 to 105% (Mean 102%)	2.4%	MRM2 422→135
Wine	M-1	0.1 (n=5)	93 to 97% (Mean 95%)	1.7%	MRM2 422→135
Wine	M-1	Overall (n=10)	93 to 105% (Mean 99%)	4.0%	MRM2 422→135
Tomato juice	M-1	0.01 (n=5)	91 to 98% (Mean 94%)	2.8%	MRM1 422→366
Tomato juice	M-1	0.1 (n=5)	91 to 98% (Mean 93%)	3.1%	MRM1 422→366
Tomato juice	M-1	Overall (n=10)	91 to 98% (Mean 94%)	2.9%	MRM1 422→366
Tomato juice	M-1	0.01 (n=5)	89 to 98% (Mean 94%)	4.3%	MRM2 422→135
Tomato juice	M-1	0.1 (n=5)	94 to 99% (Mean 96%)	2.2%	MRM2 422→135
Tomato juice	M-1	Overall (n=10)	89 to 99% (Mean 95%)	3.3%	MRM2 422→135
Canned tomatoes	M-1	0.01 (n=5)	96 to 109% (Mean 103%)	5.2%	MRM1 422→366
Canned tomatoes	M-1	0.1 (n=5)	100 to 104% (Mean 102%)	1.2%	MRM1 422→366
Canned tomatoes	M-1	Overall (n=10)	96 to 109%	3.6%	MRM1 422→366

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
			(Mean 103%)		
Canned tomatoes	M-1	0.01 (n=5)	104 to 112% (Mean 106%)	3.1%	MRM2 422→135
Canned tomatoes	M-1	0.1 (n=5)	99 to 102% (Mean 100%)	1.3%	MRM2 422→135
Canned tomatoes	M-1	Overall (n=10)	99 to 112% (Mean 103%)	4.1%	MRM2 422→135
Tomato puree	M-1	0.01 (n=5)	101 to 108% (Mean 105%)	3.2%	MRM1 422→366
Tomato puree	M-1	0.1 (n=5)	102 to 107% (Mean 105%)	1.5%	MRM1 422→366
Tomato puree	M-1	Overall (n=10)	101 to 108% (Mean 105%)	2.4%	MRM1 422→366
Tomato puree	M-1	0.01 (n=5)	106 to 114% (Mean 108%)	3.2%	MRM2 422→135
Tomato puree	M-1	0.1 (n=5)	103 to 107% (Mean 105%)	1.8%	MRM2 422→135
Tomato puree	M-1	Overall (n=10)	103 to 114% (Mean 106%)	3.2%	MRM2 422→135
Canned strawberry	M-1	0.01 (n=5)	100 to 105% (Mean 103%)	1.9%	MRM1 422→366
Canned strawberry	M-1	0.1 (n=5)	97 to 102% (Mean 99%)	2.2%	MRM1 422→366
Canned strawberry	M-1	Overall (n=10)	97 to 105% (Mean 101%)	2.9%	MRM1 422→366
Canned strawberry	M-1	0.01 (n=5)	99 to 110% (Mean 104%)	4.0%	MRM2 422→135
Canned strawberry	M-1	0.1 (n=5)	96 to 103% (Mean 100%)	2.7%	MRM2 422→135
Canned strawberry	M-1	Overall (n=10)	96 to 110% (Mean 102%)	3.8%	MRM2 422→135
Strawberry jam	M-1	0.01 (n=5)	94 to 103% (Mean 99%)	3.2%	MRM1 422→366
Strawberry jam	M-1	0.1 (n=5)	92 to 96% (Mean 94%)	1.7%	MRM1 422→366
Strawberry jam	M-1	Overall (n=10)	92 to 103% (Mean 96%)	4.0%	MRM1 422→366
Strawberry jam	M-1	0.01 (n=5)	96 to 107% (Mean 101%)	5.2%	MRM2 422→135
Strawberry jam	M-1	0.1 (n=5)	92 to 95% (Mean 93%)	1.7%	MRM2 422→135
Strawberry jam	M-1	Overall (n=10)	92 to 107% (Mean 97%)	6.0%	MRM2 422→135
Cooked beans	M-1	0.01 (n=5)	94 to 106% (Mean 97%)	5.1%	MRM1 422→366
Cooked beans	M-1	0.1 (n=5)	92 to 98% (Mean 96%)	2.4%	MRM1 422→366
Cooked beans	M-1	Overall (n=10)	92 to 106% (Mean 97%)	3.9%	MRM1 422→366
Cooked beans	M-1	0.01 (n=5)	94 to 102% (Mean 99%)	3.1%	MRM2 422→135
Cooked beans	M-1	0.1 (n=5)	91 to 98% (Mean 95%)	2.5%	MRM2 422→135
Cooked beans	M-1	Overall (n=10)	91 to 102% (Mean 97%)	3.4%	MRM2 422→135
Canned beans	M-1	0.01 (n=5)	91 to 100% (Mean 96%)	4.4%	MRM1 422→366
Canned beans	M-1	0.1 (n=5)	92 to 100% (Mean 97%)	3.6%	MRM1 422→366
Canned beans	M-1	Overall (n=10)	91 to 100% (Mean 97%)	3.9%	MRM1 422→366
Canned beans	M-1	0.01 (n=5)	93 to 99% (Mean 97%)	2.9%	MRM2 422→135

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Canned beans	M-1	0.1 (n=5)	92 to 104% (Mean 99%)	5.4%	MRM2 422→135
Canned beans	M-1	Overall (n=10)	92 to 104% (Mean 98%)	4.2%	MRM2 422→135
Malt	M-1	0.01 (n=5)	92 to 95% (Mean 94%)	1.6%	MRM1 422→366
Malt	M-1	0.1 (n=5)	90 to 96% (Mean 93%)	3.0%	MRM1 422→366
Malt	M-1	Overall (n=10)	90 to 96% (Mean 93%)	2.3%	MRM1 422→366
Malt	M-1	0.01 (n=5)	92 to 95% (Mean 94%)	1.2%	MRM2 422→135
Malt	M-1	0.1 (n=5)	88 to 94% (Mean 92%)	2.6%	MRM2 422→135
Malt	M-1	Overall (n=10)	88 to 95% (Mean 93%)	2.2%	MRM2 422→135
Cleaned hops	M-1	0.01 (n=5)	92 to 130% (Mean 110%)	12.4%	MRM 422→366 Column 1
Cleaned hops	M-1	0.1 (n=4)	104 to 113% (Mean 107%)	3.9%	MRM 422→366 Column 1
Cleaned hops	M-1	Overall (n=9)	92 to 130% (Mean 109%)	9.3%	MRM 422→366 Column 1
Cleaned hops	M-1	0.01 (n=5)	93 to 107% (Mean 102%)	5.6%	MRM 422→366 Column 2
Cleaned hops	M-1	0.1 (n=5)	99 to 116% (Mean 108%)	6.8%	MRM 422→366 Column 2
Cleaned hops	M-1	Overall (n=10)	93 to 116% (Mean 105%)	6.7%	MRM 422→366 Column 2
Beer	M-1	0.01 (n=5)	97 to 102% (Mean 100%)	2.4%	MRM1 422→366
Beer	M-1	0.1 (n=5)	98 to 102% (Mean 100%)	1.6%	MRM1 422→366
Beer	M-1	Overall (n=10)	97 to 102% (Mean 100%)	1.9%	MRM1 422→366
Beer	M-1	0.01 (n=5)	99 to 103% (Mean 100%)	1.5%	MRM2 422→135
Beer	M-1	0.1 (n=5)	98 to 103% (Mean 100%)	2.2%	MRM2 422→135
Beer	M-1	Overall (n=10)	98 to 103% (Mean 100%)	1.8%	MRM2 422→135

Table 142 Recovery results from method validation of M-3 using the analytical method RES/RAM/005 in processed crop commodities

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Apple juice	M-3	0.01 (n=5)	94 to 103% (Mean 98%)	3.6%	MRM1 422→366
Apple juice	M-3	0.1 (n=5)	95 to 102% (Mean 99%)	2.6%	MRM1 422→366
Apple juice	M-3	Overall (n=10)	94 to 103% (Mean 98%)	2.9%	MRM1 422→366
Apple juice	M-3	0.01 (n=5)	93 to 106% (Mean 101%)	5.3%	MRM2 422→135
Apple juice	M-3	0.1 (n=5)	96 to 100% (Mean 98%)	2.0%	MRM2 422→135
Apple juice	M-3	Overall (n=10)	93 to 106% (Mean 100%)	4.2%	MRM2 422→135
Apple dry pomace	M-3	0.01 (n=5)	96 to 108% (Mean 100%)	4.9%	MRM1 422→366
Apple dry pomace	M-3	0.1 (n=5)	98 to 106% (Mean 102%)	2.9%	MRM1 422→366

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Apple dry pomace	M-3	Overall (n=10)	96 to 108% (Mean 101%	3.9%	MRM1 422→366
Apple dry pomace	M-3	0.01 (n=5)	91 to 109% (Mean 98%)	7.1%	MRM2 422→135
Apple dry pomace	M-3	0.1 (n=5)	97 to 104% (Mean 101%)	2.7%	MRM2 422→135
Apple dry pomace	M-3	Overall (n=10)	91 to 109% (Mean 99%)	5.2%	MRM2 422→135
Apple sauce	M-3	0.01 (n=5)	102 to 109% (Mean 105%)	2.5%	MRM1 422→366
Apple sauce	M-3	0.1 (n=5)	105 to 109% (Mean 107%)	1.8%	MRM1 422→366
Apple sauce	M-3	Overall (n=10)	102 to 109% (Mean 106%)	2.3%	MRM1 422→366
Apple sauce	M-3	0.01 (n=5)	101 to 113% (Mean 106%)	5.2%	MRM2 422→135
Apple sauce	M-3	0.1 (n=5)	104 to 108% (Mean 106%)	1.5%	MRM2 422→135
Apple sauce	M-3	Overall (n=10)	101 to 113% (Mean 106%)	3.6%	MRM2 422→135
Dried apples	M-3	0.01 (n=5)	95 to 102% (Mean 100%)	2.7%	MRM1 422→366
Dried apples	M-3	0.1 (n=5)	93 to 100% (Mean 96%)	2.9%	MRM1 422→366
Dried apples	M-3	Overall (n=10)	93 to 102% (Mean 98%)	3.2%	MRM1 422→366
Dried apples	M-3	0.01 (n=5)	89 to 103% (Mean 96%)	5.7%	MRM2 422→135
Dried apples	M-3	0.1 (n=5)	95 to 101% (Mean 96%)	2.4%	MRM2 422→135
Dried apples	M-3	Overall (n=10)	89 to 103% (Mean 96%)	4.1%	MRM2 422→135
Raisins	M-3	0.01 (n=5)	93 to 101% (Mean 97%)	2.6%	MRM1 422→366
Raisins	M-3	0.1 (n=5)	91 to 98% (Mean 94%)	2.7%	MRM1 422→366
Raisins	M-3	Overall (n=10)	91 to 101% (Mean 96%)	2.9%	MRM1 422→366
Raisins	M-3	0.01 (n=5)	96 to 100% (Mean 97%)	1.8%	MRM2 422→135
Raisins	M-3	0.1 (n=5)	95 to 100% (Mean 98%)	1.8%	MRM2 422→135
Raisins	M-3	Overall (n=10)	95 to 100% (Mean 98%)	1.7%	MRM2 422→135
Grape juice	M-3	0.01 (n=5)	104 to 108% (Mean 105%)	1.7%	MRM1 422→366
Grape juice	M-3	0.1 (n=5)	102 to 104% (Mean 103%)	1.6%	MRM1 422→366
Grape juice	M-3	Overall (n=10)	102 to 108% (Mean 104%)	2.0%	MRM1 422→366
Grape juice	M-3	0.01 (n=5)	103 to 109% (Mean 106%)	2.2%	MRM2 422→135
Grape juice	M-3	0.1 (n=5)	102 to 107% (Mean 105%)	2.1%	MRM2 422→135
Grape juice	M-3	Overall (n=10)	102 to 109% (Mean 105%)	2.1%	MRM2 422→135
Wine	M-3	0.01 (n=5)	96 to 98% (Mean 97%)	1.2%	MRM1 422→366
Wine	M-3	0.1 (n=5)	93 to 98% (Mean 95%)	2.1%	MRM1 422→366
Wine	M-3	Overall (n=10)	93 to 98% (Mean 96%)	1.8%	MRM1 422→366
Wine	M-3	0.01 (n=5)	98 to 106%	2.7%	MRM2 422→135

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
			(Mean 102%)		
Wine	M-3	0.1 (n=5)	94 to 99% (Mean 97%)	2.4%	MRM2 422→135
Wine	M-3	Overall (n=10)	94 to 106% (Mean 99%)	3.5%	MRM2 422→135
Tomato juice	M-3	0.01 (n=5)	87 to 95% (Mean 91%)	3.4%	MRM1 422→366
Tomato juice	M-3	0.1 (n=5)	91 to 101% (Mean 93%)	4.3%	MRM1 422→366
Tomato juice	M-3	Overall (n=10)	87 to 101% (Mean 92%)	3.9%	MRM1 422→366
Tomato juice	M-3	0.01 (n=5)	89 to 94% (Mean 92%)	2.6%	MRM2 422→135
Tomato juice	M-3	0.1 (n=5)	91 to 101% (Mean 95%)	4.3%	MRM2 422→135
Tomato juice	M-3	Overall (n=10)	89 to 101% (Mean 94%)	3.85%	MRM2 422→135
Canned tomatoes	M-3	0.01 (n=5)	97 to 103% (Mean 100%)	2.3%	MRM1 422→366
Canned tomatoes	M-3	0.1 (n=5)	95 to 98% (Mean 97%)	1.8%	MRM1 422→366
Canned tomatoes	M-3	Overall (n=10)	95 to 103% (Mean 98%)	2.6%	MRM1 422→366
Canned tomatoes	M-3	0.01 (n=5)	96 to 104% (Mean 100%)	3.3%	MRM2 422→135
Canned tomatoes	M-3	0.1 (n=5)	96 to 99% (Mean 98%)	1.7%	MRM2 422→135
Canned tomatoes	M-3	Overall (n=10)	96 to 104% (Mean 99%)	2.9%	MRM2 422→135
Tomato puree	M-3	0.01 (n=5)	102 to 106% (Mean 103%)	2.3%	MRM1 422→366
Tomato puree	M-3	0.1 (n=5)	102 to 108% (Mean 105%)	2.4%	MRM1 422→366
Tomato puree	M-3	Overall (n=10)	102 to 108% (Mean 104%)	2.3%	MRM1 422→366
Tomato puree	M-3	0.01 (n=5)	99 to 106% (Mean 103%)	2.9%	MRM2 422→135
Tomato puree	M-3	0.1 (n=5)	102 to 111% (Mean 107%)	2.9%	MRM2 422→135
Tomato puree	M-3	Overall (n=10)	99 to 111% (Mean 105%)	3.4%	MRM2 422→135
Canned strawberry	M-3	0.01 (n=5)	101 to 108% (Mean 103%)	2.5%	MRM1 422→366
Canned strawberry	M-3	0.1 (n=5)	101 to 105% (Mean 103%)	1.7%	MRM1 422→366
Canned strawberry	M-3	Overall (n=10)	101 to 108% (Mean 103%)	2.0%	MRM1 422→366
Canned strawberry	M-3	0.01 (n=5)	97 to 107% (Mean 101%)	4.2%	MRM2 422→135
Canned strawberry	M-3	0.1 (n=5)	98 to 105% (Mean 101%)	2.8%	MRM2 422→135
Canned strawberry	M-3	Overall (n=10)	97 to 107% (Mean 101%)	3.4%	MRM2 422→135
Strawberry jam	M-3	0.01 (n=5)	85 to 97% (Mean 90%)	5.7%	MRM1 422→366
Strawberry jam	M-3	0.1 (n=5)	84 to 93% (Mean 89%)	4.5%	MRM1 422→366
Strawberry jam	M-3	Overall (n=10)	84 to 97% (Mean 89%)	4.%	MRM1 422→366
Strawberry jam	M-3	0.01 (n=5)	83 to 99% (Mean 92%)	6.5%	MRM2 422→135
Strawberry jam	M-3	0.1 (n=5)	84 to 92% (Mean 87%)	4.1%	MRM2 422→135

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Strawberry jam	M-3	Overall (n=10)	83 to 99% (Mean 90%)	5.8%	MRM2 422→135
Cooked beans	M-3	0.01 (n=5)	85 to 96% (Mean 90%)	5.3%	MRM1 422→366
Cooked beans	M-3	0.1 (n=5)	88 to 94% (Mean 91%)	3.2%	MRM1 422→366
Cooked beans	M-3	Overall (n=10)	85 to 96% (Mean 91%)	4.2%	MRM1 422→366
Cooked beans	M-3	0.01 (n=5)	89 to 91% (Mean 91%)	1.1%	MRM2 422→135
Cooked beans	M-3	0.1 (n=5)	94 to 98% (Mean 96%)	1.9%	MRM2 422→135
Cooked beans	M-3	Overall (n=10)	89 to 98% (Mean 93%)	3.2%	MRM2 422→135
Canned beans	M-3	0.01 (n=5)	91 to 97% (Mean 94%)	2.9%	MRM1 422→366
Canned beans	M-3	0.1 (n=5)	92 to 100% (Mean 96%)	3.9%	MRM1 422→366
Canned beans	M-3	Overall (n=10)	91 to 100% (Mean 95%)	3.4%	MRM1 422→366
Canned beans	M-3	0.01 (n=5)	87 to 99% (Mean 93%)	4.9%	MRM2 422→135
Canned beans	M-3	0.1 (n=5)	91 to 100% (Mean 95%)	4.3%	MRM2 422→135
Canned beans	M-3	Overall (n=10)	87 to 100% (Mean 94%)	4.5%	MRM2 422→135
Malt	M-3	0.01 (n=5)	88 to 95% (Mean 92%)	3.3%	MRM1 422→366
Malt	M-3	0.1 (n=5)	87 to 94% (Mean 91%)	3.0%	MRM1 422→366
Malt	M-3	Overall (n=10)	87 to 95% (Mean 91%)	3.0%	MRM1 422→366
Malt	M-3	0.01 (n=5)	88 to 94% (Mean 91%)	2.2%	MRM2 422→135
Malt	M-3	0.1 (n=5)	85 to 93% (Mean 91%)	3.5%	MRM2 422→135
Malt	M-3	Overall (n=10)	85 to 94% (Mean 91%)	2.8%	MRM2 422→135
Cleaned hops	M-3	0.01 (n=5)	81 to 96% (Mean 90%)	6.3%	MRM 422→366 Column 1
Cleaned hops	M-3	0.1 (n=5)	74 to 81% (Mean 78%)	4.6%	MRM 422→366 Column 1
Cleaned hops	M-3	Overall (n=10)	74 to 96% (Mean 84%)	9.3%	MRM 422→366 Column 1
Cleaned hops	M-3	0.01 (n=5)	83 to 103% (Mean 93%)	8.2%	MRM 422→366 Column 2
Cleaned hops	M-3	0.1 (n=5)	76 to 85% (Mean 80%)	3.7%	MRM 422→366 Column 2
Cleaned hops	M-3	Overall (n=10)	76 to 103% (Mean 87%)	10.1%	MRM 422→366 Column 2
Beer	M-3	0.01 (n=5)	92 to 97% (Mean 94%)	2.3%	MRM1 422→366
Beer	M-3	0.1 (n=5)	96 to 99% (Mean 97%)	1.2%	MRM1 422→366
Beer	M-3	Overall (n=10)	92 to 99% (Mean 95%)	2.4%	MRM1 422→366
Beer	M-3	0.01 (n=5)	90 to 100% (Mean 94%)	3.8%	MRM2 422→135
Beer	M-3	0.1 (n=5)	96 to 100% (Mean 98%)	1.1%	MRM2 422→135
Beer	M-3	Overall (n=10)	90 to 100% (Mean 96%)	3.3%	MRM2 422→135

Table 143 Characteristics for the analytical method used for validation of fenpyroximate, M-1 and M-3 residues in processed crop commodities

	Fenpyroximate	M-1	M-3
Specificity (all crops)	blank value < 30% LOQ Product ion MS scan presented	blank value < 30% LOQ Product ion MS scan presented	blank value < 30% LOQ Product ion MS scan presented
Calibration (type, number of data points)	typical calibration data presented| typical calibration line equation presented linear, no weighting applied r=≥0.996 6 or 7 data points Matrix matched used Both MRM1 and MRM2 were reported for all crops except hops. For hops linearity for both columns was reported	typical calibration data presented| typical calibration line equation presented linear, no weighting applied r=≥0.996 6 or 7 data points Matrix matched used Both MRM1 and MRM2 were reported for all crops except hops. For hops linearity for both columns was reported	typical calibration data presented| typical calibration line equation presented linear, no weighting applied r=≥0.998 6 or 7 data points Matrix matched used Both MRM1 and MRM2 were reported for all crops except hops. For hops linearity for both columns was reported
Calibration range	0.1 to 10.0 ng/mL or 0.1 to 5 ng/mL for all matrices Corresponding calibration range 0.0025 to 0.25 mg/kg or 0.0025 to 0.125 mg/kg fenpyroximate	0.1 to 10.0 ng/mL or 0.1 to 5 ng/mL for all matrices Corresponding calibration range 0.0025 to 0.25 mg/kg or 0.0025 to 0.125 mg/kg M-1	0.1 to 10.0 ng/mL or 0.1 to 5 ng/mL for all matrices Corresponding calibration range 0.0025 to 0.25 mg/kg or 0.0025 to 0.125 mg/kg M-3
Assessment of matrix effects is presented	Yes, significant in some commodities but matrix matched standards used for all matrices	Yes, significant in some commodities but matrix matched standards used for all matrices	Yes, significant in some commodities but matrix matched standards used for all matrices
Limit of determination/quantification	LOQ=0.01 mg/kg in all matrices	LOQ=0.01 mg/kg in all matrices	LOQ=0.01 mg/kg in all matrices
Comment	Acceptable against SANCO/3029/99 rev. 4 and SANCO/825/00 rev 8.1 criteria. Method suitable for data generation in processed commodities	Acceptable against SANCO/3029/99 rev. 4 and SANCO/825/00 rev 8.1 criteria. Method suitable for data generation in processed commodities	Acceptable against SANCO/3029/99 rev. 4 and SANCO/825/00 rev 8.1 criteria. Method suitable for data generation in processed commodities

The method RES/RAM/005 based on QuEChERS was successfully validated in all processed crop matrix types and is compliant with SANCO/3029/99 rev. 4 and SANCO/825/00 rev. 8.1. The method is appropriate for measuring concentrations of fenpyroximate or its metabolites M-1 and M-3 in processed crop commodities for data generation risk assessment purposes.

GC-MS method

DFG S19 method by GC-MS (Fenpyroximate/Crops/DB/01/2) for determination of residues of fenpyroximate and M-1 in strawberries (field and protected) was validated for linearity, specificity, precision and accuracy (Oxspring S., 2003a, report No.: R-4162; Oxspring S., 2003b, report No.: R-4163). Residues of fenpyroximate or M-1 in high acid crops (100 g sample weight) are determined by direct extraction with acetone (200 mL) and water (100 mL–the expected water volume in 100 g of crop) by maceration for 3 minutes. Samples are filtered and a known volume (200 mL) is transferred to a separating funnel. Sodium chloride (20 g) is added and the extracts are partitioned with dichloromethane (100 mL) with the layers allowed to separate. The upper organic layer is collected and dried over anhydrous sodium sulphate and then reduced to dryness under rotary evaporation. The extract is then dissolved in cyclohexane:ethyl acetate (10 mL, 1:1 v/v) and purified using gel permeation chromatography, collecting the eluant fraction known to contain any fenpyroximate or M-1. Extracts are taken to dryness under rotary evaporation and adjusted to a final volume with ethyl

acetate (5 mL) prior to determination using GC-MS employing a DB-1 12 m × 0.2 mm i.d. capillary column, 0.3 µm df film thickness. Two selected ions are monitored for quantification or possible confirmation. The lowest level tested (LOQ) was 0.01 mg/kg fenpyroximate or M-1 in strawberries.

Table 144 Recovery results from method validation of fenpyroximate using the analytical method Fenpyroximate/Crops/DB/01/2 in strawberries

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Strawberry (high acid)	fenpyroximate	0.01 (n=4)	97 to 110% (Mean 93%)	2.1%	R-4162
Strawberry (high acid)	fenpyroximate	0.01 (n=2)	76 to 109% (mean=93%)	NA	R-4163
Strawberry (high acid)	fenpyroximate	Combined data 0.01 (n=6)	76 to 110% (Mean 101%)	13%	R-4162 R-4163
Strawberry (high acid)	fenpyroximate	0.05 (n=1)	75%	NA	R-4162
Strawberry (high acid)	fenpyroximate	0.1 (n=1)	93%	NA	R-4162
Strawberry (high acid)	fenpyroximate	0.2 (n=1)	107%	NA	R-4162
Strawberry (high acid)	fenpyroximate	0.2 (n=1)	72%	NA	R-4163
Strawberry (high acid)	fenpyroximate	0.5 (n=1)	122%	NA	R-4162
Strawberry (high acid)	fenpyroximate	Overall (n=11)	72 to 122% (Mean 98%)	17%	R-4162 R-4163

Table 145 Recovery results from method validation of M-1 using the analytical method Fenpyroximate/Crops/DB/01/2 in strawberries

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Strawberry (high acid)	M-1	0.01 (n=4)	82 to 110% (Mean 95%)	12%	R-4162
Strawberry (high acid)	M-1	0.01 (n=2)	74 to 75% (mean=75%)	NA	R-4163
Strawberry (high acid)	M-1	Combined data 0.01 (n=6)	74 to 110% (Mean 88%)	16%	R-4162 R-4163
Strawberry (high acid)	M-1	0.05 (n=1)	78%	NA	R-4162
Strawberry (high acid)	M-1	0.1 (n=1)	85%	NA	R-4162
Strawberry (high acid)	M-1	0.2 (n=1)	121%	NA	R-4162
Strawberry (high acid)	M-1	0.2 (n=1)	71%	NA	R-4163
Strawberry (high acid)	M-1	0.5 (n=1)	136%	NA	R-4162
Strawberry (high acid)	M-1	Overall (n=11)	71 to 136% (Mean 93%)	23%	R-4162 R-4163

Table 146 Characteristics for the analytical method used for validation of fenpyroximate and M-1 residues in strawberries

	Fenpyroximate	M-1
Specificity (all crops)	blank value < 30% LOQ	blank value < 30% LOQ
Calibration (type, number of data points)	typical calibration data presented| typical calibration line equation presented linear, no weighting applied r^2=>0.997 for strawberry 4 or 5 data points Matrix matched used	typical calibration data presented| typical calibration line equation presented linear, no weighting applied r^2=>0.997 for strawberry 4 or 5 data points Matrix matched used
Calibration range	0.025 to 1.0 µg/mL (R-4162) Corresponding calibration range 0.00375 to 0.15 mg/kg fenpyroximate. 0.025 to 0.5 µg/mL (R-4163) Corresponding calibration range 0.00375 to 0.075 mg/kg fenpyroximate.	0.025 to 1.0 µg/mL (R-4162) Corresponding calibration range 0.00375 to 0.15 mg/kg M-1. 0.025 to 0.5 µg/mL (R-4163) Corresponding calibration range 0.00375 to 0.075 mg/kg M-1.

	Fenpyroximate	M-1
Assessment of matrix effects is presented	No, matrix matched standards used for strawberry	No, matrix matched standards used for strawberry
Limit of determination/quantification	LOQ=0.01 mg/kg in strawberry	LOQ=0.01 mg/kg in strawberry
Comment	Generally acceptable against SANCO/3029/99 rev. 4 criteria. Method suitable for data generation in high acid matrix types.	Generally acceptable against SANCO/3029/99 rev. 4 criteria. Method suitable for data generation in high acid matrix types.

The method Fenpyroximate/Crops/DB/01/2 based on DFG S19 was validated in strawberry matrix and is generally compliant with SANCO/3029/99 rev. 4. The method is appropriate for measuring concentrations of fenpyroximate or its metabolite M-1 in food of plant origin with high acid content for risk assessment purposes.

Methods for food of animal origin

The enforcement monitoring method

QuEChERS method by HPLC-MS/MS for determination of residues of fenpyroximate and its metabolite M-3 in dairy products was validated for linearity, specificity precision and accuracy (Ihara T., 2016a, report No.: A-4082).

Residues of fenpyroximate or M-3 in muscle, liver, kidney or eggs (10 g sample weight) are determined by direct extraction with acetonitrile : water (40 mL, 4:1, 30 seconds homogenisation). After centrifugation and transfer of the solvent to a separating funnel, the solid remaining is further extracted with acetonitrile : water (40 mL, 4:1, 1 minute shaking). After centrifugation, the solvent extracts are combined in the separating funnel with addition of sodium chloride. The combined extract is shaken and the layers allowed to separate so that the upper acetonitrile layer can be collected and the volume adjusted with acetonitrile (final volume 100 mL). An aliquot of the acetonitrile extract (20 mL, 2 g sample equivalent) is partitioned with hexane (20 mL) to remove any fat and the lower acetonitrile later is evaporated with addition of water (1 mL). Methanol and water (1 mL of each) is added to the extract prior to clean-up.

Residues of fenpyroximate or M-3 in fat (10 g sample weight) are determined by direct extraction with acetonitrile (40 mL, 30 seconds homogenisation). After centrifugation and transfer of the solvent to a separating funnel, the solid remaining is further extracted with acetonitrile (40 mL, 1 minute shaking). After centrifugation, the solvent extracts are combined in the separating funnel with addition of sodium chloride. The combined extract is shaken and the layers allowed to separate so that the upper acetonitrile layer can be collected and the volume adjusted with acetonitrile (final volume 100 mL). An aliquot of the acetonitrile extract (20 mL, 2 g sample equivalent) is partitioned with hexane (20 mL) to remove any fat and the lower acetonitrile later is evaporated with addition of water (1 mL). Methanol and water (1 mL of each) is added to the extract prior to clean-up.

Residues of fenpyroximate or M-3 in milk (20 g sample weight) are determined by direct extraction with acetonitrile (10 mL, 1 minute shaking). After centrifugation and transfer of the solvent to a separating funnel, the solid remaining is further extracted with acetonitrile (50 mL, 1 minute shaking). After centrifugation, the solvent extracts are combined in the separating funnel with addition of sodium chloride. The combined extract is shaken and the layers allowed to separate so that the upper acetonitrile layer can be collected and the volume adjusted with acetonitrile (final volume 200 mL). An aliquot of the acetonitrile extract (20 mL, 2 g sample equivalent) is partitioned with hexane (20 mL) to remove any fat and the lower acetonitrile later is evaporated with addition of water (1 mL). Methanol and water (1 mL of each) is added to the extract prior to clean-up.

All extracts are cleaned up by means of a C18 SPE tube (BondElut, 500 mg phase) with the final eluted extract containing any fenpyroximate or M-3 adjusted to a volume of 5 mL in acetonitrile : water, 1:1 (sample concentration 0.4 g/mL equivalent).

All samples were measured using LC-MS/MS employing a Cadenza CD-18 column, 3 µm particle size and monitoring at two MRM transitions for each analyte. Quantification of extracts was performed using external calibration standards over the range 0.3 to 30.0 ng/mL for all matrices and for both analytes. For analysis of fenpyroximate in all matrices, matrix matched standards were used. For analysis of M-3 in all matrices, solvent standards were used.

The lowest level tested (LOQ) was 0.005 mg/kg fenpyroximate or M-3 in all matrices.

Table 147 Recovery results from method validation of fenpyroximate using the analytical method in food of animal origin

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Milk	fenpyroximate	0.005 (n=5)	73.6 to 81.1% (Mean 78.4%)	4.2%	MRM1 422→135
Milk	fenpyroximate	0.05 (n=4)	73.5 to 75.4% (Mean 74.7%)	1.1%	MRM1 422→135
Milk	fenpyroximate	0.005 (n=5)	72.4 to 85.7% (Mean 77.4%)	6.5%	MRM2 422→107
Milk	fenpyroximate	0.05 (n=4)	72.1 to 74.7% (Mean 73.4%)	1.6%	MRM2 422→107
Egg	fenpyroximate	0.005 (n=5)	63.5 to 87.7% (Mean 77.5%)	11.8%	MRM1 422→135
Egg	fenpyroximate	0.05 (n=5)	88.4 to 115.5% (Mean 100.2%)	10.0%	MRM1 422→135
Egg	fenpyroximate	0.005 (n=5)	65.1 to 83.7% (Mean 74.7%)	10.5%	MRM2 422→107
Egg	fenpyroximate	0.05 (n=5)	92.7 to 113.2% (Mean 100.8%)	7.6%	MRM2 422→107
Muscle	fenpyroximate	0.005 (n=5)	73.4 to 90.0% (Mean 80.4%)	8.2%	MRM1 422→135
Muscle	fenpyroximate	0.05 (n=5)	76.7 to 92.5% (Mean 84.0%)	6.7%	MRM1 422→135
Muscle	fenpyroximate	0.005 (n=5)	72.5 to 81.0% (Mean 75.5%)	4.3%	MRM2 422→107
Muscle	fenpyroximate	0.05 (n=5)	76.1 to 87.7% (Mean 83.4%)	5.6%	MRM2 422→107
Fat	fenpyroximate	0.005 (n=5)	65.5 to 90.8% (Mean 74.9%)	12.6%	MRM1 422→135
Fat	fenpyroximate	0.05 (n=5)	61.3 to 75.7% (Mean 70.8%)	8.9%	MRM1 422→135
Fat	fenpyroximate	0.005 (n=5)	74.7 to 93.3% (Mean 85.0%)	8.5%	MRM2 422→107
Fat	fenpyroximate	0.05 (n=5)	60.6 to 79.9% (Mean 70.9%)	9.9%	MRM2 422→107
Liver	fenpyroximate	0.005 (n=5)	77.5 to 93.0% (Mean 84.3%)	8.4%	MRM1 422→135
Liver	fenpyroximate	0.05 (n=5)	117.3 to 122.3% (Mean 119.7%)	1.6%	MRM1 422→135
Liver	fenpyroximate	0.005 (n=5)	75.7 to 86.7% (Mean 81.3%)	5.8%	MRM2 422→107
Liver	fenpyroximate	0.05 (n=5)	117.5 to 121.5% (Mean 119.7%)	1.3%	MRM2 422→107
Kidney	fenpyroximate	0.005 (n=5)	66.6 to 72.8% (Mean 70.2%)	3.3%	MRM1 422→135
Kidney	fenpyroximate	0.05 (n=5)	80.4 to 101.0% (Mean 85.0%)	10.6%	MRM1 422→135
Kidney	fenpyroximate	0.005 (n=5)	66.9 to 73.5% (Mean 71.6%)	3.8%	MRM2 422→107
Kidney	fenpyroximate	0.05 (n=5)	77.6 to 100.0% (Mean 84.2%)	10.8%	MRM2 422→107

Table 148 Recovery results from method validation of M-3 using the analytical method in food of animal origin

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Milk	M-3	0.005 (n=5)	84.0 to 94.2% (Mean 87.1%)	4.8%	MRM1 366→231
Milk	M-3	0.05 (n=5)	87.9 to 91.1% (Mean 89.6%)	1.5%	MRM1 366→231
Milk	M-3	0.005 (n=5)	82.7 to 90.8% (Mean 87.3%)	4.1%	MRM2 366→215
Milk	M-3	0.05 (n=5)	89.4 to 93.0% (Mean 91.5%)	1.5%	MRM2 366→215
Egg	M-3	0.005 (n=5)	65.6 to 83.7% (Mean 72.6%)	9.7%	MRM1 366→231
Egg	M-3	0.05 (n=5)	86.9 to 94.2% (Mean 91.2%)	3.0%	MRM1 366→231
Egg	M-3	0.005 (n=5)	70.0 to 72.9% (Mean 71.5%)	1.5%	MRM2 366→215
Egg	M-3	0.05 (n=5)	89.9 to 94.0% (Mean 92.1%)	1.7%	MRM2 366→215
Muscle	M-3	0.005 (n=5)	77.7 to 81.4% (Mean 79.1%)	2.0%	MRM1 366→231
Muscle	M-3	0.05 (n=5)	83.7 to 91.2% (Mean 87.4%)	3.1%	MRM1 366→231
Muscle	M-3	0.005 (n=5)	75.6 to 85.3% (Mean 80.6%)	4.4%	MRM2 366→215
Muscle	M-3	0.05 (n=5)	80.7 to 89.4% (Mean 86.0%)	3.7%	MRM2 366→215
Fat	M-3	0.005 (n=5)	77.7 to 88.2% (Mean 83.2%)	4.5%	MRM1 366→231
Fat	M-3	0.05 (n=5)	83.8 to 86.4% (Mean 784.9%)	1.4%	MRM1 366→231
Fat	M-3	0.005 (n=5)	70.2 to 90.0% (Mean 79.1%)	9.2%	MRM2 366→215
Fat	M-3	0.05 (n=5)	79.6 to 85.9% (Mean 83.2%)	3.0%	MRM2 366→215
Liver	M-3	0.005 (n=5)	82.0 to 90.4% (Mean 86.9%)	4.9%	MRM1 366→231
Liver	M-3	0.05 (n=5)	86.6 to 90.0% (Mean 87.7%)	1.8%	MRM1 366→231
Liver	M-3	0.005 (n=5)	72.3 to 84.7% (Mean 80.7%)	6.2%	MRM2 366→215
Liver	M-3	0.05 (n=5)	86.7 to 90.6% (Mean 88.4%)	1.9%	MRM2 366→215
Kidney	M-3	0.005 (n=5)	75.7 to 81.2% (Mean 79.3%)	2.8%	MRM1 366→231
Kidney	M-3	0.05 (n=5)	86.3 to 91.7% (Mean 88.7%)	2.3%	MRM1 366→231
Kidney	M-3	0.005 (n=5)	75.9 to 79.2% (Mean 77.1%)	1.7%	MRM2 366→215
Kidney	M-3	0.05 (n=5)	88.5 to 92.9% (Mean 91.1%)	1.8%	MRM2 366→215

Table 149 Characteristics for the analytical method used for validation of fenpyroximate and M-3 residues in food of animal origin

	Fenpyroximate	M-3
Specificity (all matrices)	blank value < 30% LOQ Product ion MS scan presented	blank value < 30% LOQ Product ion MS scan presented
Calibration (type, number of data points)	typical calibration data presented| typical calibration line equation presented	typical calibration data presented| typical calibration line equation presented

	Fenpyroximate	M-3
	linear, no weighting applied r=≥0.997 5 data points Matrix matched used Both MRM1 and MRM2 were reported for all matrices	linear, no weighting applied r=>0.999 5 data points Non-matrix matched used (solvent) Both MRM1 and MRM2 were reported
Calibration range	0.3 to 30.0 ng/mL for all matrices Corresponding calibration range 0.00075 to 0.075 mg/kg fenpyroximate in all matrices.	0.3 to 30.0 ng/mL for all matrices Corresponding calibration range 0.00075 to 0.075 mg/kg M-3 in all matrices.
Assessment of matrix effects is presented	Yes, significant in all matrices. Matrix matched standards used	Yes, not significant in any matrices. Non-matrix matched (solvent) standards used
Limit of determination/quantification	LOQ=0.005 mg/kg in all matrices	LOQ=0.005 mg/kg in all matrices
Comment	Acceptable against SANCO/825/00 rev 8.1 criteria. Method suitable for monitoring in food of animal origin.	Acceptable against SANCO/825/00 rev 8.1 criteria. Method suitable for monitoring in food of animal origin.

A QuEChERS based method was successfully validated in all food of animal origin matrix types and is compliant with SANCO/825/00 rev. 8.1. The method is appropriate for measuring concentrations of fenpyroximate or its metabolite M-3 in food of animal origin for monitoring purposes.

The Independent Laboratory Validation of the method for determination of fenpyroximate and its metabolite M-3: was studied linearity, specificity precision and accuracy with bovine muscle and milk (Nagata T., 2016, report No.: A-4099). Residues of fenpyroximate or M-3 in muscle (10 g sample weight) are determined by direct extraction with acetonitrile : water (40 mL, 4:1, 30 seconds homogenisation). After centrifugation and transfer of the solvent to a separating funnel, the solid remaining is further extracted with acetonitrile : water (40 mL, 4:1, 1 minute shaking). After centrifugation, the solvent extracts are combined in the separating funnel with addition of sodium chloride. The combined extract is shaken and the layers allowed to separate so that the upper acetonitrile layer can be collected and the volume adjusted with acetonitrile (final volume 100 mL). An aliquot of the acetonitrile extract (20 mL, 2 g sample equivalent) is partitioned with hexane (20 mL) to remove any fat and the lower acetonitrile later is evaporated with addition of water (1 mL). Methanol and water (1 mL of each) is added to the extract prior to clean-up.

Residues of fenpyroximate or M-3 in milk (20 g sample weight) are determined by direct extraction with acetonitrile (10 mL, 1 minute shaking). After centrifugation and transfer of the solvent to a separating funnel, the solid remaining is further extracted with acetonitrile (50 mL, 1 minute shaking). After centrifugation, the solvent extracts are combined in the separating funnel with addition of sodium chloride. The combined extract is shaken and the layers allowed to separate so that the upper acetonitrile layer can be collected and the volume adjusted with acetonitrile (final volume 200 mL). An aliquot of the acetonitrile extract (20 mL, 2 g sample equivalent) is partitioned with hexane (20 mL) to remove any fat and the lower acetonitrile later is evaporated with addition of water (1 mL). Methanol and water (1 mL of each) is added to the extract prior to clean-up.

All extracts are cleaned up by means of a C18 SPE tube (BondElut, 500 mg phase) with the final eluted extract containing any fenpyroximate or M-3 adjusted to a volume of 5 mL in acetonitrile : water, 1:1 (sample concentration 0.4 g/mL equivalent).

All samples were measured using LC-MS/MS employing an L-column2 ODS, 5 µm particle size and monitoring at two MRM transitions for each analyte. Quantification of extracts was performed using external calibration standards over the range 1.5 to 150 pg (equivalent to 0.3 to 30.0 ng/mL) for both matrices and for both analytes. For analysis of fenpyroximate and M-3 in both matrices, matrix matched standards were used. The lowest level tested (LOQ) was 0.005 mg/kg fenpyroximate or M-3 in all matrices.

Table 150 Recovery results from method validation of fenpyroximate using the analytical method in food of animal origin

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Milk	fenpyroximate	0.005 (n=5)	89 to 94% (Mean 91%)	2%	MRM1 422→135
Milk	fenpyroximate	0.05 (n=5	80 to 83% (Mean 82%)	2%	MRM1 422→135
Milk	fenpyroximate	0.005 (n=5)	79 to 90% (Mean 84%)	5%	MRM2 422→107
Milk	fenpyroximate	0.05 (n=5)	78 to 84% (Mean 82%)	3%	MRM2 422→107
Muscle	fenpyroximate	0.005 (n=5)	78 to 87% (Mean 82%)	4%	MRM1 422→135
Muscle	fenpyroximate	0.05 (n=5)	72 to 82% (Mean 77%)	5%	MRM1 422→135
Muscle	fenpyroximate	0.005 (n=5)	72 to 84% (Mean 80%)	6%	MRM2 422→107
Muscle	fenpyroximate	0.05 (n=5)	72 to 83% (Mean 77%)	6%	MRM2 422→107

Table 151 Recovery results from method validation of M-3 using the analytical method in food of animal origin

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Milk	M-3	0.005 (n=5)	84 to 93% (Mean 88%)	5%	MRM1 365.9→231
Milk	M-3	0.05 (n=5)	95 to 98% (Mean 97%)	1%	MRM1 365.9→231
Milk	M-3	0.005 (n=5)	86 to 92% (Mean 89%)	3%	MRM2 365.9→215
Milk	M-3	0.05 (n=5)	94 to 97% (Mean 96%)	2%	MRM2 365.9→215
Muscle	M-3	0.005 (n=5)	87 to 92% (Mean 90%)	2%	MRM1 365.9→231
Muscle	M-3	0.05 (n=5)	91 to 94% (Mean 93%)	1%	MRM1 365.9→231
Muscle	M-3	0.005 (n=5)	83 to 88% (Mean 86%)	2%	MRM2 365.9→215
Muscle	M-3	0.05 (n=5)	91 to 99% (Mean 95%)	3%	MRM2 365.9→215

Table 152: Characteristics for the analytical method used for validation of fenpyroximate and M-3 residues in food of animal origin

	Fenpyroximate	M-3
Specificity (all crops)	blank value < 30% LOQ Product ion MS scan presented	blank value < 30% LOQ Product ion MS scan presented
Calibration (type, number of data points)	typical calibration data presented| typical calibration line equation presented linear, no weighting applied r=≥0.999 5 data points Matrix matched used Both MRM1 and MRM2 were reported for all matrices	typical calibration data presented| typical calibration line equation presented linear, no weighting applied r=>0.999 5 data points Matrix matched used Both MRM1 and MRM2 were reported
Calibration range	1.5 to 150 pg (equivalent to 0.3 to 30.0 ng/mL) for both matrices	1.5 to 150 pg (equivalent to 0.3 to 30.0 ng/mL) for both matrices

	Fenpyroximate	M-3
	Corresponding calibration range 0.00075 to 0.075 mg/kg fenpyroximate in all matrices.	Corresponding calibration range 0.00075 to 0.075 mg/kg M-3 in all matrices.
Assessment of matrix effects is presented	Yes, significant in bovine muscle, not significant in milk. Matrix matched standards used	Yes, not significant in any matrices. Matrix matched standards used
Limit of determination/quantification	LOQ=0.005 mg/kg in all matrices	LOQ=0.005 mg/kg in all matrices
Comment	Acceptable against SANCO/825/00 rev 8.1 criteria. Method suitable for monitoring in food of animal origin.	Acceptable against SANCO/825/00 rev 8.1 criteria. Method suitable for monitoring in food of animal origin.

A QuEChERS based method was successfully independently validated in all food of animal origin matrix types and is compliant with SANCO/825/00 rev. 8.1. The method is appropriate for measuring concentrations of fenpyroximate or its metabolite M-3 in food of animal origin for monitoring purposes.

Methods for data generation

Validation and radiovalidation of the analytical residue method for the determination of fenpyroximate and its metabolites in cow and goat tissues and milk was conducted (Baker F.C., Bautista A.V. and Bacher R., 1999, report No. A-4039)

Milk extraction method:

The milk was extracted with acetone: water (2:1, v/v) and add the supernatant to the round bottom flask. Acidify the combined extracts with concentrated HCl and rotary evaporate to remove all the acetone. Transfer the aqueous solution to a separating funnel and wash the flask with ethyl acetate and add to the separating funnel. Partition the solution and collect the ethyl acetate (upper) layer. Repeat the partition a further two times, each time combining the ethyl acetate layers. If necessary, add NaCl to the solution to reduce any emulsion/interphase. Partition the combined ethyl acetate fractions three times with aqueous sodium carbonate.

To the ethyl acetate extract add anhydrous sodium sulfate. Pour the ethyl acetate phase through anhydrous sodium sulfate into a round bottom flask. Wash the separating funnel and sodium sulfate with ethyl acetate and combine with previously dried fraction. Rotary evaporate to dryness. Using diethyl ether, transfer the residue into a test tube. Evaporate to oil in a warm water bath. Extract the oil four times with acetonitrile, transfer the extracts to a clean test tube. Evaporate to dryness and dissolve the residue in hexane:Et_2O (9:1, v/v). Extract with SPE. Rinse the cartridge with hexane:Et_2O (9:1, v/v) and discard the eluate. Elute the cartridge with diethyl ether into a test tube, then evaporate the solvent under nitrogen. Dissolve the residue in methanol and add aqueous potassium hydroxide and mix thoroughly.

Place the test tube in a water bath at 60 °C for two hours. Evaporate the solution to remove the methanol, then add water. Partition the solution twice with hexane, discarding the hexane fractions. Acidify the solution to ~pH 1using concentrated HCl. Partition three times with diethyl ether and transfer the ether to a clean test tube. Add 0.1% acetic acid in methanol and excess diazomethane in ether to the solution and allow to stand for 10 minutes. Evaporate to dryness and dissolve the residue in hexane:Et_2O (9:1, v/v). Extract the solution by SPE and elute the cartridge with hexane:Et_2O (9:1, v/v). Evaporate the solution to dryness and dissolve the residue in acetone for GC analysis.

Muscle extraction method

The muscle was extracted with acetone:water (2:1, v/v) 2 times. Transfer the extract to a separating funnel, rinse the flask with ethyl acetate and add to the separating funnel. Partition the solution three

times, each time collecting the ethyl acetate layer. If required add NaCl during the partitioning to help break up any emulsions. Partition the ethyl acetate fraction with aqueous sodium carbonate.

Pour the ethyl acetate phase through anhydrous sodium sulfate into a round bottom flask. Wash the separating funnel with sodium sulfate and ethyl acetate and combine with previously dried fraction. Rotary evaporate to dryness. Using diethyl ether, transfer the residue into a test tube and evaporate under nitrogen to oil. Extract the oil four times with acetonitrile and transfer to a clean test tube. Evaporate to dryness and dissolve the residue in hexane:Et_2O (9:1, v/v). Extract with SPE and elute the cartridge with hexane:Et_2O (9:1, v/v) into a test tube and evaporate to dryness. Dissolve the residue in methanol, add aqueous potassium hydroxide and mix thoroughly.

Place the test tube in a water bath at 60 °C for two hours. Evaporate the solution to remove all the methanol, then add water. Partition the solution twice with hexane, discarding the hexane fractions. Acidify the solution to ~pH 1using concentrated HCl. Partition three times with diethyl ether and transfer the ether to a clean test tube. Add excess diazomethane in ether to the solution and allow to stand for 10 minutes. Evaporate to dryness and dissolve the residue in hexane:Et_2O (9:1, v/v). Extract the solution by SPE and elute the cartridge with hexane:Et_2O (9:1, v/v). Evaporate the solution to dryness and dissolve the residue in acetone for GC analysis.

Fat extraction method:

The fat was extracted twice with acetonitrile. Combine the supernatants in a round bottom flask and then evaporated to dryness. Transfer the residue into a separating funnel using rinses of hexane. Partition the solution twice with aqueous sodium carbonate or 0.1 M ammonium hydroxide and collect the aqueous layers. If an interphase layer forms, add NaCl as necessary to clarify.

Collect the hexane fraction into a flask and rinse the separating funnel with hexane, also adding to the flask. Evaporate the solution to dryness and transfer the residue to a centrifuge tube using diethyl ether. Evaporate the solution to dryness, leaving an oily residue. Dissolve the residue in hexane:ether (9:1, v/v) and extract by SPE. Rinse the flask with hexane:ether (9:1, v/v) and pass the solution through the SPE cartridge. Elute the cartridge with ether and collected into a test tube. Evaporate the solution to dryness and dissolve the residue in methanol.

Add aqueous potassium hydroxide to the solution, mix thoroughly and then place in a water bath at 60 °C for two hours. Evaporate the solution to remove the methanol and add water. Partition the solution twice with hexane, discarding the hexane fractions. Acidify the aqueous solution with concentrated HCl. Partition with diethyl ether and transfer the ether to a new test tube. Concentrate the ether to a smaller volume and add 0.1% acetic acid in methanol, followed by diazomethane in ether. Mix the solution and allow to sit for 10 minutes, then evaporated to dryness. Dissolve the residue in acetone for GC analysis.

Liver or kidney extraction method:

The liver or kidney were extracted twice with acetonitrile:water (8:2, v/v) and transfer an aliquot of the extract into a separating funnel. Acidify the aliquot with concentrated acetic acid and add NaCl. Shake the samples to separate the phases and collect the acetonitrile (upper) phase. Extract the aqueous (lower) phase with acetonitrile. Pool the two acetonitrile phases and concentrate to dryness.

Dissolve the residue in ethyl acetate and add NaCl:Na_2SO_4 (1:1, w/w) and cyclohexane. Filter the extract through a syringe membrane. Inject 5 mL of the solution into GPC and collect a fraction from 19-32 minutes. Evaporate the GPC eluate to dryness.

Dissolve the residue in diethyl ether, add diazomethane in diethyl ether and leave the solution to react at ambient temperature for one hour. Evaporate the solution to dryness under nitrogen dissolve the residue in acetonitrile (final extract). Analyse the final extract by LC/MS.

A radiovalidation study was conducted to demonstrate the effectiveness of the ‘cold’ residue method when applied to incurred [^{14}C] residues of fenpyroximate in goat milk and tissues. Goat milk and tissue samples were extracted by the methods described above and analysed by HPLC.

The method for cow milk was validated with two low level fortification samples (0.005 mg/kg), two mid-level fortifications (0.010 mg/kg) and two high level fortifications (0.100 mg/kg).

The method for cow muscle and fat was validated with two low level fortifications (0.01 mg/kg), two mid-level fortifications (0.05 mg/kg) and two high level fortifications (0.500 mg/kg).

The method for cow liver and kidney was validated with three low level fortifications (0.01 mg/kg), three mid-level fortifications (0.05 mg/kg) and three high level fortifications (0.500 mg/kg).

The method for goat milk, muscle and fat was validated with one control and two fortifications at 0.05 mg/kg and the method for goat liver and kidney was validated with one control and three fortifications at 0.05 mg/kg.

The average recoveries were within the acceptable range of 70% to 120% with the exception of the highest fortification level for cows milk, with an average recovery was 64%, cow fat, with an average recovery of 137% and goat muscle, with an average recovery of 66%. In a separate validation set the method was validated for 0.100 mg/kg fenpyroximate, with an average recovery of 118%. However the method was not validated for 0.010 mg/kg fenpyroximate in that analytical set.

The LOQ was 0.05 mg/kg in milk and 0.010 mg/kg in tissues (liver, kidney, muscle and fat).

Recovery data for fenpyroximate from cows and goats are presented in Table 153.

Table 153 Summary of results for fenpyroximate

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range (%)	RSD (%)	Number of replicates
Cow					
Milk	0.005	113	92-134	NA	2
	0.010	71	65-77	NA	2
	0.100	64	58-69	NA	2
Muscle	0.010	99	90-108	NA	2
	0.050	93	91-94	NA	2
	0.500	95	80-109	NA	2
Fat	0.010	137	117-156	NA	2
	0.050	83	69-96	NA	2
	0.500	79	64-93	NA	2
Liver	0.01	119	113-127	6.2	3
	0.05	99	98-101	1.5	3
	0.50	103	99-109	3.5	3
Kidney	0.01	118	118-118	0	3
	0.05	103	98-108	4.9	3
	0.50	101	91-106	8.3	3
Goat					
Milk	0.050	78	66-89	NA	2
Muscle	0.050	66	50-82	NA	2
Fat	0.050	71	69-72	NA	2
Liver	0.050	92	90-93	NA	2
Kidney	0.050	85	78-89	7.5	3

Methods for soil

Method for enforcement

An enforcement monitoring method for determination of residues of fenpyroximate in soil by LC-MS/MS (RES-000601) was validated with two soil types ((loamy sand, LUFA Speyer soils type 2.2 and clayey loam, 6S) for linearity, specificity precision and accuracy (Brown D., 2016, report No. A-

4092). 25g soil samples are extracted with methanol (50 mL, 20 minutes mechanical shaking). Centrifugation allows the removal of the organic extract with filtering to avoid particulates. The remaining soil is sequentially extraction by shaking with methanol (2× 50 mL), acetone (50 mL), methanol : water (1:1, v/v, 50 mL) and methanol : 0.1M hydrochloric acid (1:1, v/v, 50 mL) and the organic phases are combined and mixed. The volume is adjusted to 500 mL with water. An aliquot is removed for determination.

All samples were measured using LC-MS/MS employing an Ascentis Express C18 column, 2.7 µm particle size and monitoring at two MRM transitions. Quantification of extracts was performed using external calibration standards over the range 0.15 to 12.5 ng/mL. Matrix matched standards were used.

The lowest level tested (LOQ) was 0.01 mg/kg fenpyroximate in soil.

Table 154 Recovery results from method validation of fenpyroximate using the analytical method in soil

Matrix	Analyte	Fortification level (mg/kg), (n=x)	Range and (Mean) recovery (%)	RSD (%)	Comments
Soil (clayey loam)	fenpyroximate	0.01 (n=5)	97.7 to 101.2% (Mean 99.4%)	1.3%	MRM1 422→366
Soil (clayey loam)	fenpyroximate	0.1 (n=5)	100.6 to 103.3% (Mean 102.0%)	1.0%	MRM1 422→366
Soil (clayey loam)	fenpyroximate	Overall (n=10)	97.7 to 103.3% (Mean 100.7%)	1.7%	MRM1 422→366
Soil (clayey loam)	fenpyroximate	0.01 (n=5)	95.2 to 102.9% (Mean 98.4%)	2.9%	MRM2 422→135
Soil (clayey loam)	fenpyroximate	0.1 (n=5)	100.7 to 104.4% (Mean 102.5%)	1.7%	MRM2 422→135
Soil (clayey loam)	fenpyroximate	Overall (n=10)	95.2 to 104.4% (Mean 100.5%)	3.1%	MRM2 422→135
Soil (loamy sand)	fenpyroximate	0.01 (n=5)	96.0 to 97.4% (Mean 96.7%)	0.6%	MRM1 422→366
Soil (loamy sand)	fenpyroximate	0.1 (n=5)	97.6 to 100.1% (Mean 99.0%)	1.0%	MRM1 422→366
Soil (loamy sand)	fenpyroximate	Overall (n=10)	96.0 to 100.1% (Mean 97.8%)	1.5%	MRM1 422→366
Soil (loamy sand)	fenpyroximate	0.01 (n=5)	94.3 to 99.3% (Mean 96.3%)	3.0%	MRM2 422→135
Soil (loamy sand)	fenpyroximate	0.1 (n=5)	97.4 to 100.6% (Mean 98.7%)	1.3%	MRM2 422→135
Soil (loamy sand)	fenpyroximate	Overall (n=10)	94.3 to 100.6% (Mean 97.5%)	2.5%	MRM2 422→135

Table 155 Characteristics for the analytical method used for validation of fenpyroximate residues in soil

	Fenpyroximate
Specificity	blank value < 30% LOQ Product ion MS scan presented
Calibration (type, number of data points)	typical calibration data presented| typical calibration line equation presented linear, 1/x weighting applied r=>0.998 6 data points Matrix matched used
Calibration range	0.15 to 12.5 ng/mL Corresponding calibration range 0.003 to 0.25 mg/kg fenpyroximate in soil
Assessment of matrix effects is presented	Yes, not significant but matrix matched standards used for validation
Limit of determination/quantification	LOQ=0.01 mg/kg in soil

	Fenpyroximate
Comment	Acceptable against SANCO/825/00 rev. 8.1 criteria. Method suitable for monitoring in soil

The method was successfully validated in two soil types and is compliant with SANCO/825/00 rev. 8.1. The method is appropriate for measuring concentrations of fenpyroximate in soil for monitoring purposes with an LOQ of 0.01 mg/kg.

Methods for data generation

Method for determination of the fenpyroximate degradation in soil was validated for linearity, specificity, accuracy and precision (Römbke J. and Brodesser J., 1992, report No. E-4020). Fenpyroximate was extracted from soil with acetone:water (2:1) by shaking the sample. The extract was filtered and the organic solvent was evaporated. The remaining aqueous solution was extracted with ethyl acetate using a stirring procedure. The organic phase was evaporated to dryness and the extract was dissolved in hexane:ethyl acetate (9:1), which was applied to a silica cartridge. The analyte was eluted with hexane:ethyl acetate (4:1) and the eluate was evaporated to dryness. The extract was dissolved in acetonitrile. The analyte was determined by HPLC using a column switching method with RP-18 column of different selectivity. The analyte was detected by UV detection. The recoveries of method were 70-120% over the concentration range 0.01 to 0.2 mg/kg.

Table 156 Summary of results for fenpyroximate

Matrix	Fortification level (mg/kg)	Mean recovery (%)	Recovery range (%)	RSD (%)	Number of replicates
Fenpyroximate					
Soil	0.002	54	50-57*	5	2
	0.01	79	77-81	3	2
	0.02	77	65-89	17	2
	0.2	87	85-88	2	2

* The integration of these two samples was problematic due to matrix effects

Table 157 Characteristics for the analytical method used for validation of fenpyroximate residues in soil

	Fenpyroximate
Specificity	blank value < 30% LOQ Product ion MS scan presented
Calibration range	2.5 to 100 ng (6 calibration levels)
Limit of determination/quantification	LOQ=0.002 mg/kg in soil
Comment	Acceptable recoveries of between 70-120%

Stability of residues in stored analytical sample

Plant matrices

Hops

Storage stability of fenpyroximate (HOE 094552) and M-1 (HOE 112573) in hop (dried cones) was conducted (Weber H., 1995, report No. A-4022). 24 samples for each test substance were fortified with fenpyroximate or M-1 at 9.60 and 9.68 mg/kg, respectively and were analysed at 1 day, 3, 6, 12, 18 and 24 months after storage at ≤ -18 °C. Two storage stability samples were analysed for residues of fenpyroximate and M-1 on each date of analysis. Fortified samples used for recovery determination

were prepared at a rate of 10 mg/kg. Sample analysis was performed using GC-TID. No significant decrease of fenpyroximate was observed in the storage stability samples up to a storage period of 24 months at ≤ -18 °C in the dark. Concerning M-1, isomerisation to fenpyroximate up to a rate of 40% was observed in both M-1 fortified samples for recovery and in storage stability samples of M-1. No significant decrease of the sum of M-1 and fenpyroximate was observed in the storage stability samples up to a storage period of 24 months at ≤ -18 °C in the dark.

Table 158 Recovery of fenpyroximate in hop (dried cones) during storage stability analysis over a period of up to 24 months

Storage duration	Sample variant	Fortification level (mg/kg)	Residues of fenpyroximate (mg/kg)	Residues of M-1 (mg/kg)	Recovery of fenpyroximate (%)	Mean (%)
1 day	Recovery	10.00	8.3	n.d.	82	-
	Storage stability	9.60	8.1	n.d.	84	97.0
			10.5	n.d.	110	
3 months	Recovery	10.00	9.5	n.d.	95	-
	Storage stability	9.60	7.6	n.d.	80	84.5
			8.6	n.d.	89	
6 months	Recovery	10.00	10.4	n.d.	104	-
	Storage stability	9.60	9.2	n.d.	96	97.0
			9.4	n.d.	98	
12 months	Recovery	10.00	8.7	n.d.	87	-
	Storage stability	9.60	8.2	n.d.	85	88.0
			8.8	n.d.	91	
18 months	Recovery	10.00	12.1	n.d.	120	-
	Storage stability	9.60	7.7	n.d.	80	91.0
			9.8	n.d.	102	
24 months	Recovery	10.00	10.5	n.d.	105	-
	Storage stability	9.60	11.1	n.d.	116	108.5
			9.7	n.d.	101	

n.d.–not determinable (limit of determination for fenpyroximate and M-1=1 mg/kg)

Table 159 Recovery of M-1 in hop (dried cones) during storage stability analysis over a period of up to 24 months

Storage duration	Sample variant	Fortificat-ion level (mg/kg)	Residues of fenpyroxi-mate (mg/kg)	Residues of M-1 (mg/kg)	Recovery of M-1 (%)	Mean (%)	Recovery (sum of Fenp.+M-1) (%)	Mean (%)
1 day	Recovery	10.00	3.5	8.5	84	-	119	-
	Storage stability	9.68	2.6	6.7	69	74.5	96	104.0
			3.1	7.7	80		112	
3 months	Recovery	10.00	1.2	6.4	64	-	76	-
	Storage stability	9.68	1.4	8.4	86	88.5	100	106.5
			2.1	8.8	91		113	
6 months	Recovery	10.00	1.8	9.2	91	-	109	-
	Storage stability	9.68	1.3	7.3	76	77.5	89	95.0
			2.1	7.6	79		101	
12 months	Recovery	10.00	4.2	6.7	66	-	108	-
	Storage stability	9.68	4.5	5.7	58	60.0	104	105.0
			4.3	6.0	62		106	
18 months	Recovery	10.00	1.8	9.5	96	-	114	-
	Storage stability	9.68	2.2	8.3	86	89.5	108	108
			1.5	9.0	93		108	
24 months	Recovery	10.00	2.8	7.9	80	-	108	-
	Storage stability	9.68	4.3	6.1	63	66.0	107	104.5
			3.2	6.7	69		102	

n.d.–not determinable (limit of determination for fenpyroximate and M-1=1 mg/kg)

Apple

The storage stability of fenpyroximate and metabolite M-1 was investigated in apples (Specht W., 1992, report No. R-4136). 200 g of homogenised apples fortified either with a fenpyroximate or M-1 solution were stored for 90, 180, 360 and 540 days at -20 °C. Sample analysis was performed using HPLC with an UV-detector. The residues of fenpyroximate on day 90, 180, 360 and 540 amounted to 75%, 67%, 93% and 102%, respectively, of the value of day 0. Residues of M-1 on day 90, 180, 360 and 450 were found to be 76%, 69%, 113% and 62%, respectively, of the value of day 0. As the determination on day 360 was considered to be an outlier, a slight degradation of M-1 during the 540 days of storage was observed.

Table 160 Storage stability of fenpyroximate and its metabolite M-1 in apples during storage over a period of up to 540 days

Storage duration (days)	Fortification level (mg/kg)	Residues of fenpyroximate (mg/kg)	Residues of fenpyroximate (% day 0)	Residues of M-1 (mg/kg)
Fenpyroximate				
0	0.125	0.107	-	< 0.01
		0.120		n.d.
90	0.125	0.081	75	n.d.
		0.091		n.d.
180	0.125	0.070	67	n.d.
		0.077		n.d.
360	0.125	0.105	93	< 0.01
		0.105		n.d.
540	0.125	0.117	102	0.02
		0.110		< 0.01
Metabolite M-1				
0	0.117	< 0.01	-	0.095
		< 0.01		0.099
90	0.117	< 0.01	76	0.064
		0.01		0.083
180	0.117	0.01	69	0.063
		0.01		0.070
360	0.117	0.01	113*	0.112
		0.01		0.112
540	0.117	< 0.01	62	0.063
		< 0.01		0.060

n.d.–not detectable (limit of determination for fenpyroximate and M-1 < 0.01 mg/kg)
* considered to be an outlier

The mean procedural recoveries were within the acceptable range of 70–110%,

Storage stability of [pyrazole-^{14}C]-fenpyroximate (NNI-850) in apples was investigated as a part of metabolism study (Wyss-Benz M., 1994a, report No. A-4023). The apples were stored for approximately three years at about -20 °C. The radioactive components in washings and cake were characterised by normal and reverse phase TLC.

Due to the fact that different subsamples were compared it was not surprising to find slightly different residue levels, i.e. 0.020 mg parent equivalents/kg apples (fresh weight) in the analysis of the stability test after 3 years storage, compared to 0.031 mg/kg found in the first analysis. However, relative residue levels found, expressed in percent of the respective sample in contrast to the absolute ones, were found to be similar in both projects. Results of the analyses are summarised in Table 161.

Table 161 Comparison of residue levels of [Pyrazole-^{14}C]-fenpyroximate found in apples analysed in the course of a metabolism study and again analysed after three years of storage at -20 °C

Fraction	Metabolism study		Stability test	
	mg/kg [a]	% [b]	mg/kg [a]	% [b]
Cake				
Extracted + Washing				

Fraction	Metabolism study		Stability test	
	mg/kg [a]	% [b]	mg/kg [a]	% [b]
Fenpyroximate	0.015	48.2	0.010	50.7
M1	0.005	16.1	0.003	15.2
Unknown fraction WA3	< 0.001 [c]	----	< 0.001 [c]	----
Unknown fraction WA4	< 0.001 [c]	----	< 0.001 [c]	----
Unknown fraction in the organic phase OAC 5+6	0.002	6.4	0.001	5.1
Aqueous phase	0.005	16.1	0.003	15.2
Non-Extracted	0.001	3.6	0.001	3.7
Juice	0.003	9.6	0.002	10.1
Total ^{14}C-residues	0.031	100.0	0.020	100.0

[a] Values given in mg fenpyroximate equivalent per kg apples (fresh weight)

[b] % of total ^{14}C-residues

[c] limit of detection=0.001 mg/kg

[Pyrazole-^{14}C]-fenpyroximate and its metabolic fractions, analysed in apples in the course of a metabolism study, were found to be stable over a time period of approximately three years when samples were stored frozen at approximately -20 °C.

Grapes

Storage stability of [pyrazole-^{14}C]-fenpyroximate (NNI-850) in grapes (Wyss-Benz M., 1994b, report No.: A-4024) was investigated as part of metabolism study (Wyss-Benz & Mamouni (1992b),. The grapes were stored for approximately three years at about -20 °C. The radioactive components in washings and cake were characterised by normal and reverse phase TLC.

Due to the fact that different subsamples were compared it was not surprising to find slightly different residue levels, i.e. 0.045 mg parent equivalents/kg grapes (fresh weight) in the analysis of the stability test after 3 years storage, compared to 0.070 mg/kg found in the first analysis. In both studies the same pattern of metabolites was seen, but they were different in the residue levels obtained in the washing.

Table 162 Comparison of residue levels of [Pyrazole-^{14}C]-fenpyroximate found in grapes analysed in the course of a metabolism study and again analysed after three years of storage at -20 °C

Fraction	Metabolism study		Stability test	
	mg/kg [a]	% [b]	mg/kg [a]	% [b]
Cake				
Extracted + Washing				
Fenpyroximate	0.030	42.9	0.013	28.8
M1	0.004	5.7	0.003	6.6
Unknown fraction WG2	0.002	2.9	0.008	17.7
Unknown fraction WG3	0.015	21.4	0.003	6.6
Unknown fraction in the organic phase OGC 4	0.001	1.4	0.002	4.4
Unknown fraction in the organic phase OGC 5	0.001	1.4	< 0.001	1.1
Unknown fraction in the organic phase OGC 6	0.001	1.4	< 0.001	1.1
Aqueous phase	0.006	8.6	0.005	11.1
Non-Extracted	0.003	4.2	0.004	9.2
Juice	0.007	10.0	0.006	13.3
Total ^{14}C-residues	0.070	100.0	0.045	100.0

[a] Values given in mg fenpyroximate equivalent per kg grapes (fresh weight)

[b] % of total ^{14}C-residues

Limit of detection=0.001 mg/kg

[Pyrazole-^{14}C]-fenpyroximate and its metabolic fractions, analysed in grapes in the course of a metabolism study, were found to be stable over a time period of approximately three years when

samples were stored frozen at about -20 °C, but the surface activity was found to have changed slightly.

A reduction in the residue of fenpyroximate was observed after 3 years of storage under deep frozen conditions of >30%. Assuming a constant rate of decline in residue with time, stability of fenpyroximate is indicated for 1 year. The residue of M-1 in stored grapes was observed to be comparable at the 0 and 3 year time points.

Pome Fruit, Cotton & Grapes

The stability of Fenpyroximate in pome fruit (high water), cotton (high oil) and grapes (high acid) was summarized in US-EPA Memorandum (Kramer, G.F., 2003, report No.: R-4114 (45649901); R-4115 (45649902); R-4116 (45649903); A-4039 (45649904); A-4040 (45649905); R-4113 (45649906); R-4123 (45649913); R-4154 (45775501)). Several of the magnitude of the residue studies contained concurrent storage stability data. These data and the actual storage intervals are summarised in Table 163.

Table 163 summary of stability of stored samples of pome fruit, cotton and grapes

Matrix (RAC)	Actual Storage Duration (days)	Limits of Demonstrated Storage Stability (days)	Reference (MRID#)
Apples	36-68	None	R-4119 (45649907)
Grapes	116-190	268	R-4121 (45649908)
Cotton, undelinted seed	126-173	168	R-4117 (45649909)
Cotton, gin by-products	141-180	175	R-4117 (45649909)
Apple juice	238	None	R-4220 (45649910)
Apple wet pomace	238	None	R-4220 (45649910)
Cottonseed oil	105	112	R-4118 (45649912)
Cottonseed meal	103	104	R-4118 (45649912)
Cottonseed hulls	138	146	R-4118 (45649912)
Grape juice	158	NS	R-4122 (45649911)
Grape wet pomace	151	177	R-4122 (45649911)
Raisins	174	195	R-4122 (45649911)
Pears	14-95	100	R-4122 (45649911)

NS=Not stable

The combined residues of fenpyroximate and M-1 were found to be stable in all commodities tested except grape juice, in which a decline of >80% was observed during the course of the study.

Lettuce, sunflower, orange and potato

Storage stability of fenpyroximate and M1 metabolite under deep frozen conditions was investigated in lettuce (high water), sunflower (high oil), orange (high acid) and potato (high starch) (Gasso-Brown, D., 2017, Report No. R-4525). Samples fortified with fenpyroximate and M-1 at 0.1 mg/kg were stored in the freezer(≤ -18 °C) and analysed using EAS method 'Fenpyroximate/crops/AJW/1' for residues of fenpyroximate and M-1 at intervals of 3, 6, 9, 12 and 18 months. Recovery of fenpyroximate and M-1 from the spiked samples stored under frozen conditions are <30%, indicating adequate stability of residues under the conditions tested. Refer to Tables 164-165 for the results for fenpyroximate and M-1 respectively.

Table 164 Interim storage stability data for fenpyroximate in a range of crops

Crop	Storage period (months)	Residue (mg/kg)	Mean % Remain
Lettuce (high water)	0	0.0975 0.0973 0.0966	97
	3	0.0803 0.0797 0.0802	80

Crop	Storage period (months)	Residue (mg/kg)	Mean % Remain
	6	0.0892 0.0803 0.086	85
	9	0.0862 0.0931 0.0943	91
	12	-	-
	18	-	-
Sunflower (high oil)	0	0.093 0.0879 0.0894	90
	3	0.0977 0.093 0.0978	96
	6	0.1025 0.0888 0.0956	96
	9 12	0.0767 0.0651 0.0673 -	70 -
	18	-	-
Orange (high acid)	0	0.1022 0.1012 0.1009	101
	3	0.0736 0.0736 0.0772	75
	6	0.1165 0.113 0.1058	112
	9	0.0896 0.0911 0.0926	88
	12	-	-
	18	-	-
Potato (high starch)	0 3	0.0968 0.0897 0.0912 0.0851 0.0832 0.0863	93 85
	6	0.1027 0.1059 0.1013	103
	9	0.0917 0.0901 0.0963	93
	12	-	-
	18	-	-

Table 165: Interim storage stability data for M-1 in a range of crops

Crop	Storage period (months)	Residue (mg/kg)	Mean % Remain
Lettuce (high water)	0	0.1025 0.0753 0.0713	83
	3	0.0776 0.0785 0.0798	78
	6	0.0858 0.0770 0.0834	82
	9	0.0806 0.0886 0.0895	86
	12	-	-
	18	-	-
Sunflower (high oil)	0	0.0976 0.0882 0.0941	93
	3	0.0941 0.0941 0.0991	96
	6	0.1009 0.0859 0.0922	93
	9	0.0773 0.0668 0.0723	72
	12	-	-
	18	-	-
Orange (high acid)	0	0.1009 0.0991 0.1099	103
	3	0.0734 0.0723 0.0751	73
	6	0.1176 0.1136 0.1060	113
	9	0.0884 0.0929 0.0948	92
	12	-	-
	18	-	-
Potato (high starch)	0	0.1163 0.0845 0.0913	97
	3	0.0819 0.0799 0.0836	82
	6	0.1000 0.1025 0.0977	100
	9	0.0915 0.0892 0.0969	92
	12	-	-
	18	-	-

The available results confirm acceptable stability of fenpyroximate and M-1 in crops representative of high water, high acid, high oil and high starch crop groups. Method performance data demonstrate acceptable method performance.

Overall Conclusions for Storage Stability in Crops

A summary of the available storage stability data and the periods for which stability has been demonstrated in different matrix types is included below. Note that for lettuce, orange, sunflower and potato, the submitted results are from an ongoing study and data from later storage times out to 18 months will be available.

Table 166 Summary of storage stability in crops

Crop Matrix Type	Crops tested	Max storage stability period (months)
High water content	Apple Apple Lettuce	36 (Fen + M-1) 18 (Fen + M-1) 9 (Fen + M-1; interim)
High acid content	Grape Orange	12 (Fen), 36 (M-1) 9 (Fen + M-1; interim)
High oil content	Hops Sunflower	24 (fen + M-1) 9 (Fen + M-1; interim)
High protein content	-	-
High starch content.	Potato	9 (Fen + M-1; interim)

Animal commodities

The stored stability in animal commodities was provided in US-EPA Memorandum (Kramer, G.F., 2003, report No.: S-4027). These data and the actual storage intervals are summarised in Table 167.

Table 167 Summary storage stability in animal commodities

Matrix (RAC)	Actual Storage Duration (days)	Limits of Demonstrated Storage Stability (days)	Reference (MRID#)
Milk	73-79	79*	R-4113 (45649906)
Muscle	51-56	56#	R-4113 (45649906)
Fat	49-54	54#	R-4113 (45649906)
Liver	53	53^	R-4113 (45649906)
Kidney	55	53^	R-4113 (45649906)

* Fenpyroximate, Fen-ON, M-21

\# Fenpyroximate, Fen-OH, M-3

^ Fenpyroximate, N-desmethyl-M-3, M-3, M-22

The combined residues of fenpyroximate and M-1 were found to be stable in all commodities tested. Thus there are no storage stability issues or corrections which need to be applied to any of the magnitude of residue studies.

USE PATTERN

Fenpyroximate has been registered many countries to control mites in many crops. The information available to the Meeting on registered uses is summarized in table.

Table 168 Registered uses of fenpyroximate

Crop	Country	Formulation		Application					PHI (days)	remarks
		g a.s./L	type	Method	Rate (g ai/ha)	g ai./hL	Water (L/ha)	No		
Citrus fruit (Group 001)										
Citrus	Japan	50	SC	Foliar spray	-	2.5–5	2 000–7 000	2	14	
	USA	50	EC	Foliar spray	117–234	12.5–25	935–1,870	2	14	
	Brazil	50	SC	Foliar spray	50–120	5	1 000–2,400	3	15	
	Chile	50	SC	Foliar	-	2.5		1	14	

Crop	Country	Formulation		Application					PHI (days)	remarks
		g a.s./L	type	Method	Rate (g ai/ha)	g ai./hL	Water (L/ha)	No		
				spray						
	Greece	53.4	SC	Foliar spray	80–159	4–5.3	2 000–3 000	1	14	
	Portugal	53	SC	Foliar spray	53–80	5.3–8	1000	1	14	
	Spain	50	SC	Foliar spray	51.2–102.4	5.1–10.2	1 000–2 000	1	14	
Pome fruit (Group 002)										
Apples	Austria	51.3	SC	Foliar spray	20.4–81.6	5.1	400–1,600	1	14	
	Australia	50	SC	Foliar spray	38–88	2.5–5.9	1,500	1		
	Japan	50	SC	Foliar spray	-	2.5–5	2 000–7 000	1	14	
	Brazil	50	SC	Foliar spray	50	5	1 000	1	15	
	Portugal	53	SC	Foliar spray	53–80	5.3–8	1 000	1	14	
	Greece	53.4	SC	Foliar spray	40–106	4–5	1 000–2 000	1	7	
	Belgium	50	SC	Foliar spray	76.5	5.1	1,500	1	7	
	France	50	SC	Foliar spray	40–120	8	500–1,500	1	21	
	Germany	51.2	SC	Foliar spray	38.4	8	500–1,500	1	21	
	Poland	50	SC	Foliar spray	50– 75	7–15	500–750	2	7	
	Slovakia	50	SC	Foliar spray	50–62.5	5.1–6.25	600–1 000	1	21	
Pears	Japan	50	SC	Foliar spray	-	2.5–5	2 000–7 000	1	7	
	USA	50	EC	Foliar spray	117	12.5	936	1	14	
	Portugal	53	SC	Foliar spray	53–80	5.3–8	1 000	1	14	
	Belgium	50	SC	Foliar spray	76.5	5	1,500	1	7	
	Germany	51.2	SC	Foliar spray	38.4–115	7.68	500–1,500	1	21	
	Hungary	50	SC	Foliar spray	35–50	4–5	400–2 000	1	4	
	Poland	50	SC	Foliar spray	50	7–10	500–750	1	7	
Stone fruits (Group 003)										
Cherries	USA	50	EC	Foliar spray	117	-	-	2	7	
Peaches and apricots	USA	50	EC	Foliar spray	117	-	-	2	7	
	Europe (NEU) (Francs)	50	SC	Foliar spray	102.4	6.4	1600	1	14	
	Spain	50	SC	Foliar spray	51.2–76.8	10–12.5	1 000–1,600	1	14	
	France	50	SC	Foliar spray	102.4	6.4	1600	1	14	
Plums	USA	50	EC	Foliar spray	117	-	-	2	7	
	Europe (Germany)	51.3	SC	Foliar spray	38.4–115	7.68	500–1,500	1	21	
Small fruits (Group 004)										

Crop	Country	Formulation		Application					PHI (days)	remarks
		g a.s./L	type	Method	Rate (g ai/ha)	g ai./hL	Water (L/ha)	No		
Grapes	USA	50	EC	Foliar spray	117	25	468	2	14	
	Europe (NEU) (Germany)	51.3	SC	Foliar spray	123	7.7–30.8	400–1,600	1	35	From EU Art 12 (2016)
	Europe (SEU) (Spain)	53	SC	Foliar spray	50	5.1	1 000-1500	1	28	From EU Art 12 (2016)
	Japan	50	SC	Foliar spray	-	2.5-5	2 000–7 000	1	14	
Raspberries	USA	50	EC	Foliar spray	117	50	234	2	1	
	Europe (Austria)	51.2	SC	Foliar spray	76.5	-	-	1	14	From EU Art 12 (2016)
Strawberries	USA	50	EC	Foliar spray	117	50	234	2	1	
	Europe (Germany, Austria)	51.2	SC	Foliar spray	102	10.2	1 000	1	7	From EU Art 12 (2016)
Assorted fruits inedible peel (Group 006)										
Avocados	USA	50	EC	Foliar spray	117	25	468	2	1	
Papaya	Brazil	50	SC	Foliar spray	40	3.75–5	800	3	3	
Fruiting vegetables edible peel (Group 011)										
Cucumber/courgette	USA	50	EC	Foliar spray	117	31.2	374	2	1	
	Germany	51.2	SC	Foliar spray	46–92	7.68	600–1,200	1	3	
	Poland	50	SC	Foliar spray	15-100	0.75–33	300–2 000	2	3	
	Italy	51.3	SC	Foliar spray	-	7.7–10.3	-	1	14	
	Europe (NEU) (Germany)	50	SC	Foliar spray	46–92	7.68	600–1,200	1	3	From EU Art 12 (2016)
	Japan	50	SC	Foliar spray	-	2.5	1,500–3 000	3	1	
Fruiting vegetables inedible peel (Group 011)										
Melon	USA	50	EC	Foliar spray	117	62.5	187	2	3	
	Japan	50	SC	Foliar spray	-	2.5-5	1,500–3 000	3	1	
Cantaloupe	USA	50	EC	Foliar spray	117	62.5	187	2	3	
	Japan	50	SC	Foliar spray		2.5-5	1,500–3 000	3	1	
Watermelon	USA	50	EC	Foliar spray	117	62.5	187	2	3	
	Japan	50	SC	Foliar spray	-	2.5-5	1,500–3 000	3	1	
Fruiting vegetables other than cucurbits (Group 012)										
Peppers	USA	50	EC	Foliar spray	117	62.5	187	2	1	
	Italy	51.3	SC	Foliar spray	61.6–103	7.7–10.3	800–1 000	1	14	
	Romania	50	SC	Foliar spray	51	5.1	1 000	1	1	
	Europe (Italy)	51.2	SC	Foliar spray	102	-	-	1	14	From EU Art 12 (2016)
Tomatoes	USA	50	EC	Foliar	117	62.5	187	2	1	

Crop	Country	Formulation		Application					PHI (days)	remarks
		g a.s./L	type	Method	Rate (g ai/ha)	g ai./hL	Water (L/ha)	No		
				spray						
	Poland	50	SC	Spray	15–100	0.75–33	300–2 000	2	3	
	Romania	50	SC	Foliar spray	51	5.1	1 000	2	1	
	Italy	51.3	SC	Spray	61.6–103	7.7–10.3	800–1 000	1	14	
	Spain	50	SC	Foliar spray	51–102	5.1–10.2	1 000	1	3	
	Europe (south) (Spain)	50	SC	Foliar spray	76.8	7.68	1 000	1	7	From EU Art 12 (2016)
	Japan	50	SC	Foliar spray	-	2.5–5	1,500–3 000	3	1	
Sweet corn	USA	50	EC	Foliar spray	117	125	93.6	2	14	
	Europe	51.2	SC	Foliar spray	50	-	-	1	28	
Legume vegetables (Group 014)										
Beans	USA	50	EC	Foliar spray	117	42	280	2	1	
	Spain	50	SC	Foliar spray	102	5.1–10.2	1 000	1	7	
Root and tuber vegetables (Group 016)										
Potatoes	USA	50	EC	Foliar spray	117	31.2	187	2	7	
Cereal grains (Group 020)										
Maize	USA	50	EC	Foliar spray	117	125	93.6	2	14	
	Europe	51.2	SC	Foliar spray	50	-	-	1	28	
Nuts and seeds (Group 022 and Group 024)										
Tree nuts	USA	50	EC	Foliar spray	88–234	<6	-	2	14	
Coffee beans	Brazil	50	SC	Foliar spray	50-100	-	-	2	15	
Herbs (Group 027)										
Hops	Europe (Austria)	51.3	SC	Foliar spray	76.8–268.8	7.68	1 000–3,500	1	21	
	Japan	50	EC	Foliar spray	-	5	2 000–7 000	1	14	
Teas (Group 066)										
Tea	Japan	50	SC	Foliar spray	-	1.7–5	2 000–10 000	2	7	
	India	50	SC	Foliar spray	25	-	-	1	7	

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised trials have been conducted to support MRLs for fenpyroximate used as a pre-harvest applied acaricide in a wide range of fruit, root vegetable, cereals, oilseeds and other crops. The results of these supervised trials are summarized in the following table:

Table 169 summary of supervised tirals

Class	Commodity	Table
Citrus fruits	Lemon	170, 171
	Grapefruit	172

Class	Commodity	Table
	Orange	173, 174, 175
	Mandarin	176, 177
	Chinese Citron	178
	Natsudaidai	179
	Tangor	180
	Yuzu orange	181
Pome fruits	Apple	182-186
	Pear	187
Stone Fruits	Cherries	188
	Peach	189
	apricot	190
	Plum	191
Berries and other small fruits	Grapes	191-195
	Raspberry	196
	Strawberry	5197, 198, 199
Assorted tropical and sub-tropical fruits-inedible peel	Avocado	200
	Papaya	201
Fruiting vegetables, Cucurbits, edible peel	Cucumber	202
	Courgette	203
Fruiting vegetables, Cucurbits, inedible peel	Melon	204
	Cantaloupe	205
	Watermelon	206
Fruiting vegetables other than Cucurbits	Pepper	207
	Tomato	208, 209
	Sweet corn	210
Legume vegetables	Fresh beans with pods	210
Root and tuber vegetables	Potato	211
Cereal grain	Maize	212, 213
Nuts and seeds	Almond	214
	Walnut	215
	Pecan	216
Coffee	Coffee	217
Herbs	Hops	218
Teas	Tea	219
Animal feed	Bean forage	220
	Maize forage and stover	221
	Maize silage	222
	Almond hull	223

In many trials, especially those conducted in the USA, duplicate or multiple field samples from replicate plots were taken at each sampling period and were analysed separately, individuals and mean of these analytical results was presented. When residues were not quantifiable they are shown as below the LOQ (e.g. < 0.01 mg/kg). In some instances, residues have been determined to be below the limit of detection (LOD), in which case the notation 'ND' (Not Detected) is used. Residues, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure so as not to represent spuriously high precision. Control data are not reported in the summary tables unless residues in control samples exceeded the LOQ. Results have not been corrected for concurrent method recoveries unless indicated. Data used in estimation of MRL, HR and STMR values are underlined in the residue trials summary tables. Where higher residue values occurred at longer PHIs than the GAP, these higher values have been underlined and used in the estimation.

Citrus fruits

Lemon

Table 170 Residues in Lemons (outdoor trails) from supervised trials in USA involving foliar applications of Fenpyroximate 5% SC or EC

Lemons	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha) (GPA)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: AZ Maricopa, AZ, USA, 1994-1995 (Limonera)	235.2 + 246.4 14 day interval	4440 4710	2	14	Whole fruit	0.171,0.132 <u>0.152</u>	< 0.008, < 0.008	0.179,0.140 <u>0.160</u>	Report: R-4107 Study: AA940422 (EC)
Trial: CA1 Tulare, CA, USA, 1994-1995 (Pryor)	246.4 + 257.6 14 day interval	4794 4913	2	14	Whole fruit	0.116,0.228 <u>0.172</u>	< 0.008, < 0.008	0.124,0.230 <u>0.180</u>	Report: R-4107 Study: AA940422 (EC)
Trial: CA2 Kern, CA, USA, 1994-1995 (Lisbon)	246.4 + 257.6 14 day interval	4654 4704	2	14	Whole fruit	0.142,0.214 <u>0.178</u>	< 0.008, < 0.008	0.150,0.222 <u>0.186</u>	Report: R-4107 Study: AA940422 (EC)
Trial: 01-418-07 Porterville, CA, USA, 2001 (Prior)	448.1	2092	1	14	Whole fruit	ND, 0.092, 0.102 0.097 (sum of fenpyroximate + M-1)			Report: R-4156 Study: GR01-418 (EC)
Trial: 01-418-08 Stuart, FL, USA, 2001 (Bearss)	448.1	1474	1	14	Whole fruit	ND, 0.139, 0.109 0.124 (sum of fenpyroximate + M-1)			Report: R-4156 Study: GR01-418 (EC)
Trial: 860.1500-08-520-01C-07 Porterville, CA, USA, 2009 (Pryor)	224 + 225.1 14 day interval	1468 1405	2	14	Fruit	0.010	0.167	0.177	Report: R-4446 Study: 1872W (EC)
	230.7+ 222.9 14 day interval	1468 1405	2	14	Fruit	<u>0.017</u>	0.179	<u>0.196</u>	Report: R-4446 Study: 1872W (SC)
Trial: TCI-13-374-19 Clermont, FL, USA, 2013 (Bearss)	231.1 + 228.5 14 day interval	729 711	2	3	Fruit	0.251,0.314 0.283	0.0550,0.0732 0.064	0.306,0.3872 0.347	Report: R-4484 Study: TCI-13-374 (EC)
	225 + 226 14 day interval	1412 1421	2	3	Fruit	0.211,0.202 0.207	0.0584,0.0511 0.055	0.2694,0.2531 0.262	

Lemons	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha) (GPA)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: TCI-13-374-20 Fillmore, CA, USA, 2013 (Allen/Mac)	226.3 + 224 13 day interval	720 711	2	3	Fruit	0.348,0.300 0.324	0.0750,0.0725 0.074	0.423,0.3725 0.398	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-21 Somis, CA, USA, 2013 (Eureka)	221.8 + 222.9 14 day interval	2992 2992	2	3	Fruit	0.306,0.302 0.304	0.0624,0.0622 0.062	0.3684,0.3642 0.366	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-22 Porterville, CA, USA, 2013 (Pryor)	224 + 225.1 14 day interval	795 795	2	3	Fruit	0.316,0.278 0.297	0.0468,0.0443 0.046	0.3628,0.3222 0.343	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-23 Richgrove, CA, USA, 2013 (Lisbon)	222.9 + 224 14 day interval	1898 1898	2	3	Fruit	0.284,0.215 0.250	0.0215,0.0176 0.020	0.3055,0.2326 0.27	Report: R-4484 Study: TCI-13-374 (EC)

Table 171 Residues in Lemons (outdoor trails) from supervised trials in EU involving foliar applications of Fenpyroximate 5% SC

Lemons	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: - Lamezia Terme, Italy, 1990 (Ineapucciato)	120	1600 1600	2	21 95 145 21 95 145 21 95 145	Fruit peel Fruit peel Fruit peel Juice Juice Juice Whole fruit* Whole fruit* Whole fruit*	0.18 0.12 0.13 0.04 < 0.01 < 0.01 0.082 0.043 0.046	< 0.05 < 0.05 < 0.05 < 0.01 < 0.01 < 0.01 0.022 0.022 0.022	0.23 0.17 0.18 0.05 < 0.02 < 0.02 0.104 0.065 0.068	Report: R-4080 Study: A50252
Trial: - Lamezia Terme,Italy, 1990 (S. Teresa)	120	1600 1600	2	21 95 145 21 95 145 21 95 145	Fruit peel Fruit peel Fruit peel Juice Juice Juice Whole fruit* Whole fruit* Whole fruit*	0.16 0.09 0.10 0.02 < 0.01 < 0.01 0.062 0.034 0.037	< 0.05 < 0.05 < 0.05 < 0.01 < 0.01 < 0.01 0.022 0.022 0.022	0.21 0.14 0.15 0.03 < 0.02 < 0.02 0.084 0.056 0.059	Report: R-4081 Study: A50253
Trial: - Palicoro, Italy, 1990 (Monachello)	240	1600 1600	2	21 81 132 21 81 132 21 81 132	Fruit peel Fruit peel Fruit peel Juice Juice Juice Whole fruit* Whole fruit* Whole fruit*	0.45 0.47 0.22 0.03 < 0.01 < 0.01 0.156 0.148 0.073	< 0.05 < 0.05 < 0.05 < 0.01 < 0.01 < 0.01 0.022 0.022 0.022	0.50 0.52 0.27 0.04 < 0.02 < 0.02 0.178 0.17 0.095	Report: R-4082 Study: A50254

Lemons	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: - Lamezia Terme, Italy, 1990 (Ineapucciato)	240	1600 1600	2	21 95 145 21 95 145 21 95 145	Fruit peel Fruit peel Fruit peel Juice Juice Juice Whole fruit* Whole fruit* Whole fruit*	0.28 0.37 0.19 0.04 < 0.01 < 0.01 0.112 0.118 0.064	< 0.05 < 0.05 < 0.05 < 0.01 < 0.01 < 0.01 0.022 0.022 0.022	0.33 0.42 0.24 0.05 < 0.02 < 0.02 0.134 0.14 0.086	Report: R-4083 Study: A50255
Trial: - Lamezia Terme, Italy, 1990 (S. Teresa)	240	1600 1600	2	21 95 145 21 95 145 21 95 145	Fruit peel Fruit peel Fruit peel Juice Juice Juice Whole fruit* Whole fruit* Whole fruit*	0.29 0.32 0.27 0.01 < 0.01 < 0.01 0.094 0.103 0.088	< 0.05 < 0.05 < 0.05 - < 0.01 < 0.01 0.022 0.022 0.022	0.34 0.37 0.32 0.01 < 0.02 < 0.02 0.116 0.125 0.11	Report: R-4084 Study: A50256

Grapefruit

Table 172 Residues in Grapefruits (outdoor trails) from supervised trials in USA involving foliar applications of Fenpyroximate 5% SC or 5% EC

Grapefruits	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha) (GPA)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: 860.1500-08-520-01D-05 Haines City, FL, USA, 2009 (Ruby Red)	222.9 + 226.3 14 day interval	772 770	2	14	Fruit	0.017	0.053	0.07	Report: R-4446 Study: 1872W (EC)
	224 + 225.1 14 day interval	772 770	2	14	Fruit	< 0.01	0.076	0.077	Report: R-4446 Study: 1872W (SC)
Trial: CA5 Tulare, FL, USA, 1994-1995 (Mello Gold)	246.4 + 246.4 14 day interval	4692 4723	2	14	Whole fruit	0.057, 0.088 0.073	< 0.008	0.065, 0.096 0.081	Report: R-4107 Study: AA940422 (SC)
Trial: FL3 Palm Beach, FL, USA, 1994-1995 (White Marsh)	246.4 + 246.4 14 day interval	4621 4621	2	14	Whole fruit	0.015, 0.019 0.017	< 0.008, < 0.008	0.023,0.027 0.025	Report: R-4107 Study: AA940422 (SC)
Trial: TX1 Willacy, TX, USA, 1994-1995 (White Marsh)	246.4 + 246.4 14 day interval	4719 4704	2	14	Whole fruit	0.039, 0.039 0.039	< 0.008	0.047,0.047 0.047	Report: R-4107 Study: AA940422 (SC)
Trial: 01-418-09 Loxahatchee, FL, USA, 2001 (White Marsh)	448.1	1298	1	14	Whole fruit	ND, 0.166, 0.186 0.176(sum of fenpyroximate + M-1)			Report: R-4156 Study: GR01-418 (EC)
Trial: 01-418-10 Myakka City, FL, USA, 2001 (Ruby Red)	448.1	2254	1	14	Whole fruit	ND, 0.085, 0.118 0.102(sum of fenpyroximate + M-1)			Report: R-4156 Study: GR01-418 (EC)

Grapefruits	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha) (GPA)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: 01-418-11 Yuma, AZ, USA, 2001 (Rio Red)	459.3	1257	1	14	Whole fruit	ND, 0.065, 0.051 0.058(sum of fenpyroximate + M-1)			Report: R-4156 Study: GR01-418 (EC)
Trial: TCI-13-374-13 Clermont, FL USA, 2013 (Ray)	226.3 + 225.1 13 day interval	720 711	2	3	Fruit	0.258, 0.149 0.204	0.0514, 0.0334 0.042	0.3094, 0.1754 0.246	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-14 Oviedo, FL, USA, 2013 (Ray)	219.5 + 224 13 day interval	1384 1412	2	3	Fruit	0.180, 0.125 0.153	0.0531, 0.0331 0.043	0.2331, 0.1581 0.196	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-15 Holopaw, FL, USA, 2013 (White Marsh)	226.3 + 228.5 12 day interval	720 720	2	3	Fruit	0.275, 0.201 0.238	0.0474, 0.0309 0.039	0.3224, 0.2319 0.277	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-16 Raymondville, TX, USA, 2013 (Rio Red)	225.1 + 227.4 12 day interval	720 729	2	3	Fruit	0.0526, 0.0554 0.054	< 0.01	0.0527, 0.0555 0.055	Report: R-4484 Study: TCI-13-374 (EC)
	227.4 + 227.4 12 day interval	2403 2403	2	3	Fruit	0.104, 0.0996 0.102	0.0120, 0.0118 0.012	0.116, 0.1114 0.114	
Trial: TCI-13-374-17 Starthmore, CA, USA, 2013 (Melogold)	227.4 + 224 14 day interval	1954 1907	2	4	Fruit	0.151, 0.151 0.151	0.0247, 0.0288 0.027	0.1757, 0.1798 0.178	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-18 Porterville, CA, USA, 2013 (Melogold)	225.1 + 225.1 14 day interval	729 711	2	2	Fruit	0.168, 0.0975 0.133	0.0167, < 0.01 0.013	0.1847, 0.1075 0.146	Report: R-4484 Study: TCI-13-374 (EC)

Oranges

Table 173 Residues in Oranges (outdoor trails) from supervised trials in EU and USA involving foliar applications (once) of Fenpyroximate 5% SC or 5% EC

Oranges	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1		
Trial: Palea Epidavros Argolida, Greece, 1992 (Valencia)	154.5	3000	1	0	Pulp	< 0.01	< 0.01	< 0.02	Report: R-4144 Study: A50413 Study not to GLP * calculated value
				2	Pulp	< 0.01	< 0.01	< 0.02	
				9	Pulp	< 0.01	< 0.01	< 0.02	
				16	Pulp	< 0.01	< 0.01	< 0.02	
				22	Pulp	< 0.01	< 0.01	< 0.02	
				28	Pulp	< 0.01	< 0.01	< 0.02	
				0	Peel	0.35	< 0.01	0.36	
				2	Peel	0.37	< 0.01	0.38	
				9	Peel	0.3	< 0.01	0.31	
				16	Peel	0.26	< 0.01	0.27	
				22	Peel	0.13	< 0.01	0.14	
				28	Peel	0.18	< 0.01	0.19	
				0	Whole fruit*	0.112	< 0.01	0.122	
				2	Whole fruit*	0.118	< 0.01	0.128	
				9	Whole fruit*	0.097	< 0.01	0.107	
				16	Whole fruit*	0.085	< 0.01	0.095	
				22	Whole fruit*	0.046	< 0.01	0.056	
				28	Whole fruit*	0.061	< 0.01	0.071	
Trial: Palea Epidavros Argolida, Greece, 1992	309	3000	1	0	Pulp	< 0.01	< 0.01	< 0.02	Report: R-4145 Study: A50414 Study not to GLP * calculated value
				2	Pulp	< 0.01	< 0.01	< 0.02	
				9	Pulp	< 0.01	< 0.01	< 0.02	
				16	Pulp	< 0.01	< 0.01	< 0.02	
				22	Pulp	< 0.01	< 0.01	< 0.02	
				28	Pulp	< 0.01	< 0.01	< 0.02	
				0	Peel	0.31	< 0.01	0.32	
				2	Peel	0.28	< 0.01	0.29	
				9	Peel	0.19	< 0.01	0.20	
				16	Peel	0.24	< 0.01	0.25	
				22	Peel	0.19	< 0.01	0.20	
				28	Peel	0.23	< 0.01	0.24	
				0	Whole fruit*	0.100	< 0.01	0.110	
				2	Whole fruit*	0.091	< 0.01	0.101	
				9	Whole fruit*	0.064	< 0.01	0.074	
				16	Whole fruit*	0.079	< 0.01	0.089	
				22	Whole fruit*	0.064	< 0.01	0.074	
				28	Whole fruit*	0.076	< 0.01	0.086	
						0.247	< 0.01	0.257	
Trial: NNH/092-01 Lepe, Spain, 1998 (Salustiano)	144 Miro	2001	1	14	Pulp	< 0.01	-	< 0.01	Report: R-4108 Study: NNH092/983274
				14	Peel	0.18	-	0.18	
				14	Whole fruit	0.05	-	0.05	
	145 Kendo	2007	1	14	Pulp	< 0.01	-	< 0.01	
				14	Peel	0.16	-	0.16	
				14	Whole fruit	0.05	-	0.05	
Trial: NNH/092-02 Cantillana, Spain, 1998 (Valencia Late)	144 Miro	2006	1	14	Pulp	ND	-	ND	Report: R-4108 Study: NNH092/983274
				14	Peel	0.16	-	0.16	
				14	Whole fruit	0.04	-	0.04	
	144 Kendo	2004	1	14	Pulp	< 0.01	-	< 0.01	
				14	Peel	0.18	-	0.18	
				14	Whole fruit	0.04	-	0.04	
Trial: NNH/092-03 Catania–Misterbianco, Italy, 1998 (Tarocco Comune)	101 Miro	2000	1	30	Pulp	ND	-	ND	Report: R-4108 Study: NNH092/983274
				30	Peel	0.13	-	0.13	
				30	Whole fruit	0.04	-	0.04	
	101 Kendo	2000	1	30	Pulp	ND	-	ND	
				30	Peel	0.12	-	0.12	
				30	Whole fruit	0.04	-	0.04	

Oranges	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1		
Trial: NNH/092-04 Palagonia, Italy, 1998 (Tarocco Comune)	101 Miro	2000	1	31 31 31	Pulp Peel Whole fruit	< 0.01 0.13 0.04	- - -	< 0.01 0.13 0.04	Report: R-4108 Study: NHH092/98327 4
	101 Kendo	2000	1	31 31 31	Pulp Peel Whole fruit	< 0.01 0.11 0.04	- - -	< 0.01 0.11 0.04	
Trial: SO9-02401-01 Villanueva Castellon, Valencia, Spain, 2009 (Citrus sinensis)	106.3	1557	1	0 3 7 10 14 0 3 7 10 14 0 3 7 10 14	Pulp Pulp Pulp Pulp Pulp Peel Peel Peel Peel Peel Whole fruit* Whole fruit* Whole fruit* Whole fruit* Whole fruit*	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.10 0.14 0.11 0.14 0.11 0.037 0.049 0.040 0.049 0.040	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 0.11 0.15 0.12 0.15 0.12 0.047 0.059 0.050 0.059 0.050	Report: R-4430 Study: SO9-02401 * calculated value
Trial: SO9-02401-02 Picassent, Valencia, Spain, 2010 (Citrus sinensis)	103.5	1516	1	0 0 14 14 14 14	Pulp Peel Pulp Peel Whole fruit* Whole fruit*	0.01 < 0.01 0.29 0.20 0.094 0.067	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 < 0.02 0.30 0.21 0.104 0.077	Report: R-4430 Study: SO9-02401 * calculated value
Trial: SO9-02401-04 Fondi, Latina, Italy, 2009 (Citrus sinensis)	105.8	1551	1	0 14 0 14 0 14	Pulp Pulp Peel Peel Whole fruit* Whole fruit*	0.01 < 0.01 0.20 0.12 0.067 0.043	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 < 0.02 0.21 0.13 0.077 0.053	Report: R-4430 Study: SO9-02401 * calculated value
Trial: SO9-02401-05 Fondi, Latina, Italy, 2010 (Citrus sinensis)	98.24	1439	1	0 3 7 10 14 0 3 7 10 14 0 3 7 10 14	Pulp Pulp Pulp Pulp Pulp Peel Peel Peel Peel Peel Whole fruit* Whole fruit* Whole fruit* Whole fruit* Whole fruit*	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.22 0.29 0.20 0.13 0.17 0.073 0.094 0.067 0.046 0.058	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 0.23 0.30 0.21 0.14 0.18 0.083 0.104 0.077 0.056 0.068	Report: R-4430 Study: SO9-02401 * calculated value
Trial: S10-02525-01 La Algaba, Sevilla, Spain, 2010 (Navelina)	93.87	1375	1	0 14 0 14 0 14	Pulp Pulp Peel Peel Whole fruit Whole fruit	< 0.01 < 0.01 0.23 0.17 0.08 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 0.24 0.18 0.09 0.06	Report: R-4450 Study: S10-02525

Oranges	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1		
Trial: S10-02525-02 Alcacer, Valencia, Spain, 2010 (Navelate)	98.13	1438	1	0 3 7 10 14 0 3 7 10 14 0 3 7 10 14	Pulp Pulp Pulp Pulp Pulp Peel Peel Peel Peel Peel Whole fruit Whole fruit Whole fruit Whole fruit Whole fruit	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.24 0.18 0.16 0.15 0.21 0.07 0.05 0.05 0.05 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 0.25 0.19 0.17 0.16 0.22 0.08 0.06 0.06 0.06 0.07	Report: R-4450 Study: S10-02525
Trial: S10-02525-03 Klamata, Messinia, Greece, 2010 (Navel)	103.61	1518	1	0 14 0 14 0 14	Pulp Pulp Peel Peel Whole fruit Whole fruit	< 0.01 < 0.01 0.25 0.09 0.07 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 0.26 0.10 0.08 0.04	Report: R-4450 Study: S10-02525
Trial: S10-02525-04 Piperitsa, Messinia, Greece, 2010 (Navelina)	100.84	1477	1	0 3 7 10 14 0 3 7 10 14 0 3 7 10 14	Pulp Pulp Pulp Pulp Pulp Peel Peel Peel Peel Peel Whole fruit Whole fruit Whole fruit Whole fruit Whole fruit	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.21 0.19 0.14 0.12 0.05 0.07 0.06 0.04 0.04 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 0.22 0.20 0.15 0.13 0.06 0.08 0.07 0.05 0.05 0.03	Report: R-4450 Study: S10-02525
Trial: 01-418-01 Oviedo, FL, USA, 2001 (Navel	448.1	1690	1	0 7 14 21	Whole fruit Whole fruit Whole fruit Whole frui t	ND,0.154,0.137, 0.146 ND,0.152,0.158, 0.155 ND,0.146,0.109, 0.128 ND,0.133,0.112, 0.123 (sum of fenpyroximate + M-1)			Report: R-4156 Study: GR01-418 (EC)
Trial: 01-418-02 Myakka City, FL, USA, 2001 (Pineapple)	448.1	2251	1	14	Whole fruit	ND,0.133,0.113; 0.123 (sum of fenpyroximate + M-1)			Report: R-4156 Study: GR01-418 (EC)
	896.2	2254	1	0 0 0 0 0	Unwashed fruit Washed fruit Juice Dry pulp Oil	ND,0.165, 0.165 ND,0.195, 0.195 ND, <LOQ, < 0.02 1.229,1.478, 1.354 ND,1.042,1.132, 1.087 (sum offenpyroximate + M-1)			
Trial: 01-418-03 Myakka City, FL, USA, 2001 (Navel)	436.9	2167	1	14	Whole fruit	ND,0.093,0.163, 0.128 (sum offenpyroximate + M-1)			Report: R-4156 Study: GR01-418 (EC)
Trial: 01-418-04 Oviedo, FL, USA, 2001 (Hamlin)	448.12	1412	1	14	Whole fruit	0.313,0.311 0.312(sum of fenpyroximate + M-1)			Report: R-4156 Study: GR01-418 (EC)

Oranges	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1		
Trial: 01-418-05 Loxahatchee, FL, USA, 2001 (Hamlin)	448.12	1296	1	14	Whole fruit	ND,0.282,0.308 0.295(sum of fenpyroximate + M-1)			Report: R-4156 Study: GR01-418 (EC)
Trial: 01-418-06 Taymondville, TX, USA, 2001 (N-33)	448.1	2367	1	14	Whole fruit	0.092,0.067 0.080 (sum of fenpyroximate + M-1)			Report: R-4156 Study: GR01-418 (EC)

Table 174 Residues in Oranges (outdoor trails) from supervised trials in EU and Brazil involving foliar applications (twice) of Fenpyroximate 5% SC

Oranges	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: Palagiano, Italy, 1990 (Naveline)	77 + 77 31d interval	1000 1000	2	21 64 113 21 64 113	Pulp Pulp Pulp Peel Peel Peel Whole fruit* Whole fruit* Whole fruit* * calculated value	0.05 < 0.05 < 0.05 0.38 0.39 0.35 0.149 0.152 0.14	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.10 < 0.10 < 0.10 0.43 0.44 0.40 0.199 0.202 0.19	Report: R-4143 Study: A50245 Study not to GLP
Trial: Moncada, Spain, 1989 (Valenica Late)	149 + 160 34 day interval	5800 6200	2	1 7 13 30 44 65	Fruit Fruit Fruit Fruit Fruit Fruit	0.29 0.22 0.15 0.15 0.12 0.10	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.34 0.27 0.20 0.20 0.17 0.15	Report: R-4029 Study: A48148 Study not to GLP
Trial: Moncada, Spain, 1989 (Valenica Late)	299 + 319 34 day interval	5800 6200	2	1 7 13 30 44 65	Fruit Fruit Fruit Fruit Fruit Fruit	0.46 0.41 0.25 0.23 0.20 0.21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.51 0.46 0.30 0.28 0.25 0.26	Report: R-4030 Study: A48149 Study not to GLP
Trial: Cosmopolis, Brazil, 1989-1990 (Pera Rio)	179 + 179 30 day interval	3570 3570	2	16 29 16 29 16 29	Pulp Pulp Peel Peel Whole fruit* Whole fruit* * calculated value	< 0.05 < 0.05 0.38 0.18 0.149 0.089	< 0.05 < 0.05 0.08 0.05 0.059 0.050	< 0.10 < 0.10 0.46 0.23 0.208 0.139	Report: R-4073 Study: A45494 Study not to GLP
Trial: Cosmopolis, Brazil, 1989-1990 (Pera Rio)	357 + 357 30 day interval	3570 3570	2	16 29 16 29 16 29	Pulp Pulp Peel Peel Whole fruit* Whole fruit* * calculated value	0.08 < 0.05 0.79 0.59 0.293 0.212	< 0.05 < 0.05 0.215 0.13 0.010 0.074	0.13 < 0.10 1.005 0.72 0.303 0.286	Report: R-4074 Study: A45495 Study not to GLP

Oranges	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: Palagiano, Italy, 1990 (Washington Navel)	77 + 77 31 day interval	1000 1000	2	21 63 105 21 63 105 21 63 105	Pulp Pulp Pulp Peel Peel Peel Whole fruit* Whole fruit* Whole fruit* * calculated value	< 0.05 < 0.05 < 0.05 0.30 0.26 0.36 0.125 0.113 0.143	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.10 < 0.10 < 0.10 0.35 0.31 0.41 0.175 0.163 0.193	Report: R-4075 Study: A50246 Study not to GLP
Trial: Palagiano, Italy, 1990 (Naveline)	77 + 77 31 day interval	1000 1000	2	21 63 84 21 63 84 21 63 84	Pulp Pulp Pulp Peel Peel Peel Whole fruit* Whole fruit* Whole fruit* * calculated value	0.06 < 0.05 < 0.05 0.54 0.53 0.40 0.204 0.194 0.158	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.11 < 0.10 < 0.10 0.59 0.58 0.45 0.254 0.244 0.208	Report: R-4076, Study: A50247 Study not to GLP
Trial: Palagiano, Italy, 1990 (Naveline)	155 + 155 31 day interval	1000 1000	2	21 64 113 21 64 113 21 64 113	Pulp Pulp Pulp Peel Peel Peel Whole fruit* Whole fruit* Whole fruit* * calculated value	0.08 < 0.05 < 0.05 0.73 0.77 0.62 0.275 0.266 0.221	< 0.05 < 0.05 < 0.05 0.05 < 0.05 < 0.05 0.05 < 0.05 < 0.05	0.13 < 0.10 < 0.10 0.78 0.82 0.67 0.325 0.316 0.271	Report: R-4077 Study: A50248 Study not to GLP
Trial: Palagiano, Italy, 1990 (Washington Navel)	155 + 155 31 day interval	1000 1000	2	21 63 105 21 63 105 21 63 105	Pulp Pulp Pulp Peel Peel Peel Whole fruit* Whole fruit* Whole fruit* * calculated value	< 0.05 < 0.05 < 0.05 0.96 0.57 0.71 0.323 0.206 0.248	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.10 < 0.10 < 0.10 1.01 0.62 0.76 0.373 0.256 0.298	Report: R-4078 Study: A50249 Study not to GLP
Trial: Palagiano, Italy, 1990 (Naveline)	155 + 155 31 day interval	1000 1000	2	21 63 84 21 63 84 21 63 84	Pulp Pulp Pulp Peel Peel Peel Whole fruit* Whole fruit* Whole fruit* * calculated value	0.08 < 0.05 < 0.05 0.83 0.75 0.72 0.305 0.260 0.251	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.13 < 0.10 < 0.10 0.88 0.80 0.77 0.355 0.310 0.301	Report: R-4079 Study: A50250 Study not to GLP

Table 175 Residues in Oranges (outdoor trails) from supervised trials in USA involving foliar applications (twice) of Fenpyroximate 5% SC or 5% EC

Oranges	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: CA3 Kern, CA, USA, 1994-1995 (Atwoods)	246.4 + 246.4 14 day interval	4820 4728	2	14	Whole fruit Juice	0.124, 0.170 <u>0.147</u> < 0.008	< 0.008 < 0.008	0.132,0.178, <u>0.156</u> < 0.008	Report: R-4107 Study: AA940422 (SC)
Trial: CA4 Tulare, CA, USA, 1994-1995 (Navel)	246.4 + 246.4 14 day interval	4648 4756	2	14	Whole fruit	0.246, 0.279 <u>0.263</u>	< 0.008	<u>0.254, 0.287</u> <u>0.271</u>	Report: R-4107 Study: AA940422 (SC)
	504.1 + 492.9 14 day interval 2x rate	4739 4765	2	14	Fruit Juice Molasses Oil Dried Pulp	0.452,0.387 ,0.420 < 0.008 0.032 30.9 2.22	< 0.008 < 0.008 < 0.008 < 0.008 0.046	0.460,0.395, 0.428 < 0.016 0.040 30.908 2.266	
Trial: FL1 Palm Beach, FL, USA, 1994-1995 (Hamlin)	246.4 + 257.6 14 day interval	4621 4893	2	14	Whole fruit Juice	0.044, 0.084 <u>0.066</u> < 0.008,	< 0.008 < 0.008,	0.052,0.092 <u>0.074</u> < 0.016	Report: R-4107 Study: AA940422
Trial: FL2 Palm Beach, FL, USA, 1994-1995 (Hamlin)	224 + 246 14 day interval	4466 4616	2	14	Whole fruit	0.0695, 0.186 <u>0.128</u>	< 0.08	<u>0.0775, 0.194</u> <u>0.136</u>	Report: R-4107 Study: AA940422
	504.1 + 504.1 14 day interval	4860 4752	2	14	Whole fruit Juice Molasses Oil Dried Pulp	0.246,0.467 , 0.357 < 0.008 0.009 4.67 1.69	0.0160.021 0.019 < 0.008 < 0.008 0.220 0.146	0.264,0.489,0 .386 < 0.016 0.017 4.89 1.836	
Trial: 860.1500-08-520-01B-04 Groveland, FL, USA, 2009 (Hamlin)	222.9 + 224.1 14 day interval	794 781	2	14	Fruit	0.063	0.026	0.089	Report: R-4446, Study: 1872W (EC)
	221.8 + 220.6 14 day interval	794 781	2	14	Fruit	<u>0.132</u>	0.015	<u>0.147</u>	Report: R-4446, Study: 1872W (SC)
Trial: 860.1500-08-520-01B-06 Haines City, FL, USA, 2009 (Hamlin)	225.1 + 225.1 14 day interval	772 770	2	14	Fruit	0.039	0.122	0.161	Report: R-4446, Study: 1872W (EC)
	224 + 221.8 14 day interval	772 770	2	14	Fruit	<u>0.018</u>	0.173	<u>0.191</u>	Report: R-4446 Study: 1872W (SC)
Trial: 860.1500-08-520-01C-07 Porterville, CA, USA, 2009 (Atwood Navel)	217.3 + 224 14 day interval	1277 1294	2	14	Fruit	< 0.01	0.080	0.080	Report: R-4446, Study: 1872W (EC)

Oranges	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
	221.8 + 224.06 14 day interval	1277 1294	2	14	Fruit	0.039	0.137	0.176	Report: R-4446 Study: 1872W (SC)
Trial: 860.1500-08-520-01B-09 Delano, CA, USA, 2009 (Late Lane Navel)	225.1 + 227.4 14 day interval	1366 1387	2	14	Fruit	0.010	0.064	0.074	Report: R-4446, Study: 1872W (EC)
	225.1 + 226.3 14 day interval	1366 1387	2	14	Fruit	0.011	0.078	0.089	Report: R-4446 Study: 1872W (SC)
Trial: TCI-13-374-01 Clermont, FL, USA, 2013 (Valencia)	224 + 228.5 14 day interval	701 720	2	3	Fruit	0.170, 0.146 0.158	0.0467, 0.0363 0.042	0.2167, 0.1823 0.2	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-02 Chuluota, FL, USA, 2013 (Valencia)	225.1 + 226.3 14 day interval	1412 1412	2	2	Fruit	0.213, 0.180 0.197	0.0349, 0.0289 0.032	0.2479, 0.2089 0.229	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-03 Clermont, FL, USA, 2013 (Hamlin)	227.4 + 228.5 13 day interval	711 711	2	3	Fruit	0.247, 0.269 0.258	0.0434, 0.0491 0.046	0.2904, 0.3181 0.304	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-04 Chuluota,,FL, USA, 2013 (Hamlin)	220.6 + 225.1 13 day interval	1384 1412	2	3	Fruit	0.403, 0.383 0.393	0.0761, 0.0748 0.075	0.4791, 0.4578 0.468	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-05 Oviedo, FL, USA, 2013 (Navel)	233 + 224 13 day interval	729 701	2	3	Fruit	0.0822, 0.0693 0.076	0.0288, 0.0191 0.024	0.111, 0.0884 0.1	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-06 Winter Garden, FL, USA, 2013 (Midwest)	225.1 + 225.1 13 day interval	1412 1412	2	3	Fruit	0.323, 0.289 0.306	0.0887, 0.0728 0.081	0.4117, 0.3618 0.387	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-07 Holopaw, FL, USA, 2013 (Hamlin)	226.3 + 225.1 13 day interval	701 701	2	3	Fruit	0.389, 0.250 0.320	0.0798, 0.0635 0.072	0.4688, 0.3135 0.392	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-08 Oak Hill, FL, USA, 2013 (Hamlin)	230.7 + 228.5 13 day interval	1440 1421	2	3	Fruit	0.409, 0.424 0.417	0.0515, 0.0473 0.049	0.4605, 0.4713 0.466	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-09 Raymondville, TX, USA, 2013	227.4 + 229.6 12 day interval	720 739	2	3	Fruit	0.0578, 0.0541 0.056	0.0129, 0.0115 0.012	0.0707, 0.0656 0.068	Report: R-4484 Study: TCI-13-374

Oranges	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
(N-33 Naval)	227.4 + 228.5 12 day interval	2403 2412	2	3	Fruit	0.100, 0.0946 0.097	0.0171, 0.0129 0.024	0.1171, 0.1075 0.121	(EC)
Trial: TCI-13-374-10 Porterville, CA, USA, 2013 (Newhall Navel on Trifoliate Rootstock)	222.9 + 226.3 14 day interval	711 711	2	3	Fruit	0.183, 0.146 0.165	0.0297, 0.0267 0.028	0.2127, 0.1727 0.193	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-11 Porterville, CA, USA, 2014 (Washington)	224 + 222.9 14 day interval	1861 1879	2	3	Fruit	0.177, 0.207 0.192	0.0234, 0.0254 0.024	0.2004, 0.2324 0.216	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-12 Richgrove, CA, USA, 2013 (Valencia)	222.9 + 220.6 14 day interval	701 701	2	3	Fruit	0.127, 0.198 0.163	0.0233, 0.0489 0.036	0.1503, 0.2469 0.199	Report: R-4484 Study: TCI-13-374 (EC)
Trial: TCI-13-374-24 Porterville, CA, USA, 2013 (Cara Cara)	224 + 221.8 14 day interval	888 870	2	3	Fruit	0.155, 0.126 0.141	0.0346, 0.0270 0.031	0.1896, 0.153 0.172	Report: R-4484 Study: TCI-13-374 (EC)

Mandarin

Table 176 Residues in Mandarin (protected trails) from supervised trials in Japan involving foliar applications of Fenpyroximate 5% SC

Mandarin	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate	
Trial: Aichi Japan, 1988 (Satsuma Mandarin)	250	500 L/10a	1	7 14 21 30 44 7 14 21 30 44 7 14 21 30 44	Flesh Flesh Flesh Flesh Flesh Peel Peel Peel Peel Peel Whole Fruit* Whole Fruit* Whole Fruit* Whole Fruit* Whole Fruit*	0.006 < 0.005 0.009 0.008 0.007 0.150 0.143 0.074 0.171 0.194 0.028 0.026 0.019 0.037 0.040	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005	0.011 < 0.010 0.014 0.013 0.012 0.155 0.148 0.079 0.176 0.199 0.033 0.031 0.024 0.042 0.045	Report: R-4056 Study: Study not to GLP *Calculated value

Mandarin	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate	
Trial: Oita Japan, 1988 (Satsuma Mandarin)	500	1000 L/10a	1	7	Flesh	0.026	< 0.005	0.031	Report: R-4056 Study: Study not to GLP *Calculated value
				14	Flesh	0.021	< 0.005	0.026	
				21	Flesh	0.016	< 0.005	0.021	
				30	Flesh	0.011	< 0.005	0.016	
				45	Flesh	< 0.005	< 0.005	< 0.010	
				7	Peel	0.977	0.023	1	
				14	Peel	0.975	0.044	1.019	
				21	Peel	0.673	0.034	0.707	
				30	Peel	0.662	0.040	0.702	
				45	Peel	0.702	0.043	0.745	
				7	Whole Fruit*	0.199	< 0.005	0.204	
				14	Whole Fruit*	0.209	0.009	0.218	
				21	Whole Fruit*	0.149	0.007	0.156	
				30	Whole Fruit*	0.122	0.007	0.129	
				45	Whole Fruit*	0.130	0.008	0.138	
Trial Aichi Japan, 1988 (Satsuma Mandarin)	250	500 L/10a	1	7	Flesh	0.008	-	0.008	Report: R-4240 Study NN021-03 Study not to GLP Refer also to 5.1.3/07-10 *Calculated values
				14	Flesh	0.008	-	0.008	
				21	Flesh	0.006	-	0.006	
				30	Flesh	0.015	-	0.015	
				44	Flesh	0.009	-	0.009	
				7	Whole Fruit*	0.0492	< 0.005	0.0497	
				14	Whole Fruit*	0.0392	< 0.005	0.0397	
				21	Whole Fruit*	0.0396	< 0.005	0.0446	
				30	Whole Fruit*	0.0596	< 0.005	0.0646	
				44	Whole Fruit*	0.0454	< 0.005	0.0504	
Trial Oita Japan, 1988 (Satsuma Mandarin)	500	1000 L/10a	1	7	Flesh	0.024	-	0.024	Report: R-4240 Study NN021-03 Study not to GLP Refer also to 5.1.3/07-10 *Calculated values
				14	Flesh	0.010	-	0.010	
				21	Flesh	0.010	-	0.010	
				30	Flesh	0.015	-	0.015	
				44	Flesh	0.008	-	0.008	
				7	Whole Fruit*	0.1698	< 0.005	0.1748	
				14	Whole Fruit*	0.1236	< 0.005	0.1286	
				21	Whole Fruit*	0.1648	< 0.005	0.1698	
				30	Whole Fruit*	0.1162	< 0.005	0.1212	
				44	Whole Fruit*	0.0934	< 0.005	0.0984	
Trial Aichi Japan, 1988 (Satsuma Mandarin)	250	500 L/10a	1	7	Flesh	-	< 0.005	< 0.005	Report: R-4241 Study NN021-04 Study not to GLP Refer also to 5.1.3/07-10
				14	Flesh	-	< 0.005	< 0.005	
				21	Flesh	-	< 0.005	< 0.005	
				30	Flesh	-	< 0.005	< 0.005	
				44	Flesh	-	< 0.005	< 0.005	
Trial Oita Japan, 1988 (Satsuma Mandarin)	500	1000 L/10a	1	7	Flesh	-	< 0.005	< 0.005	Report: R-4241 Study NN021-04 Study not to GLP Refer also to 5.1.3/07-10
				14	Flesh	-	< 0.005	< 0.005	
				21	Flesh	-	< 0.005	< 0.005	
				30	Flesh	-	< 0.005	< 0.005	
				44	Flesh	-	< 0.005	< 0.005	
Trial Aichi Japan, 1988 (Satsuma Mandarin)	250	500 L/10a	1	7	Peel	0.214	-	0.214	Report: R-4243 Study NN021-07 Study not to GLP Refer also to 5.1.3/07-10
				14	Peel	0.164	-	0.164	
				21	Peel	0.174	-	0.174	
				30	Peel	0.238	-	0.238	
				44	Peel	0.191	-	0.191	

Mandarin	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate	
Trial Oita Japan, 1988 (Satsuma Mandarin)	500	1000 L/10a	1	7 14 21 30 45	Peel Peel Peel Peel Peel	0.753 0.578 0.784 0.521 0.435	- - - - -	0.753 0.578 0.784 0.521 0.435	Report: R-4243 Study NN021-07 Study not to GLP Refer also to 5.1.3/07-10
Trial Aichi Japan, 1988 (Satsuma Mandarin)	250	500 L/10a	1	7 14 21 30 44	Peel Peel Peel Peel Peel	- - - - -	0.020 0.024 0.028 0.024 0.014	0.020 0.024 0.028 0.024 0.014	Report: R-4244 Study NN021-08Study not to GLP Refer also to 5.1.3/07-10
Trial Oita Japan, 1988 (Satsuma Mandarin)	500	1000 L/10a	1	7 14 21 30 45	Peel Peel Peel Peel Peel	- - - - -	0.056 0.047 0.082 0.062 0.076	0.056 0.047 0.082 0.062 0.076	Report: R-4244 Study NN021-08Study not to GLP Refer also to 5.1.3/07-10

Table 177 Residues in Mandarin (protected trails) from supervised trials in EU involving foliar applications of Fenpyroximate 5% SC

Mandarin	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial SO9-02402-01 Beniganim, Valencia, Spain, 2009-2010 (Mandarin)	97.6	1429	1	0 0 3 3 7 7 10 10 14 14	Pulp Peel Pulp Peel Pulp Peel Pulp Peel Pulp Peel	0.02 0.41 0.01 0.36 0.01 0.33 0.01 0.26 < 0.01 0.22	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.030 0.042 0.02 0.37 0.014 0.34 0.02 0.27 < 0.02 0.23	R-4431, S09-02402 Study S09-02402
Trial SO9-02402-02 Picassent, Valencia, Spain, 2009-2010 (Mandarin)	106.3	1557	1	0 0 14 14	Pulp Peel Pulp Peel	0.02 0.28 0.02 0.29	< 0.01 < 0.01 < 0.01 0.01	0.03 0.29 0.03 0.30	R-4431, S09-02402 Study S09-02402
Trial SO9-02402-04 Fondi, Italy, 2009-2010 (Mandarin)	108.5	1589	1	0 0 14 14	Pulp Peel Pulp Peel	0.03 0.58 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.04 0.59 < 0.02 < 0.02	R-4431, S09-02402 Study S09-02402

Mandarin	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial SO9-02402-05 Fondi, Italy, 2009-2010 (Mandarin)	98.1	1438	1	0 0 3 3 7 7 10 10 14 14	Pulp Peel Pulp Peel Pulp Peel Pulp Peel Pulp Peel	0.02 0.37 0.01 0.44 < 0.01 0.19 < 0.01 0.17 < 0.01 0.15	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.03 0.38 0.02 0.45 < 0.02 0.20 < 0.02 0.18 < 0.02 0.16	R-4431, S09-02402 Study S09-02402
Trial S10-02526-01 El Viso del Alcor, Sevilla, Spain, 2009-2010 (Mandarin)	101.56	1488	1	0 14	Peel Pulp Whole fruit Peel Pulp Whole frui	0.01 < 0.01 0.34 0.20 0.10 0.05	< 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01	0.02 < 0.02 0.35 0.21 0.11 0.06	R-4451, S10-02526 Study S10-02526
Trial S10-02526-02 Picassent, Horta Sud, Spain, 2009-2010 (Mandarin)	102.28	1498	1	0 3 7 10 14 0 3 7 10 14 0 3 7 10 14	Pulp Peel Whole fruit 	0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.24 0.13 0.12 0.18 0.19 0.07 0.03 0.03 0.05 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.02 0.03 < 0.01 < 0.01 < 0.01 0.01 0.01	0.02 < 0.02 < 0.02 < 0.02 < 0.02 0.25 0.14 0.13 0.2 0.22 0.08 0.04 0.04 0.06 0.06	R-4451, S10-02526 Study S10-02526
Trial S10-02526-03 Aris, Messinia, Greece, 2009-2010 (Mandarin)	101.83	1492	1	0 14	Pulp Peel Whole fruit Pulp Peel Whole fruit	< 0.01 < 0.01 0.19 0.02 0.06 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 0.20 0.03 0.07 0.02	R-4451, S10-02526 Study S10-02526
Trial S10-02526-04 Kalamata, Messinia, Greece, 2009-2010 (Mandarin)	102.19	1497	1	0 3 7 10 14 0 3 7 10 14 0 3 7 10 14	Pulp Peel Whole fruit 	0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.20 0.11 0.14 0.07 0.02 0.08 0.03 0.05 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 < 0.02 < 0.02 < 0.02 < 0.02 0.21 0.12 0.15 0.08 0.03 0.09 0.04 0.06 0.03 0.02	R-4451, S10-02526 Study S10-02526

Chinese citron

Table 178 Residues in Chinese citron (outdoor trails) from supervised trials in Japan involving foliar applications of Fenpyroximate 5% SC

Chinese citron	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial (location): Wakayama Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 45 14 21 30 45 14 21 30 45	Flesh Flesh Flesh Flesh Peel Peel Peel Peel Whole fruit Whole fruit Whole fruit Whole fruit	0.005 < 0.005 0.005 < 0.005 0.12 0.40 0.16 0.20 0.039 0.111 0.047 0.053	- - - - - - - - - - - -	0.005 < 0.005 0.005 < 0.005 0.12 0.40 0.16 0.20 0.039 0.111 0.047 0.053	Report: R-4246 Study: NN022-01
Trial (location): Miyazaki Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 45 14 21 30 45 14 21 30 45	Flesh Flesh Flesh Flesh Peel Peel Peel Peel Whole fruit Whole fruit Whole fruit Whole fruit	< 0.005 < 0.005 < 0.005 < 0.005 0.16 0.10 0.14 0.10 0.043 0.026 0.038 0.030	- - - - - - - - - - - -	< 0.005 < 0.005 < 0.005 < 0.005 0.16 0.10 0.14 0.10 0.043 0.026 0.038 0.030	Report: R-4246 Study: NN022-01
Trial (location): Wakayama Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 44 14 21 30 44 14 21 30 44	Flesh Flesh Flesh Flesh Peel Peel Peel Peel Whole fruit Whole fruit Whole fruit Whole fruit	- - - - - - - - - - - -	< 0.005 < 0.005 < 0.005 < 0.005 < 0.01 < 0.01 0.02 0.02 < 0.005 < 0.005 0.005 0.005	< 0.005 < 0.005 < 0.005 < 0.005 < 0.01 < 0.01 0.02 0.02 < 0.005 < 0.005 0.005 0.005	Report: R-4247 Study: NN022-02
Trial (location): Miyazaki Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 45 14 21 30 45 14 21 30 45	Flesh Flesh Flesh Flesh Peel Peel Peel Peel Whole fruit Whole fruit Whole fruit Whole fruit	- - - - - - - - - - - -	< 0.005 < 0.005 < 0.005 < 0.005 < 0.01 < 0.01 < 0.01 < 0.01 < 0.005 < 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005 < 0.005 < 0.01 < 0.01 < 0.01 < 0.01 < 0.005 < 0.005 < 0.005 < 0.005	Report: R-4247 Study: NN022-02
Trial (location): Wakayama Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 45	Peel Peel Peel Peel	0.12 0.40 0.16 0.20	- - - -	0.12 0.40 0.16 0.20	Report: R-4249 Study: NN022-05
Trial (location): Miyazaki Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 45	Peel Peel Peel Peel	0.16 0.10 0.14 0.10	- - - -	0.16 0.10 0.14 0.10	Report: R-4249 Study: NN022-05

Chinese citron	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial (location): Wakayama Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 45	Peel Peel Peel Peel	- - - -	< 0.01 0.01 0.02 0.02	< 0.01 0.01 0.02 0.02	Report: R-4250 Study: NN022-06
Trial (location): Miyazaki Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 45	Peel Peel Peel Peel	- - - -	< 0.01 < 0.01 0.01 0.01	< 0.01 < 0.01 0.01 0.01	Report: R-4250 Study: NN022-06
Trial (location): Wakayama Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 45	Peel Peel Peel Peel	0.34 0.32 0.28 0.26	- - - -	0.34 0.32 0.28 0.26	Report: R-4252 Study: NN022-07
Trial (location): Miyazaki Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 45	Peel Peel Peel Peel	0.14 0.25 0.06 0.24	- - - -	0.14 0.25 0.06 0.24	Report: R-4252 Study: NN022-07
Trial (location): Wakayama Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 45	Peel Peel Peel Peel	- - - -	< 0.04 < 0.04 < 0.04 < 0.04	< 0.04 < 0.04 < 0.04 < 0.04	Report: R-4253 Study: NN022-08
Trial (location): Miyazaki Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 45	Peel Peel Peel Peel	- - - -	< 0.04 < 0.04 < 0.04 < 0.04	< 0.04 < 0.04 < 0.04 < 0.04	Report: R-4253 Study: NN022-08
Trial (location): Wakayama Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 45 14 21 30 45 14 21 30 45	Flesh Flesh Flesh Flesh Peel Peel Peel Peel Whole fruit Whole fruit Whole fruit Whole fruit	< 0.005 < 0.005 < 0.005 < 0.005 0.34 0.32 0.28 0.26 0.09 0.09 0.08 0.07	- - - - - - - - - - - -	< 0.005 < 0.005 < 0.005 < 0.005 0.34 0.32 0.28 0.26 0.09 0.09 0.08 0.07	Report: R-4256 Study: NN022-03
Trial (location): Miyazaki Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 45 14 21 30 45 14 21 30 45	Flesh Flesh Flesh Flesh Peel Peel Peel Peel Whole fruit Whole fruit Whole fruit Whole fruit	< 0.005 < 0.005 < 0.005 < 0.005 0.14 0.25 0.06 0.24 0.04 0.06 0.02 0.07	- - - - - - - - - - - -	< 0.005 < 0.005 < 0.005 < 0.005 0.14 0.25 0.06 0.24 0.04 0.06 0.02 0.07	Report: R-4256 Study: NN022-03
Trial (location): Wakayama Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 45	Flesh Flesh Flesh Flesh	- - - -	< 0.005 < 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005 < 0.005	Report: R-4257 Study: NN022-04
Trial (location): Miyazaki Japan, 1988 (Kawanonatsudaidai)	250	500 L/10a	1	14 21 30 45	Flesh Flesh Flesh Flesh	- - - -	< 0.005 < 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005 < 0.005	Report: R-4257 Study: NN022-04

Natsudaidai

Table 179 Residues in Natsudaidai (outdoor trails) from supervised trials in Japan involving foliar applications of Fenpyroximate 5% SC

Natsudaidai	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial (location): Wakayama Japan, 1988 (Kawano natsudaidai)	250	5000	1	14 21 30 45 14 21 30 45 14 21 30 45	Flesh Flesh Flesh Flesh Peel Peel Peel Peel Whole fruit* Whole fruit* Whole fruit* Whole fruit*	0.005 < 0.005 0.005 < 0.005 0.12 0.40 0.16 0.20 0.040 0.124 0.052 0.064	< 0.005 < 0.005 < 0.005 < 0.005 < 0.01 0.01 0.02 0.02 0.007 0.007 0.010 0.010	0.010 < 0.010 0.010 < 0.010 0.13 0.42 0.18 0.22 0.047 0.131 0.062 0.074	Report: R-4057 Study: -
Trial (location): Miyazaki Japan, 1988 (Amanatsu)	250	5000	1	14 21 30 45 14 21 30 45 14 21 30 45	Flesh Flesh Flesh Flesh Peel Peel Peel Peel Whole fruit* Whole fruit* Whole fruit* Whole fruit*	< 0.005 < 0.005 < 0.005 < 0.005 0.16 0.10 0.14 0.10 0.052 0.034 0.046 0.034	< 0.005 < 0.005 < 0.005 < 0.005 < 0.01 < 0.01 0.01 0.01 0.007 0.007 0.007 0.007	< 0.010 < 0.010 < 0.010 < 0.010 0.17 0.11 0.15 0.11 0.059 0.041 0.053 0.041	Report: R-4057 Study: -

Tangor

Table 180 Residues in Tangor tree (outdoor trails) from supervised trials in EU involving foliar applications of Fenpyroximate 5% SC

Tangor tree	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: - Massafra, Italy, 1991 (common tangor)	100	1600	1	28 28 28	Pulp Fruit peel Whole fruit*	0.01 0.35 0.112	< 0.01 0.02 0.013	0.02 0.37 0.125	Report: R-4085 Study: A50291
Trial: - Massafra, Italy, 1991 (common tangor)	200	1600	1	28 28 28	Pulp Fruit peel Whole fruit*	0.03 0.78 0.255	< 0.01 0.03 0.016	0.04 0.81 0.271	Report: R-4086 Study: A50292
Trial: - Poucoro, Italy, 1991 (common tangor)	100	1600	1	28 28 28	Pulp Fruit peel Whole fruit*	< 0.01 0.42 0.133	< 0.01 < 0.01 0.010	< 0.02 0.43 0.143	Report: R-4087 Study: A50293
Trial: - Poucoro, Italy, 1991 (common tangor)	200	1600	1	28 28 28	Pulp Fruit peel Whole fruit*	0.03 0.86 0.279	< 0.01 < 0.01 0.010	0.04 0.87 0.289	Report: R-4088 Study: A50294

Tangor tree	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: - Mctaponto, Italy, 1991 (common tangor)	100	1600	1	0	Pulp	0.03	< 0.01	0.04	Report: R-4089 Study: A50295
				5	Pulp	0.02	< 0.01	0.03	
				10	Pulp	< 0.01	< 0.01	< 0.02	
				14	Pulp	< 0.01	< 0.01	< 0.02	
				25	Pulp	0.02	< 0.01	0.03	
				28	Pulp	< 0.01	< 0.01	< 0.02	
				0	Fruit peel	0.52	0.02	0.54	
				5	Fruit peel	0.50	0.02	0.52	
				10	Fruit peel	0.36	< 0.01	0.37	
				14	Fruit peel	0.34	< 0.01	0.35	
				25	Fruit peel	0.24	< 0.01	0.25	
				28	Fruit peel	0.13	0.01	0.14	
				0	Whole fruit*	0.177	0.013	0.19	
				5	Whole fruit*	0.164	0.013	0.177	
				10	Whole fruit*	0.115	0.010	0.125	
				14	Whole fruit*	0.109	0.010	0.119	
				25	Whole fruit*	0.086	0.010	0.096	
				28	Whole fruit*	0.046	0.010	0.056	
Trial: - Mctaponto, Italy, 1991 (common tangor)	200	1600	1	0	Pulp	0.05	< 0.01	0.06	Report: R-4090 Study: A50296
				5	Pulp	0.06	< 0.01	0.07	
				10	Pulp	0.03	< 0.01	0.04	
				14	Pulp	0.03	< 0.01	0.04	
				25	Pulp	0.02	< 0.01	0.03	
				28	Pulp	0.01	< 0.01	0.02	
				0	Fruit peel	0.45	< 0.01	0.46	
				5	Fruit peel	0.63	< 0.01	0.64	
				10	Fruit peel	0.57	0.04	0.61	
				14	Fruit peel	0.83	0.03	0.86	
				25	Fruit peel	0.59	0.03	0.62	
				28	Fruit peel	0.59	0.03	0.62	
				0	Whole fruit*	0.170	0.010	0.18	
				5	Whole fruit*	0.231	0.010	0.241	
				10	Whole fruit*	0.192	0.019	0.211	
				14	Whole fruit*	0.270	0.016	0.286	
				25	Whole fruit*	0.191	0.016	0.207	
				28	Whole fruit*	0.184	0.016	0.2	

Yuzu orange

Table 181 Residues in Yuzu Oranges (outdoor trails) from supervised trials in Japan involving foliar applications of Fenpyroximate 5% SC

Yuzu Oranges	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: Oita Japan, 1990 (Citrus junos)	250		1	14	Fruit	0.06	-	0.06	Report: R-4352 Study: NN029-01 Study not to GLP Refer also to 5.1.2/17
				14	Fruit	0.04	-	0.04	
				28	Fruit	0.02	-	0.02	
				28	Fruit	0.02	-	0.02	
				56	Fruit	0.01	-	0.01	
				98	Fruit	< 0.01	-	< 0.01	

Yuzu Oranges	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: Oita Japan, 1990 (Citrus junos)	250	500 L/10a	1	14 14 28 28 56 98	Fruit Fruit Fruit Fruit Fruit Fruit	- - - - - -	0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	Report: R-4353 Study: NN029-02 Study not to GLP Refer also to 5.1.2/16
Trial: Oita Prefectural Japan, 1990 (Citrus junos)	250	500 L/10a	1	14 28 88 14 28 56	Fruit Fruit Fruit Fruit Fruit Fruit	< 0.05 0.04 < 0.01 0.04 0.03 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.06 0.05 < 0.02 0.05 0.04 0.02	Report: R-4354 Study: NN029-03 Study not to GLP

Pome fruits

Apple

Table 182 Residues in Apples (outdoor trails) from supervised trials in EU involving foliar applications (once) of Fenpyroximate 5% SC

Apples	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial:-BR 9113 Belgium, 1991	90		1	0 1 3 7 14 21 28	Fruit Fruit Fruit Fruit Fruit Fruit Fruit	0.14 0.09 0.12 0.12 0.10 0.08 0.05	- - < 0.02 < 0.02 < 0.02 - -	0.14 0.09 0.14 0.14 0.12 0.08 0.05	Report: R-4042e Study:- RE-9205-H
	180		1	0 1 3 7 14 21 28	Fruit Fruit Fruit Fruit Fruit Fruit Fruit	0.17 0.15 0.21 0.19 0.17 0.14 0.18	- - < 0.02 < 0.02 < 0.02 - -	0.17 0.15 0.23 0.21 0.19 0.14 0.18	
Trial: S 210.91 Grossdeuvre, France, 1991 (Golden)	80	1000	1	30 50 75 106 120 144	Fruit Fruit Fruit Fruit Fruit Fruit	0.03 0.03 < 0.02 < 0.02 < 0.02 < 0.02	- - - - - -	0.03 0.03 < 0.02 < 0.02 < 0.02 < 0.02	Report: R-4047f Study:- RE-9117-H (EC)
Trial:- 0101 Winsen-Hoopte, Germany, 1990 (Gloster)	112.5	1500	1	0 7 14 28 42 56 70 84 91	Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit	0.13 0.14 0.11 0.08 0.06 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.18 0.19 0.16 0.13 0.11 < 0.10 < 0.10 < 0.10 < 0.10	Report: R-4072e, ER90DEU812, A47895, A49849 Study:-

Apples	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial:- 0401 Höchst, Germany, 1990 (Idared)	64	854	1	0 7 14 28 42 56 70 82 93	Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit	0.15 0.11 0.16 0.12 0.09 0.06 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.20 0.16 0.21 0.17 0.14 0.11 < 0.10 < 0.10 < 0.10	Report: R-4072e, ER90DEU812, A49849 Study:-
Trial:- Potamaia-Larissa-Thessalta, Greece, 1992 (Imperial)	75	1500	1	0 3 7 14 21 30	Fruit Fruit Fruit Fruit Fruit Fruit	0.04 0.05 0.04 0.04 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.05 0.06 0.05 0.05 0.04 0.03	Report: R1-4097e, A50032 Study:- Study not to GLP
Trial:- UKPVB1 Staplehurst, Kent., UK, 1993 (Cox)	5 g ai/hL Spray to runoff		1	14	Whole fruit	0.03	< 0.01	0.04	Report: R-4099e, A53347 Study: RESID/94/20
Trial:- UKPVB2 Gorefield, Cambs., UK, 1993 (Bramley)	5 g ai/hL Spray to runoff		1	14	Whole fruit	0.01	< 0.01	0.02	Report: R-4099e, A53347 Study: RESID/94/20
Trial:- UKPVB3 Alresford, Essex., UK, 1993 (Cox)	5 g ai/hL Spray to runoff		1	14	Whole fruit	0.07	< 0.01	0.08	Report: R-4099e, A53347 Study: RESID/94/20
Trial:- UKPVB4 Harrietsham, Kent., UK, 1993 (Cox)	5 g ai/hL Spray to runoff		1	14	Whole fruit	0.07	< 0.01	0.08	Report: R-4099e, A53347 Study: RESID/94/20
5.2.1/18 Report: R-4099e, A53347 Study: RESID/94/20 Trial:- UKPVB5 Ridgewell, Essex., UK, 1993 (Cox)	5 g ai/hL Spray to runoff		1	14	Whole fruit	0.03	< 0.01	0.04	Report: R-4099e, A53347 Study: RESID/94/20
Trial:- S09-2263-01 Blumberg-Elisenau, Brandenburg, Germany, 2009 (Jonagored)	115.7	502	1	0 21	Fruit Fruit	0.17 0.07	< 0.01 < 0.01	0.18 0.08	Report: R4429e Study: S09-02263
Trial:- S09-2263-03 Bages, Lyienèes Chientales, France, 2009 (Fuji)	126.0	875	1	0 20	Fruit Fruit	0.14 0.07	< 0.01 < 0.01	0.15 0.08	Report: R4429e Study: S09-02263

Apples	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial:- S09-2263-04 Calatorao, Aragon, Spain, 2009 (Starking)	125.5	1089	1	0 3 7 14 21	Fruit Fruit Fruit Fruit Fruit	0.13 0.11 <u>0.12</u> 0.08 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.14 0.12 <u>0.13</u> 0.09 0.09	Report: R4429e Study: S09-02263
Trial:- S09-2263-05 Chilleurs Aux Bois, Loiret, France,, 2009 (Golden)	115.2	800	1	0 21	Fruit Fruit	0.08 0.03	< 0.01 < 0.01	0.09 0.04	Report: R4429e Study: S09-02263
Trial:- S09-2263-06 Innenheim, Alsace, France, 2009 (Braeburn)	111.2	965	1	0 3 7 14 21	Fruit Fruit Fruit Fruit Fruit	0.07 0.07 0.04 <u>0.06</u> 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.08 0.08 0.05 <u>0.07</u> 0.06	Report: R4429e Study: S09-02263
Trial:- S09-2263-07 Moissac, Tarn et Garonne, France, 2009 (Braeburn)	115.7	602	1	0 21	Fruit Fruit	0.05 0.05	< 0.01 < 0.01	0.06 0.06	Report: R4429e Study: S09-02263
Trial:- S09-2263-08 Barboles, Spain, 2009 (Golden)	108.9	945	1	0 3 7 14 21	Fruit Fruit Fruit Fruit Fruit	0.06 0.09 <u>0.11</u> 0.07 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.07 0.10 <u>0.12</u> 0.08 0.06	Report: R4429e Study: S09-02263
Trial:- S09-2263-09 Engingen, Baden Wusttenberg, Germany, 2009 (Fuji)	116.2	706	1	0 3 7 14 21	Fruit Fruit Fruit Fruit Fruit	0.08 0.09 0.06 <u>0.07</u> 0.07*	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.09 0.10 0.07 <u>0.08</u> 0.08*	Report: R4429e Study: S09-02263 * Control contained 0.06 mg/kg
Trial:- S11-03148-01 Altlandsberg, Brandenburg, Germany, 2011 (Idared)	112.5	1000	1	21	Fruit	0.03	< 0.01	0.04	Report: R4472e Study: S11-03148
Trial:- S11-03148-02 Hechthausen, Niedersachsen, Germany, 2011 (Jonagored)	111.9	995	1	0 3 7 14 21	Fruit Fruit Fruit Fruit Fruit	0.07 0.14 <u>0.09</u> 0.06 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.08 0.15 <u>0.10</u> 0.07 0.05	Report: R4472e Study: S11-03148
Trial:- S11-03148-03 Vallères, Indre-et-Loire, France, 2011 (Pink lady)	116.0	722	1	21	Fruit	0.12	< 0.01	0.13	Report: R4472e Study: S11-03148
Trial:- S11-03148-04 Innenheim, Alsace, France, 2011 (Braeburn)	112.0	697	1	0 3 7 14 21	Fruit Fruit Fruit Fruit Fruit	0.09 0.11 0.10 <u>0.11</u> 0.10	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.10 0.12 0.11 <u>0.12</u> 0.11	Report: R4472e Study: S11-03148

Apples	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial:- S11-03149-01 Plasencia de Jalòn Spain, 2011 (Golden)	121.0	1079	1	21	Fruit	0.05	< 0.01	0.06	Report: R4473e Study: S11-03149
Trial:- S11-03149-02 Calatorao, Spain, 2011 (Fuji)	110.0	977	1	0 3 7 14 21	Fruit Fruit Fruit Fruit Fruit	0.09 0.08 0.07 0.06 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.10 0.09 0.08 0.07 0.05	Report: R4473e Study: S11-03149
Trial:- S11-03149-03 Drosero, Pella, Greece, 2011 (Granny Smith)	119.0	845	1	21	Fruit	0.03	< 0.01	0.04	Report: R4473e Study: S11-03149
Trial:- S11-03149-04 Budrio, Emilia-Romagna, Italy, 2011 (Fuji)	111.0	987	1	0 3 7 14 21	Fruit Fruit Fruit Fruit Fruit	0.08 0.09 0.08 0.05 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.09 0.10 0.09 0.06 0.05	Report: R4473e Study: S11-03149
Trial: AF/11088/NN/4 Götzdorf, Lower Saxony, Germany, 2006 Apple (Elstar)	119.7	1490	1	42 56	Fruit Fruit	0.01 0.01	< 0.01 < 0.01	0.02 0.02	Report: R-4185 Study:
Jork, Gehrdem, Lower Saxony, Germany, 2006 Apple (Gloser)	119.4	1487	1	0 14 21 42 56	Fruit Fruit Fruit Fruit Fruit	0.09 0.09 0.06 0.04 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.10 0.10 0.07 0.05 0.05	Report: R-4185 Study:
Trial: AD/6100/NN/3 Malalbergo, Emila Romagna, Italy, 2001 Apple (Fuji)	72.4	1476	1	0 3 7 10 14	Fruit Fruit Fruit Fruit Fruit	0.04 0.02 0.02 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.05 0.03 0.03 0.02 < 0.02	Report: R-4161 Study:
Trial: AD/6100/NN/4 Tintoria, Emila Romagna, Italy, 2001 Apple (Double Red)	70.1	1401	1	10 14	Fruit Fruit	0.01 0.02	< 0.01 < 0.01	0.02 0.03	Report: R-4161 Study:
Trial: AF/11088/NN/1 Sigloy, N-France, 2006 Apple (Canada)	83.2	1035	1	42 56	Fruit Fruit	0.02 0.01	< 0.01 < 0.01	0.03 0.02	Report: R-4185 Study:
Trial: AF/11088/NN/2 Chilleurs-aux-Bois, N-France, 2006 Apple (Golden)	79.8	993	1	0 14 21 42 56	Fruit Fruit Fruit Fruit Fruit	0.04 0.05 0.04 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.05 0.06 0.05 0.04 0.03	Report: R-4185 Study:

Apples	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: AD/6100/NN/1 Castelsarrasin, Tarn-et-Garonne, S-France, 2001 Apple (Granny Smith)	74	1480	1	0 3 7 10 14	Fruit Fruit Fruit Fruit Fruit	0.05 0.05 0.04 0.04 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.06 0.06 0.05 0.05 0.03	Report: R-4161 Study:

Table 183 Residues in Apples (outdoor trails) from supervised trials in EU involving foliar applications (twice) of Fenpyroximate 5% SC

Apples	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial:- S 201.89 France, 1989	6 + 6		2	53	Fruit	0.07	< 0.02	0.09	Report: R-4046e Study:- RE-9019-H
	8 + 8		2	53	Fruit	0.03	< 0.02	0.05	
Trial:- S 202.89 France, 1989	6 + 6		2	69	Fruit	0.03	< 0.02	0.05	Report: R-4046e Study:- RE-9019-H
	8 + 8		2	69	Fruit	0.04	< 0.02	0.06	
Trial:- S 302.89 France, 1989	6 + 6		2	48	Fruit	0.05	< 0.02	0.07	Report: R-4046e Study:- RE-9019-H
	8 + 8		2	48	Fruit	0.08	< 0.02	0.10	
Trial:- S 361.80 France, 1989	6		2	0 7 14 21 29	Fruit Fruit Fruit Fruit Fruit	0.10 0.08 0.03 0.02 0.03	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.12 0.10 0.05 0.04 0.05	Report: R-4046e Study:- RE-9019-H
Trial: S 302.90 France, 1990	8		2	0 7 14 21 29	Fruit Fruit Fruit Fruit Fruit	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.04 < 0.04 < 0.04 < 0.04 < 0.04	Report: R-4046e Study:- RE-9019-H
Trial: S 303.90 France, 1990	8		2	0 7 14 21 29	Fruit Fruit Fruit Fruit Fruit	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.04 < 0.04 < 0.04 < 0.04 < 0.04	Report: R-4046e Study:- RE-9019-H
Trial: S 217.90 France, 1990	6 + 6		2	68	Fruit	< 0.02	< 0.02	< 0.04	Report: R-4046e Study:- RE-9019-H
	8 + 8		2	68	Fruit	< 0.02	< 0.02	< 0.04	
Trial: S 344.90 France, 1990	6 + 6		2	24	Fruit	< 0.02	< 0.02	< 0.04	Report: R-4046e Study:- RE-9019-H
	8 + 8		2	24	Fruit	< 0.02	< 0.02	< 0.04	
Trial: S 344.90 France, 1990	6 + 6		2	45	Fruit	< 0.02	< 0.02	< 0.02	Report: R-4046e Study:- RE-9019-H
	8 + 8		2	45	Fruit	< 0.19	< 0.19	< 0.38	

Apples	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial:-DEU 89 I 801/11 Drage-Elbstorf, Germany, 1989 (Golden Delicious)	112.5 + 112.5 28 day interval	1505 1505	2	0 7 14 21	Fruit Fruit Fruit Fruit	0.10 0.12 0.11 0.10	ND ND ND ND	0.10 0.12 0.11 0.10	Report: R-4012g, A51901 Study:-
Trial:-DEU 89 I 801/21 Bornheim-Roisdorf, Germany, 1989 (James Grive)	112.5 + 112.5 20 day interval	1500 1500	2	0 7 14 21	Fruit Fruit Fruit Fruit	0.19 0.18 0.10 0.09	0.01 0.01 0.01 ND	0.2 0.19 0.11 0.09	Report: R-4012g, A51901 Study:-
Trial:-DEU 89 I 801/31 Sauters Germany, 1989 (Gloster)	64.3 + 86.8 28 day interval	857 1194	2	0 7 14 21	Fruit Fruit Fruit Fruit	0.12 0.12 0.08 0.11	ND 0.01 0.01 0.01	0.12 0.13 0.09 0.12	Report: R-4012g, A51901 Study:-
Trial:-DEU 89 I 801/41 Ffm./Hoechst, Germany, 1989 (Jonathan)	100 + 115 29 day interval	1330 1520	2	0 7 14 21	Fruit Fruit Fruit Fruit	0.21 0.19 0.15 0.16	ND 0.01 0.01 0.01	0.21 0.2 0.16 0.17	Report: R-4012g, A51901 Study:-
Trial:-DEU 89 I 811/11 Dorage-Elbstorf, Germany, 1989 (Golden Delicious)	150 + 150 28 day interval	1505 1505	2	0 7 14 21	Fruit Fruit Fruit Fruit	0.24 0.23 0.24 0.24	0.02 0.01 0.02 0.02	0.26 0.24 0.26 0.26	Report: R-4016g, A51902 Study:-
Trial:-DEU 89 I 811/31 Sauters, Germany, 1989 (Gloster)	95.3 + 132.2 28 day interval	952 1321	2	0 7 14 21	Fruit Fruit Fruit Fruit	0.12 < 0.01 0.12 0.12	< 0.01 < 0.01 0.01 0.01	0.13 < 0.02 0.13 0.13	Report: R-4016g, A51902 Study:-
Trial:- 0101 Winsen-Hoopte, Germany, 1990 (Gloster)	112.5 + 112.5 27 day interval	1500 1000	2	0 7 14 21 28	Fruit Fruit Fruit Fruit Fruit	0.21 0.17 0.13 0.08 0.11	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.26 0.22 0.18 0.13 0.16	Report: R-4071e, A49847 Study:-
Trial:- 0102 Born-heim-Roisdorf, Germany, 1990 (Cox Orange)	75 + 75 26 day interval	1000 1000	2	0 7 14 21 28 21 21 21 21 21	Fruit Fruit Fruit Fruit Fruit Cider Pomace Marc Washings (cider) Washings (pomace)	0.13 0.08 0.08 0.06 < 0.05 < 0.05 < 0.05 0.13 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.18 0.13 0.13 0.11 < 0.10 < 0.10 < 0.10 0.18 < 0.10 < 0.10	Report: R-4071e, A49847 Study:-
Trial:- 0301 Sauters-Lindau, Germany, 1990 (Roter Boskop)	114.5 + 114.5 27 day interval	1500 1500	2	0 7 14 21 28	Fruit Fruit Fruit Fruit Fruit	0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.10 < 0.10 < 0.10 < 0.10 < 0.10	Report: R-4071e, A49847 Study:-

Apples	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial:- 0401 F.-Höchst, Germany, 1990 (Alkmene)	81 + 81 28 day interval	1083 1083	2	0 7 14 21 28 21 21 21 21 21	Fruit Fruit Fruit Fruit Fruit Cider Pomace Marc Washings (cider) Washings (pomace)	0.24 0.21 0.18 0.15 0.13 < 0.05 < 0.05 0.39 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 0.05 < 0.05 < 0.05	0.29 0.26 0.23 0.20 0.18 < 0.10 < 0.10 0.44 < 0.10 < 0.10	Report: R-4071e, A49847 Study:-
Trial:- UKPVB1 Staplehurst, Kent., UK, 1993 (Cox)	2.5 +2.5 g ai/hL Spray to runoff		2	14	Whole fruit	0.02	< 0.01	0.03	Report: R-4099e, A53347 Study: RESID/94/20
Trial:- UKPVB2 Gorefield, Cambs., UK, 1993 (Bramley)	2.5 +2.5 g ai/hL Spray to runoff		2	14	Whole fruit	< 0.01	< 0.01	< 0.02	Report: R-4099e, A53347 Study: RESID/94/20
Trial:- UKPVB3 Alresford, Essex., UK, 1993 (Cox)	2.5 +2.5 g ai/hL Spray to runoff		2	14	Whole fruit	0.03	< 0.01	0.04	Report: R-4099e, A53347 Study: RESID/94/20
Trial:- UKPVB4 Harrietsham, Kent., UK, 1993 (Cox)	2.5 +2.5 g ai/hL Spray to runoff		2	14	Whole fruit	0.03	< 0.01	0.04	Report: R-4099e, A53347 Study: RESID/94/20
5.2.1/18 Report: R-4099e, A53347 Study: RESID/94/20 Trial:- UKPVB5 Ridgewell, Essex., UK, 1993 (Cox)	2.5 +2.5 g ai/hL Spray to runoff		2	14	Whole fruit	0.03	< 0.01	0.04	Report: R-4099e, A53347 Study: RESID/94/20

Table 184 Residues in Apples (outdoor trails) from supervised trials in New Zealand, Chile, Brazil and Australia involving foliar applications of Fenpyroximate 5% SC

Apples	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial:- 91/653 Hawkes Bay, New Zealand, 1991 (Braeburn)	2.5 g/100L 38-88	1500-3500	1	0 7 14 28 42	Fruit Fruit Fruit Fruit Fruit	0.07 0.07 0.12 ND ND	- - - - -	0.07 0.07 0.12 ND ND	Report: R-4049e Study:- Study not to GLP

Apples Trial Location Country,year (Variety)	Application Rate (g ai/ha)	 Water (L/ha)	 No.	DALA (days)	Commodity	Residues (mg/kg) Fenpyroximate	 M-1	 Sum of fenpyroximate and M-1	Reference & Comments
	5 g/100L 75-175	1500- 3500	1	0 7 14 28 42 56	Fruit Fruit Fruit Fruit Fruit Fruit	0.13 <u>0.10</u> 0.05 0.03 ND ND	- - - - - -	0.13 <u>0.10</u> 0.05 0.03 ND ND	
Trial:- 91/976 Ranqiora, New Zealand, 1991 (Braeburn)	2.5 g/100L 38-88	1500- 3500	1	0 7 14 28 42	Fruit Fruit Fruit Fruit Fruit	0.07 0.12 0.13 ND ND	- - - - -	0.07 0.12 0.13 ND ND	Report: R-4049e Study:- Study not to GLP
	5 g/100L 75-175	1500- 3500	1	0 7 14 28 42 56	Fruit Fruit Fruit Fruit Fruit Fruit	0.15 <u>0.07</u> 0.04 0.03 0.01 ND	- - - - - -	0.15 <u>0.07</u> 0.04 0.03 0.01 ND	
Trial:- 91/1163 Waikato, New Zealand, 1991 (Fuji)	2.5 g/100L 38-88	1500- 3500	1	0 7 14 21 28 42	Fruit Fruit Fruit Fruit Fruit Fruit	0.05 0.09 0.03 ND ND 0.01	- - - - - -	0.05 0.09 0.03 ND ND 0.01	Report: R-4049e Study:- Study not to GLP
	5 g/100L 75-175	1500- 3500	1	0 7 14 21 28 42 56	Fruit Fruit Fruit Fruit Fruit Fruit Fruit	0.20 <u>0.06</u> 0.02 ND ND ND ND	- - - - - - -	0.20 <u>0.06</u> 0.02 ND ND ND ND	
Trial:- 152/94 Molina, Chile, 1993 (Braeburn)	2.5 g ai/100 L		1	16 37 47 70	- - - -	0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.02 < 0.02 < 0.02 < 0.02	Report: R-4061e Study:- Study not to GLP
	5 g ai/100 L		1	16 37 47 70	- - - -	0.04 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.05 0.03 < 0.02 < 0.02	
Trial:- 152/94 Molina, Chile, 1993 (Gala)	2.5 g ai/100 L		1	65	-	0.00	0.00	0.00	Report: R-4061e Study:- Study not to GLP
Trial:- 152/94 Molina, Chile, 1993 (R.K. Oregon)	2.5 g ai/100 L		1	122	-	0.02	0.00	0.02	Report: R-4061e Study:- Study not to GLP
Trial: 94/699 Hastings, New Zealand, 1994 (Braeburn)	2.5 g ai/100 L 38-88	1500- 3500	1	1 7 14 21 28 35 42 49	Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit	0.08 0.06 0.03 0.04 0.03 0.02 0.02 0.02	- - - - - - - -	0.08 0.06 0.03 0.04 0.03 0.02 0.02 0.02	Report: R-4065e

Apples	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
	5 g ai/100 L 75-175	1500- 3500	1	1 7 14 21 28 35 42 49	Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit	0.13 <u>0.11</u> 0.06 0.04 0.05 0.03 0.04 0.02	- - - - - - - -	0.13 <u>0.11</u> 0.06 0.04 0.05 0.03 0.04 0.02	
Trial:- 94/1234 Waikato, New Zealand, 1994 (Royal Gala)	2.5 g ai/100 L 38-88	1500- 3500	1	1 7 14 23 27 36 43 52	Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit	0.02 0.01 ND ND ND ND ND ND	- - - - - - - -	0.02 0.01 ND ND ND ND ND ND	Report: R-4065e
	5 g ai/100 L 75-175	1500- 3500	1	1 7 14 23 27 36 43 52	Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit	0.08 <u>0.05</u> 0.03 0.02 0.02 0.01 ND ND	- - - - - - - -	0.08 <u>0.05</u> 0.03 0.02 0.02 0.01 ND ND	
Trial:- 93/1729 Rangiora, New Zealand, 1994 (Braeburn)	2.5 g ai/100 L 38-88	1500- 3500	1	1 9 14 21 28 35 42 49 54	Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit	0.11 0.05 0.04 0.04 0.02 0.02 0.02 0.01 ND	- - - - - - - - -	0.11 0.05 0.04 0.04 0.02 0.02 0.02 0.01 ND	Report: R-4065e
	5 g ai/100 L 75-175	1500- 3500	1	1 9 14 21 28 35 42 49 54	Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit	0.15 0.06 <u>0.08</u> 0.03 0.03 0.04 0.03 0.01 0.01	- - - - - - - - -	0.15 0.06 <u>0.08</u> 0.03 0.03 0.04 0.03 0.01 0.01	
Report: R-4065e Study:- Trial:- 94/1737 Rangiora, New Zealand, 1994 (Braeburn)	2.5 g ai/100 L 38-88	1500- 3500	1	1 7 14 21 28 35 42 49 56	Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit	0.04 0.01 0.03 0.02 ND 0.01 0.02 ND 0.01	- - - - - - - - -	0.04 0.01 0.03 0.02 ND 0.01 0.02 ND 0.01	Report: R-4065e Study:-
	5 g ai/100 L 75-175	1500- 3500	1	1 7 14 21 28 35 42 49 56	Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit	0.08 <u>0.07</u> 0.06 0.05 0.03 0.03 0.03 0.03 0.03	- - - - - - - - -	0.08 <u>0.07</u> 0.06 0.05 0.03 0.03 0.03 0.03 0.03	

Apples	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial:- Cosmopolis, Brazil, 1989 (Anna)	25 + 25 35 day interval	1000 1000	2	15 30	Fruit Fruit	< 0.05 < 0.05	< 0.05 < 0.05	< 0.10 < 0.10	Report: R-4093e, A46385 Study:- Study not to GLP
Trial:- Cosmopolis, Brazil, 1989 (Anna)	50 + 50 35 day interval	1000 1000	2	15 30	Fruit Fruit	< 0.05 < 0.05	< 0.05 < 0.05	< 0.10 < 0.10	R-4094e, A46386 Study:- Study not to GLP
Trial:- Cosmopolis, Brazil, 1989-90 (Ohio Beauty)	50 + 50 34 day interval	1000 1000	2	15 30	Fruit Fruit	< 0.05 < 0.05	< 0.05 < 0.05	< 0.10 < 0.10	Report: R-4095e, A46387 Study:- Study not to GLP
Trial:- Cosmopolis, Brazil, 1989-90 (Ohio Beauty)	100 + 100 34 day interval	1000 1000	2	15 30	Fruit Fruit	< 0.05 < 0.05	< 0.05 < 0.05	< 0.10 < 0.10	Report: R-4096e, A46388 Study:- Study not to GLP
Trial: Apples, 100 mL/100 L Australia, 1992 (Golden delicious)	83.3		1	0 7 14 24	Fruit Fruit Fruit Fruit	0.10 <u>0.07</u> 0.10 0.08	< 0.01 < 0.01 < 0.01 < 0.01	0.11 <u>0.08</u> 0.11 0.09	Report: R-4048 Study: 92/12/1378
Trial: Apples, 200 mL/100 L Australia, 1992 (Golden delicious)	166.6		1	0 7 14 24	Fruit Fruit Fruit Fruit	0.28 0.26 0.18 0.14	< 0.01 < 0.01 < 0.01 < 0.01	0.29 0.27 0.19 0.15	Report: R-4048 Study: 92/12/1378

Table 185 Residues in Apples (outdoor trails) from supervised trials in USA involving foliar applications of Fenpyroximate 5% EC

Apples	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: MI Ottawa, MI, USA, 1993 (Golden Delicious)	145 + 145 14 day interval	1814 1805	2	7	Whole fruit Juice Wet pomace	0.21, 0.15, 0.18 < 0.05 0.93	< 0.05 < 0.05 0.07	0.26,0.20, 0.23 < 0.10 1.00	Report: R-4106e Study:- AA940423
Trial: NY Wayne, NY, USA, 1993 (Idared)	150 + 149 14 day interval	1879 1870	2	7	Whole fruit Juice Wet pomace	0.10,0.15, 0.13 < 0.05 0.65	< 0.05 < 0.05 < 0.05	0.15,0.20, 0.18 < 0.10 0.70	Report: R-4106e Study:- AA940423
Trial: PA Berks, PA, USA, 1993 (Rome)	148 + 151 14 day interval	1823 1870	2	7	Whole fruit Juice Wet pomace	0.06,0.10, 0.08 < 0.05 0.48	< 0.05 < 0.05 < 0.05	0.11,0.15, 0.13 < 0.10 0.53	Report: R-4106e Study:- AA940423
Trial: WA Grant, WA, USA, 1993 (Red Delicious)	145 + 150 14 day interval	1833 1889	2	7	Whole fruit Juice Wet pomace	0.10,0.09, 0.10 < 0.05 0.38	< 0.05 < 0.05 < 0.05	0.15,0.14, 0.15 < 0.10 0.43	Report: R-4106e Study:- AA940423

Apples	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: NY01 North Rose, NY, USA, 1999 (Monroes)	1142	935	1	14	Whole fruit Juice Dry pomace Wet pomace	ND,0.084,0.104, 0.09 < 0.01 2.563 0.309 (residues measured as the sum of fenpyroximate + M-1)			Report: R-4120e Study:- 828W-1

Table 186 Residues in Apples (outdoor trails) from supervised trials in Japan involving foliar applications of Fenpyroximate 5% SC or 5% EC

Apples	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial:- Aomori Aomori, Japan, 1988 (Fuji)	140	2800	1	15 30 45 60	Fruit Fruit Fruit Fruit	0.11 0.08 0.03 0.04	< 0.01 < 0.01 < 0.01 < 0.01	0.12 0.09 0.04 0.05	Report: R-4058e Study: NN023-01, NN023-02
Trial:- Nagano Nagano, Japan, 1988 (Fuji)	250	5000	1	14 30 45 60	Fruit Fruit Fruit Fruit	0.05 0.03 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.06 0.04 < 0.02 < 0.02	Report: R-4058e Study:- NN023-01, NN023-02
Trial:- Aomori Japan, 1988 (Fuji)	140	280 L/10a	1	14 29 45 60	Fruit Fruit Fruit Fruit	0.09 0.07 0.04 0.06	- - - -	0.09 0.07 0.04 0.06	Report: R4260e Study:- NN023-04 Study not to GLP
Trial:- Nagano Japan, 1988 (Fuji)	250	500 L/10a	1	14 30 45 60	Fruit Fruit Fruit Fruit	0.05 < 0.01 0.03 0.01	- - - -	0.05 < 0.01 0.03 0.01	Report: R4260e Study:- NN023-04 Study not to GLP
Trial:- Aomori Japan, 1988 (Fuji)	140	280 L/10a	1	14 29 45 60	Fruit Fruit Fruit Fruit	- - - -	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	Report: R-4261e Study:- NN023-04 Study not to GLP
Trial:- Nagano Japan, 1988 (Fuji)	250	500 L/10a	1	14 30 45 60	Fruit Fruit Fruit Fruit	- - - -	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	Report: R-4261e Study:- NN023-04 Study not to GLP

Pear

Table 187 Residues in Pears (outdoor trails) from supervised trials in New Zealand, Japan, Australia, USA and EU involving foliar applications of Fenpyroximate 5% SC or 5% EC

Pears	Application			DALA	Commodity	Residues (mg/kg)			Reference
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	& Comments
Trial: 93/1730 Christchurch, New Zealand, 1994 (Winter Cole)	2.5 g ai/100 L Spray to runoff 38-88	1500-3500	1	1 9 14 21 28 35 42 49 54	Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit	0.14 0.07 0.04 0.05 0.04 0.03 0.01 0.02 0.03	-	0.14 0.07 0.04 0.05 0.04 0.03 0.01 0.02 0.03	Report: R-4066 Study: 93/35D
	5 g ai/100 L Spray to runoff 75-175	1500-3500	1	1 9 14 21 28 35 42 49 54	Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit Fruit	0.23 0.19 0.07 0.06 0.05 0.04 0.02 0.01 ND	-	0.23 0.19 0.07 0.06 0.05 0.04 0.02 0.01 ND	
Trial: 94/1738 New Zealand, 1994 (Winter Cole)	2.5 g ai/100 L Spray to runoff	1500-3500	1	1 14 21 28 42 56	Fruit Fruit Fruit Fruit Fruit Fruit	0.09 0.06 0.04 0.02 0.02 0.01	-	0.09 0.06 0.04 0.02 0.02 0.01	Report: R-4066 Study: 93/35D
	5 g ai/100 L Spray to runoff	1500-3500	1	1 14 21 28 42 56	Fruit Fruit Fruit Fruit Fruit Fruit	0.12 0.14 0.07 0.08 0.03 0.02	-	0.12 0.14 0.07 0.08 0.03 0.02	
Trial: CA1 Porterville, CA, USA, 2001 (Shinko)	450	944	1	14	Fruit	0.0817, 0.0643 0.0730	< 0.05	0.1317, 0.1143 0.1230	Report: R-4154 Study: AA010707
	453	944	1	14	Fruit	0.0733, 0.0712 0.0722	< 0.05	0.1233, 0.1212 0.1222	
Trial: CA2 Porterville, CA, USA, 2001 (Bosc)	450	916	1	14	Fruit	0.119,0.131 0.125	0.0528,0.0624 0.0576	0.1718,0.1934 0.1826	Report: R-4154 Study: AA010707 (EC)
Trial: NY1 Lyons, NY, USA, 2001 (Bartlett)	446	935	1	14	Fruit	0.232, 0.319 0.276	0.0689, 0.0741 0.0715	0.3009, 0.3931 0.3475	Report: R-4154 Study: AA010707 (EC)
Trial: OR1 Corvallis, OR, USA, 2001 (Red Anjou)	447	1122	1	14	Fruit	0.1494, 0.185 0.173	< 0.05	0.1994, 0.235 0.223	Report: R-4154 Study: AA010707
	447	954	1	14	Fruit	0.173, 0.185 0.179	< 0.05	0.223, 0.235 0.229	

Pears	Application			DALA	Commodity	Residues (mg/kg)			Reference
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	& Comments
Trial: OR2 Corvallis, OR, USA, 2001 (Cascade)	454	935	1	7 14 21 28	Fruit	0.212,0.214, 0.213 0.152,0.175,0.164 0.134,0.127,0.131 0.148,0.150,0.149	0.0623,0.0694 0.0689 0.0536,0.05250.0531 0.0556, 0.05, 0.0528 < 0.05,0.0529,0.0515	0.2743,0.2834 2819 0.2056,0.22750.2171 0.1896,0.127,0.1838 0.198,0.2029,0.2005	Report: R-4154 Study: AA010707 (EC)
Trial: WA1 Ephrata, WA, USA, 2001 (Bartlett)	448	963	1	14	Fruit	0.245,0.199 0.242	0.0643,0.0516 0.0540	0.3093,0.2506 0.296	Report: R-4154 Study: AA010707
	449	963	1	14	Fruit	0.252,0.273 0.263	< 0.05	0.302,0.323 0.313	
Trial: AD/6100/NN/2 Lagarde, Tarn-et-Garonne, S-France, 2001 (Cornice)	73.8	1476	1	10 14	Fruit Fruit	0.03 0.02	< 0.01 < 0.01	0.04 0.03	Report: R-4161 Study:
Trial: AF/11088/NN/3 Corquilleroy, N-France, 2006 (Crassane)	120.0	1493	1	0 14 21 42 56	Fruit Fruit Fruit Fruit Fruit	0.08 0.04 0.04 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.09 0.05 0.05 0.03 0.03	Report: R-4185 Study:
Trial: CA-17A1, 0.1 Madera, CA, USA, 2005 (Asian)	112	926	1	14	Fruit	< 0.05	< 0.05	< 0.10	Report: R-4201 Study: (EC)
	448	926	1	14	Fruit	0.195,0.250 0.223	0.095,0.113 0.104	0.29,0.363, 0.327	
Trial: CA-17A2, 0.1 Linda/Marysville, CA, USA, 2005 (Bartlett)	112	935	1	14	Fruit	0.070,0.101 0.086	< 0.05, < 0.05 < 0.05	0.120,0.151 0.136	Report: R-4201 Study: (EC)
	448	935	1	14	Fruit	0.345,0.265 0.305	0.05, < 0.05 0.05	0.395,0.315 0.355	
Trial: WA-17A, 0.1 Ephrata, WA, USA, 2005 (Concord)	112	935	1	14	Fruit	0.052, < 0.05 0.051	< 0.05,0.032 < 0.05	0.102,0.082 0.101	Report: R-4201 Study: (EC)
	448	935	1	14	Fruit	0.2449,0.155 0.202	0.146,0.092 < 0.05	0.395,0.247 0.252	
Trial location: Niigata Japan, 1988 (Niitaka)	250	500 L/10a	1	7 14 21 30 60	Fruit Fruit Fruit Fruit Fruit	0.066 0.044 0.032 < 0.005 0.010	- - - - -	0.066 0.044 0.032 < 0.005 0.010	Report: R-4263 Study: NN024-01
Trial location: Tokushima Japan, 1988 (Kosui)	250	500 L/10a	1	7 13 21 30 60	Fruit Fruit Fruit Fruit Fruit	0.194 0.110 < 0.005 0.119 < 0.005	- - - - -	0.194 0.110 < 0.005 0.119 < 0.005	Report: R-4263 Study: NN024-01
Trial location: Niigata Japan, 1988 (Niitaka)	250	500 L/10a	1	7 14 21 30 60	Fruit Fruit Fruit Fruit Fruit	- - - - -	0.008 0.008 0.008 < 0.005 < 0.005	0.008 0.008 0.008 < 0.005 < 0.005	Report: R-4264 Study: NN024-02
Trial location: Tokushima Japan, 1988 (Kosui)	250	500 L/10a	1	7 13 21 30 60	Fruit Fruit Fruit Fruit Fruit	- - - - -	0.019 0.012 < 0.005 0.016 < 0.005	0.019 0.012 < 0.005 0.016 < 0.005	Report: R-4264 Study: NN024-02

Pears	Application			DALA	Commodity	Residues (mg/kg)			Reference
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	& Comments
Trial location: Niigata Japan, 1988 (Niitaka)	250	500 L/10a	1	7 14 21 30 60	Fruit Fruit Fruit Fruit Fruit	0.055 0.040 0.046 < 0.005 0.018	- - - - -	0.055 0.040 0.046 < 0.005 0.018	Report: R-4266 Study: NN024-03
Trial location: Tokushima Japan, 1988 (Kosui)	250	500 L/10a	1	7 13 21 30 60	Fruit Fruit Fruit Fruit Fruit	0.172 0.118 < 0.005 0.106 < 0.005	- - - - -	0.172 0.118 < 0.005 0.106 < 0.005	Report: R-4266 Study: NN024-03
Trial: Pears, Australia, 1992 (Williams)	0.45 g ai/tree	100 mL/100 L	1	0 7 14 22	Fruit Fruit Fruit Fruit	0.08 0.06 0.05 0.06	< 0.01 < 0.01 < 0.01 < 0.01	0.09 0.07 0.06 0.07	Report: R-4048 Study:
Trial: Pears, Australia, 1992 (Williams)	0.9 g ai/tree	100 mL/100 L	1	0 7 14 22	Fruit Fruit Fruit Fruit	0.09 0.08 0.10 0.10	< 0.01 < 0.01 < 0.01 < 0.01	0.10 0.09 0.11 0.11	Report: R-4048 Study:

Stone fruits

Cherry

Table 188 Residues in Cherries (outdoor trails) from supervised trials in USA involving foliar applications of Fenpyroximate 5% EC

Cherries	Application			DALA	Commodity	Residues (mg/kg)			
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	Reference & Comments
Trial: 10-CA90 Parlier, CA, USA, 2010 (Tulare)	113, 112 (Induce)	1327 1318	2	7	Fruit without pits and stems	0.332,0.319 0.326	< 0.05	0.382,0.369 0.376	Report: R-4519; Study-10438.10-MIR02
	113, 112 (Silwet L-77)	1337 1318	2	7	Fruit without pits and stems	0.409,0.505 0.457	< 0.05	0.459,0.555 0.507	
	113, 112 (no surfactant)	1328 1309	2	7	Fruit without pits and stems	0.276,0.269 0.273	< 0.05	0.326,0.319 0.323	
Trial: 10-CO03 Montrose, CO, USA, 2010 (Montmorency)	108, 117	907 982	2	7	Cherries without pits and stems	0.617,0.754 0.686	0.0921,0.108 0.100	0.7091,0.862 0.787	Report: R-4519; Study-10438.10-MIR02
Trial: .10-MI24 Fennville, MI, USA, 2010 (Montmorency)	112, 111	561 561	2	7	Cherries without pits and stems	0.590.0.584 0.587	0.0812,0.0659 0.0736	0.6712,0.6499 0.6606	Report: R-4519; Study-10438.10-MIR02
Trial: 10-MI25 Fennville, MI, USA, 2010 (Montmorency)	111, 112	1066 1122	2	7	Cherries without pits and stems	0.839,0.897 0.868	0.112,0.123 0.118	0.951,1.02 0.986	Report: R-4519; Study-10438.10-MIR02
Trial: 10-MI26 Fennville, MI, USA, 2010 (Heidelfingen)	112, 113	1026 1066	2	7	Cherries without pits and stems	0.359,0.356 0.358	< 0.05	0.409,0.406 0.408	Report: R-4519; Study-10438.10-MIR02
Trial: 10-NY12 Ithaca, NY, USA, 2010 (Galaxy)	112, 112	1057 1103	2	7	Cherries without pits and stems	0.763,0850 0.807	0.122,0.120 0.121	0.885,0.970 0.928	Report: R-4519; Study-10438.10-MIR02

Cherries	Application			DALA	Commodity	Residues (mg/kg)			
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	Reference & Comments
Trial: 10-WA22 Procsser, WA, USA, 2010 (Bing)	114, 113 (Ad Wet 90)	1047 1318	2	7	Fruit without pits and stems	0.0889,0.0978 0.093	< 0.05	0.1389,0.1478 0.143	Report: R-4519; Study-10438.10-MIR02
	111, 106 (JMS Stylet oil)	982 991	2	7	Fruit without pits and stems	0.345,0.334, 0.340	< 0.05	0.395,0.384 0.390	
	114, 111 (no surfactant)	991 991	2	7	Fruit without pits and stems	0.313,0.285 0.299	< 0.05	0.363,0.335 0.349	
Trial: 10-WA*23 Wapato, WA, USA, 2010 (Bing)	110, 110	1000 1000	2	8	Cherries without pits and stems	0.275,0.249 0.262	< 0.05	0.325,0.299 0.312	Report: R-4519; Study-10438.10-MIR02

Peach

Table 189: Residues in Peaches (outdoor trails) from supervised trials in Japan, USA and EU involving foliar applications of Fenpyroximate 5% SC or 5% EC

Peaches	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial location: Nagano Japan, 1989 (Peach)	250	500 L/10a	1	7 14 21	Flesh–Stone Flesh–Stone Flesh–Stone	0.008 < 0.005 < 0.005	- - -	0.008 < 0.005 < 0.005	Report: R-4134 Study: -
Trial location: Okayama Japan, 1989 (Peach)	150	500 L/10a	1	7 14 21	Flesh–Stone Flesh–Stone Flesh–Stone	< 0.005 < 0.005 < 0.005	- - -	< 0.005 < 0.005 < 0.005	Report: R-4134 Study: -
Trial: 9051 TL1 Fronton, S-France, 1999 (Peach)	82.2	1000	1	21	Fruit	0.025	-	0.025	Report: R-4147 Study: 9051
Trial: 9051 BD1 Pont-de-l'lsere, S-France, 1999 (Peach)	76.6	1000	1	21	Fruit	0.039	-	0.039	Report: R-4147 Study: 9051
Trial: AF/6101/NN/1 St Hilaire St Mesmin, Loiret, N-France, 2001 (Sun Crest)	73.2	1464	1	0 3 7 10 14 0 3 7 10 14	Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Whole fruit Whole fruit Whole fruit Whole fruit Whole fruit	0.17 0.11 0.07 0.06 0.04 0.15 0.09 0.06 0.05 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.18 0.12 0.08 0.07 0.05 0.16 0.10 0.07 0.06 0.05	Report: R-4166 Study: AF/6101/NN
Trial: AF/6101/NN/2 Clery Saint Andre, Loiret, N-France, 2001 (Velvet)	77.2	1544	1	10 14 10 14	Fruit–Stones Fruit–Stones Whole fruit Whole fruit	0.01 0.02 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.02 0.03 0.02 0.03	Report: R-4166 Study: AF/6101/NN

Peaches	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: AF/6771/NN/1 Catelsarrasin, Tarn-et-Garonne, S-France, 2002 (Royal Glorie)	80.2	1601	1	0 3 7 10 14 0 3 7 10 14	Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Whole fruit Whole fruit Whole fruit Whole fruit Whole fruit	0.07 0.05 0.04 0.01 < 0.01 0.06 0.05 0.04 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.08 0.06 0.05 0.02 < 0.02 0.07 0.06 0.05 0.02 < 0.02	Report: R-4174 Study: AF/6771/NN
Trial: AF/6771/NN/2 Moissac, Tarn-et-Garonne, S-France, 2002 (Royal Glory)	75.9	1518	1	10 14 10 14	Fruit–Stones Fruit–Stones Whole fruit Whole fruit	0.05 0.02 0.05 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.06 0.03 0.06 0.03	Report: R-4174 Study: AF/6771/NN
Trial: AF/6771/NN/3 San Jose de la Rinconada, Seville, S-Spain, 2002 (SP4)	71.6	1432	1	10 14 10 14	Fruit–Stones Fruit–Stones Whole fruit Whole fruit	0.03 0.04 0.03 0.04	< 0.01 < 0.01 < 0.01 < 0.01	0.04 0.05 0.04 0.05	Report: R-4174 Study: AF/6771/NN
Trial: AF/6771/NN/4 Calatorao, Zaragoza, Spain, 2002 (Sudanel)	74.6	1491	1	0 3 7 10 14 0 3 7 10 14	Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Whole fruit Whole fruit Whole fruit Whole fruit Whole fruit	0.06 0.03 0.02 0.02 0.02 0.06 0.03 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.07 0.04 0.03 0.03 0.03 0.07 0.04 0.03 0.03 0.03	Report: R-4174 Study: AF/6771/NN
Trial: AF/6771/NN/5 Utebo, Zaragoza, N-Spain, 2002 (Baby Gold 7)	75.3	1505	1	10 14 10 14	Fruit–Stones Fruit–Stones Whole fruit Whole fruit	0.03 0.02 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.04 0.03 0.04 0.03	Report: R-4174 Study: AF/6771/NN
Trial: AF/6773/NN/1 Fotenay, Linieres de Touraine, N-France, 2002 (Dixy Red)	79.8	1595	1	0 3 7 10 14 0 3 7 10 14	Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Whole fruit Whole fruit Whole fruit Whole fruit Whole fruit	0.07 0.05 0.05 0.03 0.03 0.05 0.04 0.04 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.08 0.06 0.06 0.04 0.04 0.06 0.05 0.05 0.04 0.03	Report: R-4175 Study: AF/6773/NN
Trial: AF/6773/NN/2 La Giberdiere, Valleres, N-France, 2002 (Rouge Sanguine)	76.0	1519	1	10 14 10 14	Fruit–Stones Fruit–Stones Whole fruit Whole fruit	0.04 0.03 0.04 0.03	< 0.01 < 0.01 < 0.01 < 0.01	0.05 0.04 0.05 0.04	Report: R-4175 Study: AF/6773/NN

Peaches	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: AF/6773/NN/3 Voutezac, Correze, N- France, 2002 (Flavor Crest)	76.5	1529	1	10 14 10 14	Fruit–Stones Fruit–Stones Whole fruit Whole fruit	0.06 0.04 0.05 0.04	< 0.01 < 0.01 < 0.01 < 0.01	0.07 0.05 0.06 0.05	Report: R-4175 Study: AF/6773/NN
Trial: 01 Quatretonda, Valencia, Spain, 2009 (Royal Gladys)	77.4	1547	1	0 3 7 10 14	Whole fruit Whole fruit Whole fruit Whole fruit Whole fruit	0.08 0.09 0.08 0.07 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.09 0.10 0.09 0.08 0.09	Report: R-4428 Study: S09-02265
Trial: 02 Cofrentes, Spain, 2009 (Melcoton de Cofrentes)	77.0	1539	1	0 14	Whole fruit Whole fruit	0.37 0.12	< 0.01 < 0.01	0.38 0.13	Report: R-4428 Study: S09-02265
Trial: .10-AR04 Fayetteville, AR, USA, 2010 (Cresthaven)	115 + 115 14 day interval	1197 1206	2	7	Fruit (without pit)	0.0731	< 0.05	0.1231	Report: R-4520 Study: 10468 (EC)
Trial: .10-CA93 Parlier, CA, USA, 2010 (Crimson Lady)	114 + 114 13 day interval	804 804	2	8	Fruit (without pit)	0.0983	< 0.05	0.1483	Report: R-4520 Study: 10468 (EC)
Trial: .10-CA94 Parlier, CA, USA, 2010 (Henry II)	114 + 114 14 day interval (Induce)	1066 1075	2	8	Fruit (without pit)	0.1470	< 0.05	0.1970	Report: R-4520 Study: 10468 (EC)
	112 + 115 14 day interval (Silwet L-77)	1047 1075	2	8	Fruit (without pit)	0.1803	< 0.05	0.2303	
	112 + 114 14 day interval (no surfactant)	1057 1066	2	8	Fruit (without pit)	0.0978	< 0.05	0.1478	
Trial: .10-CA95 Davis, CA, USA, 2010 (O'Henry)	111 + 115 7 day interval (Pro 90)	1206 1356	2	2 7 11 14	Fruit (without pit)	0.2396,0.2767,0.2582 0.1904,0.2000,0.1952 0.1572,0.1566,0.1569 0.0987,0.1587,0.1287	< 0.05 < 0.05 < 0.05 < 0.05	0.2896,0.3267, 0.,3082 0.2404,0.2500 0.2452 0.2072,0.2066 0.2069 0.1487,0.2087 0.1787	Report: R-4520 Study: 10468 (EC)
	111 + 119 7 day interval (Silwet L-77)	1197 1384	2	7	Fruit (without pit)	0.1325,0.1618,0.1472	< 0.05	0.1825,0.2118 0.1972	
Trial: .10-MI27 Fennville, MI, USA, 2010 (Red Haven)	111 + 112 14 day interval	1038 1038	2	7	Fruit (without pit)	0.1332,0.1257 0.1295	< 0.05	0.1832,0.1757 0.1795	Report: R-4520 Study: 10468 (EC)
Trial: .10-NC18 Raleigh, NC, USA, 2010 (Contender)	112 + 112 15 day interval (Induce)	1085 1075	2	6	Fruit (without pit)	0.0771,0.0833 0.0802	< 0.05	0.1271,0.1333 0.1302	Report: R-4520 Study: 10468 (EC)

Peaches	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
	113 + 113 15 day interval (Trigard)	1085 1085	2	6	Fruit (without pit)	0.1067,0.1215 0.1141	< 0.05	0.1567,0.1715 0.1641	
	114 + 111 15 day interval (no surfactant)	1094 1066	2	6	Fruit (without pit)	0.0599,0.0589 0.0594	< 0.05	0.1099,0.1089 0.1094	
Trial: .10-NC19 Raleigh, NC, USA, 2010 (Contender)	111 + 113 13 day interval	1057 1075	2	6	Fruit (without pit)	0.1704,0.1281 0.1493	< 0.05	0.2204,0.1781 0.1993	Report: R-4520 Study: 10468 (EC)
Trial: .10-NJ09 Cream Ridge, NJ, USA, 2010 (Suncrest)	112 + 113 13 day interval	1309 1318	2	7	Fruit (without pit)	0.1180,0.1333 0.1257	< 0.05	0.1680,0.1833 0.1757	Report: R-4520 Study: 10468 (EC)
Trial: .10-NJ10 Cream Ridge, NJ, USA, 2010 (Loring)	113 + 115 13 day interval	1318 1262	2	7	Fruit (without pit)	0.0962,0.0750 0.0856	< 0.05	0.1462,0.1250 0.1356	Report: R-4520 Study: 10468 (EC)
Trial: .10-TX12 Weslaco, TX, USA, 2010 (Sentinel)	112 + 106 14 day interval	944 926	2	7	Fruit (without pit)	0.0783,0.0714 0.0749	< 0.05	0.1283,0.1214 0.1249	Report: R-4520 Study: 10468 (EC)

Apricot

Table 190 Residues in Apricots (outdoor trails) from supervised trials in EU involving foliar applications of Fenpyroximate 5% SC

Apricots	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: 01 Dangolsheim, Alsace, N-France, 2015 (Tardif de Tain)	79.1	1010	1	0 1 3 7 14 0 1 3 7 14	Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Whole fruit Whole fruit Whole fruit Whole fruit Whole fruit	0.35 0.30 0.27 0.24 0.23 0.32 0.28 0.25 0.22 0.22	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.36 0.31 0.28 0.25 0.24 0.33 0.29 0.26 0.23 0.23	Report: R-4514 Study: S15-03092

Apricots	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: 02 Meckenheim, Rheinland-Pfalz, Germany, 2015 (Kaito)	78.7	1005	1	0 1 3 7 14 0 1 3 7 14	Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Whole fruit Whole fruit Whole fruit Whole fruit Whole fruit	0.37 0.24 0.21 0.20 0.14 0.32 0.22 0.19 0.18 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.38 0.25 0.22 0.21 0.15 0.33 0.23 0.20 0.19 0.14	Report: R-4514 Study: S15-03092
Trial: 03 Dangolsheim, Bas-Rhin,N-France, 2015 (Goldrich)	79.2	1011	1	14 14	Fruit-Stone Whole fruit	0.04 0.04	< 0.01 < 0.01	0.05 0.05	Report: R-4514 Study: S15-03092
Trial: 04 Obersulm, Baden-Württemberg, Germany, 2015 (Hargand)	80.4	1027	1	14 14	Fruit-Stone Whole fruit	0.10 0.10	0.01 < 0.01	0.11 0.11	Report: R-4514 Study: S15-03092
Trial: 05 Llutxent, Valencia,Spain, 2015 (Tadeo)	81.2	1038	1	0 1 3 7 14 0 1 3 7 14	Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Whole fruit Whole fruit Whole fruit Whole fruit Whole fruit	0.28 0.22 0.11 0.09 0.11 0.26 0.20 0.10 0.09 0.10	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.29 0.23 0.12 0.10 0.12 0.27 0.21 0.11 0.10 0.11	Report: R-4514 Study: S15-03092
Trial: 06 Altedo, Emila Romagna, Italy, 2015 (Precoce d'Imola)	78.9	1008	1	0 1 3 7 14 0 1 3 7 14	Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Fruit–Stones Whole fruit Whole fruit Whole fruit Whole fruit Whole fruit	0.06 0.03 0.04 0.03 0.01 0.05 0.03 0.04 0.03 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.07 0.04 0.05 0.04 0.02 0.06 0.04 0.05 0.04 < 0.02	Report: R-4514 Study: S15-03092
Trial: 07 Tobarra, Albacete,Spain, 2015 (Apricot Moniquí)	79.1	1010	1	14 14	Fruit-Stone Whole fruit	0.09 0.08	< 0.01 < 0.01	0.10 0.09	Report: R-4514 Study: S15-03092
Trial: 08 Zola Predosa, Emilia Romagna, Italy, 2015 (Lady Elena)	83.6	1068	1	14 14	Fruit-Stone Whole fruit	0.05 0.05	0.01 < 0.01	0.06 0.06	Report: R-4514 Study: S15-03092

Plum

Table 191 Residues in Plums (outdoor trails) from supervised trials in USA and EU involving foliar applications of Fenpyroximate 5% SC or 5% EC

Plums	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: - Gruenendelch, Germany, 1989 (Ortenaner)	112.5	1500	1	0 7 14 21	Fruit Fruit Fruit Fruit	0.02 0.04 0.015 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.03 0.05 0.025 0.03	Report: R-4024 Study: -
Trial: - Bornheim, Germany, 1989 (Purpur Gold)	112.5	1500	1	0 7 14 21	Fruit Fruit Fruit Fruit	0.07 0.05 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01	0.08 0.06 0.04 0.04	Report: R-4025 Study: -
Trial: - Bischweler, Germany, 1989 (Zimmers)	112.5	1500	1	0 7 14 21	Fruit Fruit Fruit Fruit	0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 < 0.02 < 0.02	Report: R-4026 Study: -
Trial: - Hoechst, Germany, 1989 (Ortenaner)	63	840	1	0 7 14 21	Fruit Fruit Fruit Fruit	0.03 0.03 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.04 0.04 < 0.02 < 0.02	Report: R-4027 Study: -
Trial: 0101 Grünendeich, Germany, 1990 (Ortenaner)	112.5	1500	1	0 7 14 21 28	Fruit Fruit Fruit Fruit Fruit	< 0.05 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.10 0.10 < 0.10 < 0.10 < 0.10	Report: R-4135 Study: -
Trial: 0102 Bornheim, Germany, 1990 (Ortenaner)	79	1000	1	0 7 14 21 28	Fruit Fruit Fruit Fruit Fruit	0.09 < 0.05 0.06 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.14 < 0.10 0.11 < 0.10 < 0.10	Report: R-4135 Study: -
Trial: 0301 Bischweier, Germany, 1990 (Zimmers)	54.5	727	1	0 7 14 21 28	Fruit Fruit Fruit Fruit Fruit	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.10 < 0.10 < 0.10 < 0.10 < 0.10	Report: R-4135 Study: -
Trial: 0401 Hochst, Germany, 1990 (Hauszwetsche)	74	987	1	0 7 14 21 28	Fruit Fruit Fruit Fruit Fruit	0.06 0.07 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.11 0.12 < 0.10 < 0.10 < 0.10	Report: R-4135 Study: -
Trial: AF/6102/NN/1 Lafrancaise, Tarn-et-Garonne, S-France, 2001 (Bavee)	75	1503	1	0 3 7 10 14	Fruit incl stone Fruit incl stone Fruit incl stone Fruit incl stone Fruit incl stone	0.03 0.03 0.02 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.04 0.04 0.03 0.04 0.03	Report: R-4169 Study: -
Trial: AF/6102/NN/2 Lizac, Tarn-et-Garonne, S-France, 2001 (President)	75	1501	1	10 14	Fruit incl stone Fruit incl stone	0.03 0.01	< 0.01 < 0.01	0.04 0.02	Report: R-4169 Study: -

Plums	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: AF/6770/NN/1 Calatorao, La Almunia de Dona Godina, Spain, 2002 (Claudia)	75.5	1510	1	10 14	Fruit incl stone Fruit incl stone	0.03 < 0.01	< 0.01 < 0.01	0.03 < 0.02	Report: R-4170 Study: -
Trial: AF/6772/NN/1 Jussy, N-France, 2002 (President)	76.4	1527	1	10 14	Fruit incl stone Fruit incl stone	0.01 0.01	< 0.01 < 0.01	0.02 0.02	Report: R-4171 Study: -
Trial: AF/6772/NN/2 Dirmstein, Germany, 2002 (Auerbacher)	76.1	1522	1	10 14	Fruit incl stone Fruit incl stone	0.02 0.02	< 0.01 < 0.01	0.03 0.03	Report: R-4171 Study: -
Trial: AF/6772/NN/3 Meckesheim, Germany, 2002 (Schufer)	75.8	1515	1	10 14	Fruit incl stone Fruit incl stone	0.02 0.01	< 0.01 < 0.01	0.03 0.02	Report: R-4171 Study: -
Trial: AF/6772/NN/4 Lobenfeld, Germany, 2002 (Cacaks Schone)	76.8	1536	1	10 14	Fruit incl stone Fruit incl stone	0.02 0.02	< 0.01 < 0.01	0.03 0.03	Report: R-4171 Study: -
Trial: CA96 Parlier, CA, USA, 2010 (President)	113, 113 14d interval	832 842	2	7	Fruit w/o stone	< 0.05	< 0.05	< 0.10	Report: R-4481 Study: - (EC)
Trial: CA97 Parlier, CA, USA, 2010 (President)	114, 113 572, 570 572, 570 13d interval	1328 1328 1318	2	7	Fruit w/o stone Fruit w/o stone Fruit w/o stone (dried)	0.251,0.290, 0.271 1.84 3.18	< 0.05 0.208 0.316	0.301,0.340,0.321 2.048 3.496	Report: R-4481 Study: - (EC)
Trial: CA98 Parlier, CA, USA, 2010 (President)	114, 115 14d interval	813 822	2	7	Fruit w/o stone	< 0.05	< 0.05	< 0.10	Report: R-4481 Study: - (EC)
Trial: CA99 Davis, CA, USA, 2010 (President)	115, 115 14d interval	1150 1159	2	7	Fruit w/o stone	0.215,0.175 0.195	< 0.05	0.265,0.225 0.245	Report: R-4481 Study: - (EC)
Trial: MI28 Fennville, MI, USA, 2010 (President)	112, 112 15d interval	1066 1057	2	7	Fruit w/o stone	0.116,0.142 0.13	< 0.05	0.166,0.192 0.18	Report: R-4481 Study: - (EC)
Trial: OR23 Aurora, OR, USA, 2010 (President)	111, 111 14d interval	1141 1206	2	6	Fruit w/o stone	0.0804, 0.0866 0.08	< 0.05	0.1304, 0.1366 0.13	Report: R-4481 Study: - (EC)

Plums	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: S15-03091-01 Spain, 2015 (President)	78		1	0 1 3 8 14	Fruits	0.05 0.05 0.03 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.06 0.06 0.04 0.04 0.04	Report: R-4510 Study: S15-03091
Trial: S15-03091-02 Italy, 2015 (Anna Spath)	77		1	0 1 3 7 14	Fruits	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Report: R-4510 Study: S15-03091
Trial: S15-03091-03 Spain, 2015 (Angeleno)	76		1	14	Fruits	0.01	< 0.01	0.02	Report: R-4510 Study: S15-03091
Trial: S15-03091-04 S-France, 2015 (President)	73		1	14 26	Fruits	0.01 < 0.01	< 0.01 < 0.01	0.02 < 0.02	Report: R-4510 Study: S15-03091
Trial: S09-02264-01 Innenheim, Alsace, N-France, 2009 (Quetsche D'Alsace)	74.6	1491	1	0 3 7 10 14	Fruit Fruit Fruit Fruit Fruit	0.02 0.04 0.03 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.03 0.05 0.04 0.04 0.03	Report: R-4436 Study: S09-02264
Trial: S09-02264-03 Stotzheim, Alsace, N-France, 2009 (Elena)	74.0	1481	1	0 14	Fruit Fruit	0.04 0.03	< 0.01 < 0.01	0.05 0.04	Report: R-4436 Study: S09-02264
Trial: S09-02264-02 Calatorao, Zaragoza, Spain, 2009 (Anappar)	69.8	1395	1	0 3 7 10 14	Fruit Fruit Fruit Fruit Fruit	0.01 0.01 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 0.02 0.02 0.02	Report: R-4436 Study: S09-02264

Grapes

Table 192 Residues in Grapes (outdoor trails) from supervised trials in EU involving foliar applications (once) of Fenpyroximate 5% SC

Grapes	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: S362.90 S-France, 1989 (Carignan)	60		1	0 7 14 21 29	Fruit Fruit Fruit Fruit Fruit	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.04 < 0.04 < 0.04 < 0.04 < 0.04	Report: R-4044 Study: -

Grapes	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: S363.90 S-France, 1990 (Carignan)	80		1	0 7 14 21 30	Fruit Fruit Fruit Fruit Fruit	< 0.10 0.05 < 0.05 0.08 0.07	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.12 0.05 < 0.07 0.10 0.09	Report: R-4044 Study: -
Trial: S933.89 S-France, 1989 (Merlot)	60 80		1 1	36 36	Fruit Fruit	0.05 < 0.02	< 0.02 < 0.02	0.07 < 0.04	Report: R-4044 Study: -
Trial: S321.89 S-France, 1989 (Carignana)	60 80		1 1	47 47	Fruit Fruit	< 0.02 < 0.02	< 0.02 < 0.02	< 0.04 < 0.04	Report: R-4044 Study: -
Trial: S401.89 N-France, 1989 (Pinot Noir)	60		1	37 37	Fruit Wine	0.07 < 0.02	< 0.02 < 0.02	0.09 < 0.04	Report: R-4044 Study: -
	80		1	37 37	Fruit Wine	0.14 < 0.02	< 0.02 < 0.02	0.16 < 0.04	
Trial: S345.90 S-France, 1990 (Cinsault)	60		1	55 55	Fruit Wine	0.06 < 0.02	< 0.02 < 0.02	0.08 < 0.04	Report: R-4044 Study: -
	80		1	55 55	Fruit Wine	0.04 < 0.02	< 0.02 < 0.02	0.06 < 0.04	
Trial: S402.90 N-France, 1990 (Pinot Noir)	60		1	46 46	Fruit Wine	0.07 < 0.02	< 0.02 < 0.02	0.09 < 0.04	Report: R-4044 Study: -
	80		1	46 46	Fruit Wine	0.05 < 0.02	< 0.02 < 0.02	0.07 < 0.04	
Trial: S403.90 N-France, 1990 (Pinot Noir)	60		1	42 42	Fruit Wine	0.05 < 0.02	< 0.02 < 0.02	0.07 < 0.04	Report: R-4044 Study: -
	80		1	42 42	Fruit Wine	0.08 < 0.02	< 0.02 < 0.02	0.10 < 0.04	
Trial: 01 Faenza,Italy, 1991 (Albana)	81 165	1319 1319	1 1	14 14	Fruit Fruit	0.47 0.57	0.02 0.03	0.49 0.6	Report: R-4050 Study: A52051
Trial: 01 S. Martino in Riparotta, Italy, 1991 (Trebbiano)	91 188	1510 1498	1 1	14 14	Fruit Fruit	0.17 0.52	0.03 0.03	0.2 0.55	Report: R-4050 Study: A52051
Trial: - S. Martino in Riparotta, Italy, 1991 (Trebbiano)	94	1510	1	14	Fruit	0.17	0.01	0.18	Report: R-4052 Study: A50133
Trial: - S. Martino in Riparotta, Italy, 1991 (Trebbiano)	187.3	1498	1	14	Fruit	0.52	0.03	0.55	Report: R-4053 Study: A50132
Trial: - Trani, Italy, 1991 (Regina)	64.4	1000	1	14	Fruit	0.07	< 0.01	0.08	Report: R-4054 Study: A50131
Trial: - Trani, Italy, 1991 (Regina)	128.8	1000	1	14	Fruit	0.19	0.01	0.20	Report: R-4055 Study: A50134

Grapes	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: 01 Bollullos del Condado, Huelva, Spain, 1988 (Cardinal)	95.1	991	1	14	Fruit	0.04	-	0.04	Report: R-4110 Study: NHH/093
	96.7	1007	1	14	Fruit	0.06	-	0.06	
Trial: 02 Los Palacios, Seville, Spain, 1988 (Cardinal)	97.3	1014	1	14	Fruit	0.04	-	0.04	Report: R-4110 Study: NHH/093
	98.8	1029	1	14	Fruit	0.04	-	0.04	
Trial: 03 Granieri, Catania, Italy, 1988 (Italia)	51.3	1014	1	28	Fruit	0.04	-	0.04	Report: R-4110 Study: NHH/093 (protected)
	50.7	1002	1	28	Fruit	0.02	-	0.02	
Trial: 04 Mazzarrone, Catania, Italy, 1988 (Italia)	50.5	999	1	28	Fruit	0.04	-	0.04	Report: R-4110 Study: NHH/093 (protected)
	50.7	1002	1	28	Fruit	0.03	-	0.03	
Trial: S08-02423-01 Castel San Pietro Terme, Bologna, Italy, 2008 (Barbera)	51.2	1004	1	28	Fruit	< 0.01	< 0.01	< 0.02	Report: R-4205 Study: S08-02423
Trial: S08-02423-02 Sistels, Tarn-et-Garonne, France, 2008 (Merlot)	52.2	1024	1	0 7 14 21 28	Fruit Fruit Fruit Fruit Fruit	0.08 0.04 0.07 0.04 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.09 0.05 0.08 0.05 0.06	Report: R-4205 Study: S08-02423
Trial: S08-02423-03 Villaneuva del Ariscal, Sevilla, Spain, 2008 (Garrido Fino)	51.5	1009	1	28	Fruit	< 0.01	< 0.01	< 0.02	Report: R-4205 Study: S08-02423
Trial: S08-02423-05 Malejan, Aragon, Spain, 2008 (Garnacha)	51.1	1002	1	0 7 14 21 28	Fruit Fruit Fruit Fruit Fruit	0.08 0.08 0.05 0.05 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.09 0.09 0.06 0.06 0.04	Report: R-4205 Study: S08-02423
Trial: S09-02663-01 Poggio Grande, Bologna, Italy, 2009 (Sangiovese)	50.0	981.2	1	28	Fruit	0.01	< 0.01	0.02	Report: R-4219 Study: S09-02663
Trial: S09-02663-02 Grayssas, Lot Et Garonne, France, 2009 (Tammat)	54.4	1067	1	0 7 14 21 28	Fruit Fruit Fruit Fruit Fruit	0.12 0.11 0.11 0.07 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.13 0.12 0.12 0.08 0.06	Report: R-4219 Study: S09-02663

Grapes	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: S09-02663-03 Sinarcas, Valencia, Spain, 2009 (Bobal)	53.8	1055	1	28	Fruit	0.02	< 0.01	0.03	Report: R-4219 Study: S09-02663
Trial: S09-02663-04 Quatretande, Valencia, Spain, 2009 (Monastrell)	54.4	1067	1	0 7 14 21 29	Fruit Fruit Fruit Fruit Fruit	0.03 0.05 0.03 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.04 0.06 0.04 0.03 0.03	Report: R-4219 Study: S09-02663
Trial: S11-02944-03 Gertwiller, Alsace, France, 2011 (Gewurztraminer)	50.9	990	1	0 7 14 21 28	Fruit Fruit Fruit Fruit Fruit	0.06 0.06 0.04 0.05 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.07 0.07 0.05 0.06 0.05	Report: R-4469 Study: S11-02944
Trial: S11-02944-04 Heidelberg, Baden-Württemberg, Germany2011 (Müller-Thurgau)	50.5	983	1	0 7 14 21 28	Fruit Fruit Fruit Fruit Fruit	0.02 0.04 0.02 0.03 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.03 0.04 0.03 0.04 0.02	Report: R-4469 Study: S11-02944
Trial: S15-03093-01 N-France, 2015 (Pinot Auxerrois)	97			0 7 14 21 28	Fruit Fruit Fruit Fruit Fruit	0.16 0.08 0.03 0.04 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.17 0.09 0.04 0.05 < 0.02	Report: R-4504 Study: S15-03093
Trial: S15-03093-02 N-France, 2015 (Cabernet)	90			0 7 14 21 28	Fruit Fruit Fruit Fruit Fruit	0.17 0.07 0.08 0.08 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.18 0.08 0.09 0.09 0.08	Report: R-4504 Study: S15-03093
Trial: S15-03093-03 Germany, 2015 (Kemer)	91			0 7 14 21 28	Fruit Fruit Fruit Fruit Fruit	0.05 0.03 0.02 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.06 0.04 0.03 0.04 0.03	Report: R-4504 Study: S15-03093
Trial: S15-03093-04 Germany, 2015 (Reisling)	85			0 7 14 21 28	Fruit Fruit Fruit Fruit Fruit	0.20 0.04 0.07 0.06 0.01	< 0.01 < 0.01 0.01 < 0.01 < 0.01	0.21 0.05 0.08 0.07 0.02	Report: R-4504 Study: S15-03093
Trial: S15-03093-05 N-France, 2015 (Gewurztraminer)	84			28	Fruit	0.04	< 0.01	0.05	Report: R-4504 Study: S15-03093
Trial: S15-03093-06 N-France, 2015 (Cabernet Sauvignon)	92			28	Fruit	0.08	< 0.01	0.09	Report: R-4504 Study: S15-03093

Grapes	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: S15-03093-07 Germany, 2015 (Reisling)	89			28	Fruit	0.05	< 0.01	0.06	Report: R-4504 Study: S15-03093

Table 193 Residues in Grapes (outdoor trails) from supervised trials in EU involving foliar applications (twice) of Fenpyroximate 5% SC

Grapes	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: W-6507 Ingelheim, Germany, 1990 (Portugeiser)	45 135	300 600	2	0 7 14 28 35	Fruit Fruit Fruit Fruit Fruit	0.17 0.18 0.10 0.07 0.08	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.22 0.23 0.15 0.12 0.13	Report: R-4168 Study: ER90DEU803
Trial: W-6909 Muehlhausen, Krg, Germany, 1990 (Spätbergunder)	45 135	300 600	2	0 7 14 28 35	Fruit Fruit Fruit Fruit Fruit	0.24 0.17 0.19 0.24 0.15	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.29 0.22 0.24 0.29 0.20	Report: R-4168 Study: ER90DEU803
Trial: 020102 Nittel, Germany, 1991 (Müller-Thurgau)	45 135		2	0 7 14 28 35	Fruit Fruit Fruit Fruit Fruit	0.22 0.16 0.16 0.10 0.08	0.02 0.02 0.02 0.01 0.01	0.24 0.18 0.18 0.11 0.09	Report: R-4067 Study: ER91DEU814
Trial: 020202 Mühlhofen, Germany, 1991 (Kerner)	45 135		2	0 7 14 28 35	Fruit Fruit Fruit Fruit Fruit	0.36 0.29 0.17 0.12 0.11	0.04 0.03 0.02 0.02 0.01	0.4 0.32 0.19 0.14 0.12	Report: R-4067 Study: ER91DEU814
Trial: 0201 Wachenheim, Germany, 1990 (Riesling)	45 135	300 600	2	0 7 14 28 35	Fruit Fruit Fruit Fruit Fruit	0.13 0.13 0.09 0.14 0.09	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.18 0.18 0.14 0.19 0.14	Report: R-4069 Study: ER90DEU805
Trial: 0202 Opfingen, Germany, 1990 (Müller-Thurgau)	45 135	300 600	2	0 7 14 28 35	Fruit Fruit Fruit Fruit Fruit	0.15 0.17 0.14 0.08 0.08	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.20 0.22 0.19 0.13 0.13	Report: R-4069 Study: ER90DEU805
Trial: 21 Pfeddersheim, Germany, 1989 (Schwartz-reisling)	180 180	600 600	2	0 7 14 28 35 35 35 35	Fruit Fruit Fruit Fruit Fruit Must Young wine Mature wine	0.20 0.145 0.14 0.21 0.15 < 0.10 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.21 0.155 0.15 0.22 0.16 < 0.11 < 0.02 < 0.02	Report: R-4102 Study: DEU89I803

Grapes	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: 22 Willsbach, Germany, 1989 (Müller-Thurgau)	180 180	600 600	2	0 7 14 28 35 35 35 35	Fruit Fruit Fruit Fruit Fruit Must Young wine Mature wine	0.19 0.18 0.24 0.16 0.135 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.20 0.19 0.25 0.17 0.145 0.03 < 0.02 < 0.02	Report: R-4102 Study: DEU89I803
Trial: 21 Muβbach, Germany, 1989 (Reisling)	180 180	600 600	2	0 7 14 28 35	Fruit Fruit Fruit Fruit Fruit	0.26 0.16 0.10 0.13 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.27 0.17 0.11 0.14 0.14	Report: R-4103 Study: DEU89I805
Trial: 22 Kappelrodeck, Germany, 1989 (Müller-Thurgau)	180 180	600 600	2	0 7 14 28 35	Fruit Fruit Fruit Fruit Fruit	0.29 0.30 0.18 0.12 0.16	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.30 0.31 0.19 0.13 0.17	Report: R-4103 Study: DEU89I805
Trial: 21 Muβbach, Germany, 1989 (Reisling)	135 135	600 600	2	0 7 14 28 35	Fruit Fruit Fruit Fruit Fruit	0.17 0.12 0.11 0.09 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.18 0.13 0.12 0.10 0.07	Report: R-4104 Study: DEU89I813
Trial: 22 Kappelrodeck, Germany, 1989 (Spätbergunder)	135 135	600 600	2	0 7 14 28 35	Fruit Fruit Fruit Fruit Fruit	0.41 0.41 0.31 0.32 0.40	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.42 0.42 0.32 0.33 0.41	Report: R-4104 Study: DEU89I813
Trial: 0201 Ingelhelm, Germany, 1990 (Portugeiser)	45 135	300 600	2	0 7 14 28 35	Fruit Fruit Fruit Fruit Fruit	0.17 0.18 0.10 0.07 0.08	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.22 0.23 0.15 0.12 0.13	Report: R-4168 Study: ER90DEU803
Trial: 0201 Muehlhausen, Germany, 1990 (Spätbergunder)	45 135	300 600	2	0 7 14 28 35	Fruit Fruit Fruit Fruit Fruit	0.24 0.17 0.19 0.24 0.15	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.29 0.22 0.24 0.29 0.20	Report: R-4168 Study: ER90DEU803

Table 194 Residues in Grapes (protected trails) from supervised trials in Japan involving foliar applications of Fenpyroximate 5% SC

Grape	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: Ishikawa Japan, 1989 (Delaware)	200	400 L/10a	1	14 21 30 60	Fruit Fruit Fruit Fruit	0.394 0.432 0.349 0.060	< 0.005 0.012 0.010 0.006	0.399 0.444 0.359 0.066	Report: R-4059 + R-4285 Study: NN025-01 Study not to GLP
Trial: Fukui Japan, 1989 (Delaware)	200	400 L/10a	1	13 20 29	Fruit Fruit Fruit	0.430 0.512 0.502	< 0.005 < 0.005 0.010	0.435 0.517 0.512	Report: R-4060 + R-4288 Study: NN025-02

Grape	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
	2 x 200	400 L/10a	2	13 20	Fruit Fruit	1.15 1.14	0.014 0.014	1.164 1.154	Study not to GLP
Trial: Ishikawa Japan, 1988 (Delaware)	200	400 L/10a	1	14 21 30 60	Fruit Fruit Fruit Fruit	0.311 0.260 0.244 0.040	- - - -	0.311 0.260 0.244 0.040	Report: R-4291 Study: NN025-04 Study not to GLP
Trial: Fukui Japan, 1988 (Delaware)	200	400 L/10a	1	13 20 29	Fruit Fruit Fruit	0.430 0.444 0.408	- - -	0.430 0.444 0.408	Report: R-4291 Study: NN025-04 Study not to GLP
	2 x 200	400 L/10a	2	13 20	Fruit Fruit	0.928 0.848	- -	0.928 0.848	
Trial: Ishikawa Japan, 1988 (Delaware)	200	400 L/10a	1	14 21 30 60	Fruit Fruit Fruit Fruit	- - - -	0.006 0.007 0.009 < 0.005	0.006 0.007 0.009 < 0.005	Report: R-4291 Study: NN025-04 Study not to GLP
Trial: Fukui Japan, 1988 (Delaware)	200	400 L/10a	1	13 20 29	Fruit Fruit Fruit	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	Report: R-4291 Study: NN025-04 Study not to GLP
	2 x 200	400 L/10a	2	13 20	Fruit Fruit	- -	0.010 0.010	0.010 0.010	

Table 195 Residues in Grapes (outdoor trails) from supervised trials in USA involving foliar applications of Fenpyroximate 5% EC

Grape	Application			DALA	Commodity	Residues (mg/kg)		Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	
Trial: NY01 Dundee, NY, USA, 1999 (DeChaunac)	224	377	1	14	Fruit	0.220,0.252 0.236 (sum of fenpyroximate + M-1)		Report: R-4121 Study: 826W
Trial: NY02 Dundee, NY, USA, 1999 (Concord)	224	377	1	14	Fruit	0.192,0.282 0.187 (sum of fenpyroximate + M-1)		Report: R-4121 Study: 826W
Trial: CA01 Fresno, CA, USA, 1999 (Thompson seedless)	224	376	1	14	Fruit	0.114,0.099 0.107 (sum of fenpyroximate + M-1)		Report: R-4121 Study: 826W
Trial: CA02 Fresno, CA, USA, 1999 (Thompson seedless)	224	386	1	14	Fruit	0.166,0.129 0.148 (sum of fenpyroximate + M-1)		Report: R-4121 Study: 826W
Trial: CA03 Fresno, CA, USA, 1999 (Thompson seedless)	224	355	1	14	Fruit	0.108,0.140 0.124 (sum of fenpyroximate + M-1)		Report: R-4121 Study: 826W
Trial: CA04 Fresno, CA, USA, 1999 (Thompson seedless)	224	384	1	14	Fruit	0.170,0.058 0.114 (sum of fenpyroximate + M-1)		Report: R-4121 Study: 826W

Grape	Application			DALA	Commodity	Residues (mg/kg)		Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	
Trial: CA05 Fresno, CA, USA, 1999 (Thompson seedless)	224	378	1	14	Fruit	0.092,0.093 0.093 (sum of fenpyroximate + M-1)		Report: R-4121 Study: 826W
Trial: CA06 Porterville, CA, USA, 1999 (Thompson seedless)	224	385	1	14	Fruit	< 0.05, < 0.05 < 0.05 (sum of fenpyroximate + M-1)		Report: R-4121 Study: 826W
Trial: CA07 Porterville, CA, USA, 1999 (Flame seedless)	224	376	1	14	Fruit	< 0.05, < 0.05 < 0.05 (sum of fenpyroximate + M-1)		Report: R-4121 Study: 826W
Trial: CA08 Manteca, CA, USA, 1999 (Palomino Wine)	224	374	1	14	Fruit	0.060, < 0.05 < 0.05 (sum of fenpyroximate + M-1)		Report: R-4121 Study: 826W
Trial: ID01 Payette, ID, USA, 1999 (Concord)	224	381	1	14	Fruit	0.091,0.056 0.074 (sum of fenpyroximate + M-1)		Report: R-4121 Study: 826W
Trial: WA01 Ephrata, WA, USA, 1999 (White Reisling)	224	375	1	14	Fruit	0.093,0.074 0.084 (sum of fenpyroximate + M-1)		Report: R-4121 Study: 826W
Trial: CA01 Fresno, CA, USA, 1999 (Thompson seedless)	1120	381	1	14	Fruit Juice Dry pomace Wet pomace Raisins Raisin waste	0.511,0.381, 0.446 < 0.05 4.266 1.251 1.212 1.580 (sum of fenpyroximate + M-1)		Report: R-4122 Study: 827W

Raspberries

Table 196 Residues in Raspberry (outdoor trails) from supervised trials in EU involving foliar applications of Fenpyroximate (5% SC formulation)

Raspberry	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: S09-02709-01 Pithiviers-le-Vieil, Loiret, France, 2009 (Heritage)	77.8	1000	1	0 3 7 10 14	Fruits	0.16 0.15 0.10 0.05 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.17 0.16 0.11 0.06 0.05	Report: R-4435 Study: -
Trial: S09-02709-01 Ruschwedel, Lower Saxony, Germany, 2009 (Erika)	76.7	1000	1	0 3 6 9 14	Fruits	0.23 0.18 0.18 0.12 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.24 0.19 0.19 0.13 0.09	Report: R-4435 Study: -

Raspberry	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: S10-02527-01 Heldelberg, Baden-Würtemberg, Germany, 2010 (Malling Promise)	73.0	1000	1	0 14	Fruits	1.09 0.10	< 0.01 < 0.01	1.10 0.11	Report: R-4457 Study: -
Trial: S10-02527-01 Nottensdorf, Niedersachsen, Germany, 2010 (Glen Ample)	77.0	1000	1	0 3 7 10 14	Fruits	0.14 0.07 0.03 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.15 0.08 0.04 0.03 0.02	Report: R-4457 Study: -

Strawberries

Table 197 Residues in Strawberries (outdoor trails) from supervised trials in EU involving foliar applications of Fenpyroximate 5% SC

Strawberries	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Boara, Italy, 1991 (Hanoy)	154.5	1000	1	0 14 21 28	Fruit Fruit Fruit Fruit	0.10 0.03 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.11 0.04 0.02 < 0.02	Report: R-4142 Study: A50814 Study not to GLP
North Wheatley, Nottinghamshire, UK, 2001 (Elsanta)	98.8	964	1	0 3 7 10	Fruit Fruit Fruit Fruit	0.04 0.02 <u>0.03</u> 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.05 0.03 0.04 0.03	Report: R-4162 Study: AF/6011/NN
Tillington, Hereford and Worcestershire, UK, 2001 (Symphony)	105.7	1031	1	0 3 6	Fruit Fruit Fruit	0.01 0.04 <u>0.02</u>	< 0.01 < 0.01 < 0.01	0.02 0.05 0.03	Report: R-4162 Study: AF/6011/NN
Kings Lynn, Cambridgeshire, UK, 2001 (Elsanta)	101.9	750	1	3 7	Fruit Fruit	0.02 <u>< 0.01</u>	0.01 < 0.01	0.02 < 0.02	Report: R-4162 Study: AF/6011/NN
Walpole St. Andrew, Norfolk, UK, 2001 (Elsanta)	104.9	750	1	3 7	Fruit Fruit	0.05 <u>0.01</u>	< 0.01 < 0.01	0.06 0.02	Report: R-4162 Study: AF/6011/NN
Balignac, Tarn-et-Garonne, France, 2001 (Siloue)	98.1	761	1	0 3 7 10	Fruit Fruit Fruit Fruit	0.38 0.17 <u>0.05</u> 0.05	0.03 < 0.01 0.01 < 0.01	0.38 0.18 0.05 0.06	Report: R-4162 Study: AF/6011/NN
Labastide-du-Temple, Tarn-et-Garonne, France, 2001 (Tetis)	102.0	979	1	3 7	Fruit Fruit	0.04 <u>0.01</u>	0.01 0.01	0.04 0.01	Report: R-4162 Study: AF/6011/NN

Strawberries	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Argelato, Emilia Romagna, Italy, 2001 (Marmolada)	100.5	980	1	0 3 7 10	Fruit Fruit Fruit Fruit	< 0.01 < 0.01 <u>< 0.01</u> < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02	Report: R-4162 Study: AF/6011/NN
Granarolo, Emilia Romagna, Italy, 2001 (Selva)	100.7	737	1	3 7	Fruit Fruit	0.09 <u>0.05</u>	0.02 0.01	0.09 0.05	Report: R-4162 Study: AF/6011/NN
Hom Green, Herefordshire, UK, 2003 (Everest)	103	1006	1	3 7	Fruit Fruit	0.10 <u>0.07</u>	< 0.01 < 0.01	0.11 0.08	Report: R-4167 Study: AF/7640/NN
North Wheatley, Nottinghamshire, UK, 2003 (Flamenco)	102	990	1	3 7	Fruit Fruit	0.19 <u>0.08</u>	< 0.01 < 0.01	0.20 0.09	Report: R-4167 Study: AF/7640/NN
Elmhurst, Staffordshire, UK, 2003 (Everest)	103	1001	1	0 3 7 10	Fruit Fruit Fruit Fruit	0.18 0.12 <u>0.19</u> 0.06	< 0.01 < 0.01 < 0.01 < 0.01	0.19 0.13 0.20 0.07	Report: R-4167 Study: AF/7640/NN
Gnosall, Staffordshire, UK, 2003 (Diamante)	102.3	998	1	0 3 7 10	Fruit Fruit Fruit Fruit	0.24 0.20 <u>0.06</u> 0.05	< 0.01 < 0.01 < 0.01 < 0.01	0.25 0.21 0.07 0.06	Report: R-4167 Study: AF/7640/NN
Labarthe, S-France, 2002 (Ciloe)	101.7	992	1	3 7 7 7 7 7 7 7	Fruit Fruit Whole fruit Washed fruit Wash water Jam Blanch water Canned fruit	0.23 <u>0.13</u> 0.105 0.075 0.01 0.01 < 0.01 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.24 0.14 0.115 0.085 0.02 0.02 < 0.02 0.04	Report: R-4177 Study: AF/6783/NN
Puygaillard de Lomagne, S-France, 2002 (Ciloe)	99.7	973	1	3 7 7 7 7 7 7 7	Fruit Fruit Whole fruit Washed fruit Wash water Jam Blanch water Canned fruit	0.08 <u>0.05</u> 0.045 0.04 < 0.01 0.01 < 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.09 0.06 0.055 0.05 < 0.02 0.02 < 0.02 0.03	Report: R-4177 Study: AF/6783/NN
Balignac, S-France, 2002 (Valeta)	106	1036	1	0 3 7 10 7 7 7 7 7 7	Fruit Fruit Fruit Fruit Whole fruit Washed fruit Wash water Jam Blanch water Canned fruit	0.11 0.06 <u>0.10</u> 0.08 0.07 0.05 0.01 0.01 < 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.12 0.07 0.11 0.09 0.08 0.06 0.02 0.02 < 0.02 0.03	Report: R-4177 Study: AF/6783/NN
Svoronos, Pieria, Greece, 2002 (Diamant)	98	960	1	0 3 7 10	Fruit Fruit Fruit Fruit	0.10 0.14 <u>0.07</u> 0.05	< 0.01 < 0.01 < 0.01 < 0.01	0.11 0.15 0.08 0.06	Report: R-4177 Study: AF/6783/NN

Strawberries	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Italy, 2011 (Nayad)	100.88		2	0 1 3 5 7	Fruit Fruit Fruit Fruit Fruit	0.28 0.18 0.21 0.10 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.29 0.19 0.22 0.11 0.06	Report: R-4449 Study: 52003A001

Table 198 Residues in Strawberries (outdoor trails) from supervised trials in USA involving foliar applications of Fenpyroximate 5% EC

Strawberries	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Oviedo, FL, USA, 2008 (Camarosa)	109 + 112	3.26	2	1	Fruit	0.614,0.383 0.499	0.081,0.049 0.065	0.695,0.432 0.564	Report: R-4233 + R-4445 Study: 52003A001
	109 + 112	3.26	2	1	Fruit	0.590,0.473 0.532	0.015,0.014 0.015	0.605,0.487 0.547	
Leggett, Porterville, CA, USA, 2008 (Diamante)	111 + 112	5.68	2	1	Fruit	0.293,0.265 0.279	0.057,0.050 0.054	0.350,0.315 0.333	Report: R-4233 + R-4445 Study: 52003A001
	112 + 112	5.68	2	1	Fruit	0.182,0.185 0.184	0.003,0.005 0.004	0.185,0.190 0.188	
Salinas, CA, USA, 2008 (Albion)	112 + 113	5.5	2	1	Fruit	0.238,0.178 0.208	0.017,0.012 0.015	0.255,0.190 0.223	Report: R-4233 + R-4445 Study: 52003A001
	111 + 116	5.5	2	1	Fruit	0.242,0.233 0.238	0.005,0.005 0.005	0.247,0.238 0.243	
East Williamson, NY, USA, 2010 (Idea)	112+113 112+112 113+113 113+113	281	2	1	Fruit	0.0371,0.0503 0.04 0.0787,0.0248 0.05 0.0575,0.0610 0.06 0.0746,0.0715 0.07	< 0.01 < 0.01 < 0.01 < 0.01	0.0421,0.06609,0.05 0.0891,0.03831,0.06 0.06327,0.0749,0.07 0.08397,0.092, 0.08	Report: R-4438 Study: TCI-10-273 1=no surfactant 2=InduceNIS 3=Dyme-Amic MSO 4=Silwet L77 Organosilicone
Sever Springs, NC, USA, 2010 (Camino Real)	113+109 112+112 111+113 113+112	413	2	1 1 1 1	Fruit Fruit Fruit Fruit	0.0947,0.104, 0.10 0.163,0.181, 0.17 0.105,0.173, 0.14 0.213,0.159, 0.19	< 0.01 < 0.01 < 0.01 < 0.01	0.10873,0.1192,0.11 0.17864,0.1989,0.18 0.11794,0.1900 0.15 0.22138,0.16430. 20	
St. Stephen, MN, USA, 2010 (Honeyoye)	112+112 111+111 112+111 112+111	234	2	1 1 1 1	Fruit Fruit Fruit Fruit	0.0607,0.04110 .05 0.0298,0.03290 .03 0.0703,0.05780 .06 0.0199,0.04320 .03	ND ND ND ND	0.0607,0.04110 .05 0.0298,0.03290 .03 0.0703,0.05780 .06 0.0199,0.04320 .03	
Salinas, CA, USA, 2010 (Albion)	112+114 113+114 112+114 114+113	464	2	1 1 1 1	Fruit Fruit Fruit Fruit	0.164,0.178, 0.17 0.216,0.209, 0.21 0.230,0.249, 0.24 0.176,0.242, 0.21	0.04 0.04 0.03 0.03	0.1965,0.21560 .21 0.2533,0.24480 .25 0.2545,0.275,0. 27 0.2089,0.27570 .24	

Strawberries	Application			DALA	Comm	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)	odity	Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Elmira, OR, USA, 2010 (Benton)	115+119 114+117 112+117 115+118	241	2	1 1 1 1	Fruit Fruit Fruit Fruit	0.201,0.179, 0.19 0.197,0.180, 0.19 0.101,0.150, 0.13 0.135,0.228, 0.18	0.0342,0.0292 0.03 0.0218,0.0246 0.02 0.00915,0.009 0.01 0.0159,0.0342 0.03	0.2352,0.2082, 0.22 0.2188,0.2046, 0.21 0.11015,0.1596, 0.14 0.1509,0.2622, 0.21	

Table 199 Residues in Strawberries (protected trails) from supervised trials in EU involving foliar applications of Fenpyroximate 5% SC

Strawberries	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Kings Bromley, Staffordshire, UK, 2002 (Elsanta)	100.9	984	1	0 3 7 10	Fruit Fruit Fruit Fruit	0.04 0.06 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.05 0.07 0.04 0.03	Report: R-4157 Study: AF/6776/NN
Meauzac, Tarn-et-Garonne, Southern France, 2002 (Mara des Bois)	103.8	1013	1	0 3 7 10	Fruit Fruit Fruit Fruit	0.08 0.08 0.04 0.07	< 0.01 < 0.01 < 0.01 < 0.01	0.09 0.09 0.05 0.08	Report: R-4157 Study: AF/6776/NN
Burntwood, Staffordshire, UK, 2002 (Elsanta)	102.9	1004	1	3 7	Fruit Fruit	0.03 0.07	< 0.01 < 0.01	0.04 0.08	Report: R-4157 Study: AF/6776/NN
Vazerac, Tarn-et-Garonne, Southern France, 2002 (Tiara des Bois)	102.1	996	1	3 7	Fruit Fruit	0.07 0.04	< 0.01 < 0.01	0.08 0.05	Report: R-4157 Study: AF/6776/NN
Cendieux, Dordogne, Southern France, 2002 (Aromace)	97.3	949	1	3 7	Fruit Fruit	0.15 0.12	< 0.01 < 0.01	0.16 0.13	Report: R-4157 Study: AF/6776/NN
Halam, Nottinghamshire, UK, 2001 (Elsanta)	102.7	922	1	0 3 7 10	Fruit Fruit Fruit Fruit	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02	Report: R-4163 Study: AF/6026/NN
Puygaillard de Lomagne, Tarn-et-Garonne, Southern France, 2001 (Gariguette)	105.1	761	1	0 3 7 10	Fruit Fruit Fruit Fruit	0.28 0.07 0.08 0.03	0.02 0.01 0.01 0.01	0.3 0.08 0.09 0.04	Report: R-4163 Study: AF/6026/NN
Meauzac, Tarn-et-Garonne, Southern France, 2001 (Miranda)	104.1	1016	1	3 7	Fruit Fruit	0.08 0.07	0.01 0.01	0.09 0.08	Report: R-4163 Study: AF/6026/NN
Argelato, Emilia Romagna, Italy, 2001 (Miss)	98.8	858	1	3 7	Fruit Fruit	0.13 0.08	0.01 0.01	0.14 0.09	Report: R-4163 Study: AF/6026/NN

Assorted tropical and sub-tropical fruit–inedible peel

Avocado

Table 200 Residues in Avocados (outdoor trails) from supervised trials in USA involving foliar applications of Fenpyroximate 5% SC

Avocados	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Paso Robles, CA, USA, 2008 (Haas)	111, 112 14d interval	1788	2	1	Fruit	0.06	-	0.06	Report: R-4459 Study: 10007
Paso Robles, CA, USA, 2008 (Haas)	112, 112 13d interval	1677	2	1	Fruit	< 0.05	-	< 0.05	Report: R-4459 Study: 10007
Parlier, CA, USA, 2009 (Haas)	115, 114 17d interval	1280	2	1	Fruit	< 0.05	-	< 0.05	Report: R-4459 Study: 10007
Parlier, CA, USA, 2009 (Haas)	115, 114 14d interval	1631	2	1	Fruit	< 0.05	-	< 0.05	Report: R-4459 Study: 10007
Homestead, FL, USA, 2009 (Simmonds)	102, 102 14d interval	1369	2	1	Fruit	0.10	-	0.10	Report: R-4459 Study: 10007

Papaya

Table 201 Residues in Papaya (outdoor trails) from supervised trials in Brazil involving foliar applications of Fenpyroximate 5% SC

Papaya	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Linhares (ES), Brazil, 2002 (Golden)	50 + 50 14 day interval	1000	2	0 3 5 7 14	Fruit Fruit Fruit Fruit Fruit	0.06 0.2 0.2 0.2 0.2	- - - - -	0.06 0.2 0.2 0.2 0.2	Report: R-4496 Study: 74/4534/03 Study not to GLP
	100 + 100 14 day interval	1000	2	0 3 5 7 14	Fruit Fruit Fruit Fruit Fruit	< 0.05 0.2 0.2 0.3 0.3	- - - - -	< 0.05 0.2 0.2 0.3 0.3	
Santa Fe do Sul (SP), Brazil, 1997/1998 (Papaya)	100 + 100 14 day interval	1000	2	0 3 5 7 14	Fruit Fruit Fruit Fruit Fruit	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1	- - - - -	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1	Report: R-4496 Study: - Study not to GLP
	30 + 30 14 day interval	600	2	0 3 5 7 14	Fruit Fruit Fruit Fruit Fruit	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1	- - - - -	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1	

Papaya	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country,year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Ilha Solteira (SP), Brazil,1997/1998 (Papaya)	60 + 60 14 day interval	600	2	0 3 5 7 14	Fruit Fruit Fruit Fruit Fruit	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1	- - - - -	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1	Report: R-4496 Study: - Study not to GLP
	40 + 40 14 day interval	800	2	0 3 5 7 14	Fruit Fruit Fruit Fruit Fruit	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1	- - - - -	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1	

Fruiting vegetables, Cucurbits

Cucumber

Table 202 Residues in cucumber from supervised trials in EU, Japan and USA involving foliar applications of Fenpyroximate 5%SC or 5%EC.

Cucumber	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Study: A48128 Cheste SPAIN, 1990 (Dinastic 2) (Protected)	103	2000	1	0 3 7 10	Fruit Fruit Fruit Fruit	0.12 0.04 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.13 0.05 0.02 0.02	Report: R-4036 5%SC
Study: A48129 Cheste SPAIN,1990 (Dinastic 2) (Protected)	154.5	2000	1	0 3 7 14	Fruit Fruit Fruit Fruit	0.09 0.06 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.10 0.07 0.03 0.02	Report: R-4037 5%SC
Trial: AF/6097/NN/2 Montauban S FRANCE,2001 (Defence) (Protected)	108.5	1059	1	0 3 7 10	Fruit Fruit Fruit Fruit	0.03 0.02 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.04 0.03 0.03 < 0.02	Report: R-4172, Study: AF/6097/NN 5%SC
Trial: AF/6097/NN/3 St Jory S FRANCE, 2001 (1976 Bruisma) (Protected)	102.9	1004	1	7 10	Fruit Fruit	0.02 < 0.01	< 0.01 < 0.01	0.03 < 0.02	
Trial: AF/6097/NN/4 Riolo Terme ITALY, 2001 (Darina) (Protected)	107.3	1047	1	7 10	Fruit Fruit	< 0.01 < 0.01	< 0.01 < 0.01	< 0.02 < 0.02	
Trial: AF/6779/NN/01 Los Palacio y Villafranca Seville SPAIN,2002 (not stated) (Protected)	103.5	1010	1	Post T1 3 7 10	Fruit Fruit Fruit Fruit	0.04 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.05 0.02 < 0.02 < 0.02	Report: R-4173 Study: AF/6779/NN 5%SC
Trial: AF/6779/NN/02 Burstwick, East Yorkshire UK,2002 (Amaada) (Protected)	105	1025	1	Post T1 3 7 10	Fruit Fruit Fruit Fruit	0.03 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.04 0.03 < 0.02 < 0.02	

Cucumber	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: AF/6779/NN/03 Beverley, East Yorkshire UK,2002 (Aviance) (Protected)	110	1069	1	Post T1 3 7 10	Fruit Fruit Fruit Fruit	0.04 0.03 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.05 0.04 0.03 < 0.02	
Trial: AF/6779/NN/04 Keyingham, East Yorkshire UK,2002 (Korinda) (Protected)	90	881	1	7 10	Fruit Fruit	< 0.01 < 0.01	< 0.01 < 0.01	< 0.02 < 0.02	
Trial: AF/6779/NN/05 Los Palacio y Villafranca, Sevilla SPAIN,2002 (Dona) (Protected)	103	1000	1	7 10	Fruit Fruit	< 0.01 < 0.01	< 0.01 < 0.01	< 0.02 < 0.02	
Trial: AF/6779/NN/06 Rieux, Orgueil S FRANCE,2002 (Beluga) (Protected)	103	1008	1	7 10	Fruit Fruit	0.01 < 0.01	< 0.01 < 0.01	0.02 < 0.02	
Trial: Gunma A Gunma JAPAN, 1989 (Sharp One) (Protected)	100	2000	1	1 3 7	Fruit Fruit Fruit	0.058 0.029 0.008	- - -	0.058 0.029 0.008	Report: R-4348 Study: NN028-01 -Study not to GLP 5%EC Refer also to 5.6.1/08 5%EC
	50	2000	1	1 3 7	Fruit Fruit Fruit		- - -	0.019 0.014 0.006	
Study NN028-01 Trial Kochi A Kochi JAPAN, 1989 (Sharp One) (Protected)	100	2000	1	1 3 7	Fruit Fruit Fruit	0.105 0.026 0.006	- - -	0.105 0.026 0.006	
	50	2000	1	1 3 7	Fruit Fruit Fruit	0.040 0.010 < 0.005	- - -	0.040 0.010 < 0.005	
Trial: Gunma A Gunma JAPAN, 1989 (Sharp One) (Protected)	100	2000	1	1 3 7	Fruit Fruit Fruit	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	R-4349, NN028-02 -Study not to GLP 5%EC -Study not to GLP Refer also to 5.6.1/07
	50	2000	1	1 3 7	Fruit Fruit Fruit	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	
Trial Kochi A Kochi JAPAN, 1989 (Sharp One) (Protected)	100	2000	1	1 3 7	Fruit Fruit Fruit	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	
	50	2000	1	1 3 7	Fruit Fruit Fruit	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	
Trial Gunma A Gunma JAPAN, 1990 (Sharp One) (Protected)	100	2000	1	1 3 7	Fruit Fruit Fruit	0.028 0.028 < 0.005	- - -	0.028 0.028 < 0.005	Report: R-4350 Study NN028-03 -Study not to GLP 5%EC Refer also to 5.6.1/10
	50	2000	1	1 3 7	Fruit Fruit Fruit	< 0.005 0.008 < 0.005	- - -	< 0.005 0.008 < 0.005	
Study NN028-03 Trial Kochi A Kochi JAPAN, 1990 (Sharp One) (Protected)	100	2000	1	1 3 7	Fruit Fruit Fruit	0.084 0.020 < 0.005	- - -	0.084 0.020 < 0.005	
	50	2000	1	1 3 7	Fruit Fruit Fruit	0.038 < 0.005 < 0.005	- - -	0.038 < 0.005 < 0.005	

Cucumber Trial Location Country, year (Variety)	Application Rate (g ai/ha)	 Water (L/ha)	 No.	DALA (days)	Commodity	Residues (mg/kg) Fenpyroximate	 M-1	 Sum of fenpyroximate and M-1	Reference & Comments
Trial: Gunma A Gunma JAPAN, 1990 (Sharp One) (Protected)	100	2000	1	1 3 7	Fruit Fruit Fruit	- - -	1 3 7		Report: R-4351 Study: NN028-04 -Study not to GLP 5%EC Refer also to 5.6.1/09
	50	2000	1	1 3 7	Fruit Fruit Fruit	- - -	1 3 7		
Trial: Kochi A Kochi JAPAN, 1990 (Sharp One) (Protected)	100	2000	1	1 3 7	Fruit Fruit Fruit	- - -	< 0.005 0.006 < 0.005	< 0.005 0.006 < 0.005	
	50	2000	1	1 3 7	Fruit Fruit Fruit	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	
Trial: Chiba A Chiba JAPAN, 1997 (Sharp One) (Protected)	50	2000	1	1	Fruit	0.046	< 0.005	0.051	Report: R-4404 Study: NN028-05 -Study not to GLP -Study carried out in 1997 5%EC
	80	2000	1	1 3 7	Fruit Fruit Fruit	0.118 0.066 0.015	< 0.005 < 0.005 < 0.005	0.123 0.071 0.020	
Study: NN028-05 Trial: Miyazaki A Miyazaki JAPAN, 1997 (Suisei Fuchinari #2) (Protected)	80	2000	1	1 3 7	Fruit Fruit Fruit	0.091 0.048 0.018	< 0.005 < 0.005 < 0.005	0.096 0.053 0.023	
Trial: Chiba A Chiba JAPAN, 1997 (Sharp One) (Protected)	80	2000	1	1 3 7	Fruit Fruit Fruit	0.110 0.076 0.022	< 0.005 < 0.005 < 0.005	0.115 0.081 0.027	Report: R-4405 Study: NN028-06 -Study not to GLP 5%EC
	50	2000	1	1	Fruit	0.061	< 0.005	0.066	
Trial: Miyazaki A Miyazaki JAPAN, 1997 (Suisei Fuchinari #2) (Protected)	120	3000	1	1 3 7	Fruit Fruit Fruit	0.086 0.046 0.019	< 0.005 < 0.005 < 0.005	0.091 0.051 0.024	
	75	3000	1	1	Fruit	0.057	< 0.005	0.062	
Gunma JAPAN, 1997 (Honor) (Protected)	80	2000	1	1 3 7	Fruit Fruit Fruit	0.076 0.032 0.010	< 0.005 < 0.005 < 0.005	0.081 0.037 0.015	Report: R-4406 Study: NN028-07 -Study not to GLP 5%EC
Trial Aichi A Aichi JAPAN, 1997 (Sharp 301) (Protected)	120	3000	1	1 3 7	Fruit Fruit Fruit	0.182 0.081 0.026	< 0.005 < 0.005 < 0.005	0.187 0.086 0.031	
Trial: S15-03095-03 El Viso del Alcor, Seville, SPAIN, 2015 (Alanis)	104.2	833	1	0 1 3 5 7	Fruit Fruit Fruit Fruit Fruit	0.02 0.02 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.03 0.03 < 0.02 < 0.02 < 0.02	Report: R-4511 Study: S15-03095 5%SC
Trial: S15-03095-04 Monte San Biagio, ITALY, 2015 (Ekron)	105.6	1056	1	0 1 3 5 7	Fruit Fruit Fruit Fruit Fruit	0.06 0.07 0.03 0.04 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.07 0.08 0.04 0.05 0.03	

Cucumber	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: S15-03095-05 Saint-Laurent-de-la-Salanque, Pyrénées-Orientales, S FRANCE, 2015 (Roccken)	100.4	803	1	7	Fruit	0.04	< 0.01	0.05	
Trial: S15-03095-06 Los Palacios, Andalucia, SPAIN, 2015 (Alanis)	95.5	955	1	7	Fruit	0.06	< 0.01	0.07	
Trial: S15-03095-09 Saint-Laurent-de-la-Salanque, Pyrénées-Orientales, S FRANCE, 2015 (Marketer)	103	928	1	7	Fruit	0.02	< 0.01	0.03	
Trial: NC08 Clinton, NC USA, 2009 (Cross country)	110.9 + 109.7 13 day interval	382 380	2	1	Fruit	0.0604,0.088 0.08	< 0.05	0.1104,0.138 0.13	Report: R-4460 Study: IR-4 PR No. 09032 5%EC
Trial: SC*05 Charleston, SC USA, 2009 (Calypso Hybrid)	112 + 113.1 13 day interval	443 428	2	1	Fruit	0.10,0.12 0.11	< 0.05	0.15,0.17 0.16	
Trial: SC*06 Charleston, SC USA, 2009 (Poinsett 76)	112 + 112 14 day interval	439 427	2	1	Fruit	0.19,0.15 0.17	< 0.05	0.24,0.20 0.22	
Trial: GA*02 Tifton, GA USA, 2009 (National Pickling)	112 + 112 13 day interval	412 410	2	1	Fruit	0.069,0.060 0.065	< 0.05	0.119,0.110 0.115	
Trial: GA*03 Tifton, GA USA, 2009 (Straight 8)	113.1 + 112 13 day interval	414 412	2	1	Fruit	0.053,0.056 0.55	< 0.05	0.103,0.106 0.60	
Trial: OH*01 Fremont, OH USA, 2009 (Dasher II)	110.9 + 107.5 16 day interval	495 506	2	1	Fruit	< 0.05	< 0.05	< 0.10	
Trial: OH*02 Fremont, OH USA, 2009 (Sassy)	112 + 110.9, 16 day interval	500 524	2	1	Fruit	< 0.05	< 0.05	< 0.10	
Trial: TX*21 Weslaco, TX USA, 2009 (Poinsett 76)	112 + 113.1 14 day interval	403 399	2	1	Fruit	0.08,0.07 0.08	< 0.05	0.13,0.12 0.13	
Trial: CA44 Holtville, CA USA, 2009 (Cobra)	112 + 112 13 day interval	484 490	2	1	Fruit	0.059,0.066 0.063	< 0.05	0.109,0.116 0.113	

Courgette

Table 203 Residues in courgette from supervised trials in EU, Japan and USA involving foliar applications of Fenpyroximate 5%SC or 5%EC

Courgette	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: S15-03095-01 Saint-Laurent-de-la-Salanque, Pyrénées-Orientales, S FRANCE, 2015 Courgette (Diamant)	101.9	918	1	0 1 3 5 7	Fruit Fruit Fruit Fruit Fruit	0.07 0.05 <u>0.01</u> < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.08 0.06 <u>0.02</u> < 0.02 < 0.02	Report: R-4511 Study: S15-03095 5%SC
Trial: S15-03095-02 Carmona, Andalucia, SPAIN, 2015 Courgette (Sabla)	98.4	984	1	0 1 3 5 7	Fruit Fruit Fruit Fruit Fruit	0.04 0.02 <u>< 0.01</u> < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.05 0.03 <u>< 0.02</u> < 0.02 < 0.02	
Trial: S15-03095-07 Conil de la Frontera, Cádiz, SPAIN, 2015 Courgette (Gelide)	105.3	1053	1	7	Fruit	< 0.01	< 0.01	< 0.02	
Trial: S15-03095-08 Castenaso, Bologna, ITALY, 2015 Courgette (Apollonia)	101.8	1018	1	7	Fruit	< 0.01	< 0.01	< 0.02	
Trial: S13-03365-01 Saint Laurent de la Salanque, Pyrenees-Orientales, S FRANCE, 2013 Courgette (Greyzini)	101.4	691.7	1	0 3 7 14 21	Fruit Fruit Fruit Fruit Fruit	0.07 <u>0.02</u> < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.08 <u>0.03</u> < 0.02 < 0.02 < 0.02	Report: R-4486 Study: S13-03365 5%SC
Trial: S13-03365-02 Budrio, Bologna ITALY, 2013 Courgette (Carisma)	102.1	595.6	1	0 3 7 14 21	Fruit Fruit Fruit Fruit Fruit	0.05 <u>0.01</u> < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.06 <u>0.02</u> < 0.02 < 0.02 < 0.02	
Trial: S13-03365-03 Valle Marine, Latina ITALY, 2013 Courgette (Greyzini)	103.1	1002	1	0 3 7 14 21	Fruit Fruit Fruit Fruit Fruit	0.13 <u>0.03</u> < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.14 <u>0.04</u> < 0.02 < 0.02 < 0.02	
Trial: S13-03365-04 Vazerac, Tarm et Garonne S FRANCE, 2013 Courgette (Bauera)	103.9	504.8	1	0 3 7 14 21	Fruit Fruit Fruit Fruit Fruit	0.07 <u>0.02</u> < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.08 <u>0.03</u> < 0.02 < 0.02 < 0.02	

Melons

Table 204 Residues in Melon from supervised trials in EU and Japan involving one foliar application of Fenpyroximate 5%SC

Melon	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: JPPA A JPPA Japan, 1988 (Andes) (Protected)	150	300	1	1 3 7	Flesh Flesh Flesh	< 0.005 < 0.005 < 0.005	- - -	< 0.005 < 0.005 < 0.005	Report: R-4306 Study: NN026-01 - Study not to GLP 5%SC
Trial: JPPA-Kochi A JPPA-Kochi Japan, 1988 (W. S. Earl's) (Protected)	175	350	1	1 3 7	Flesh Flesh Flesh	< 0.005 < 0.005 < 0.005	- - -	< 0.005 < 0.005 < 0.005	
Trial: JPPA A JPPA Japan, 1988 (Andes) (Protected)	150	300	1	1 3 7	Flesh Flesh Flesh	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	Report: R-4307 Study: NN026-02 - Study not to GLP 5%SC
Trial: Kochi A JPPA-Kochi Japan, 1988 (W. S. Earl's) (Protected)	175	350	1	1 3 7	Flesh Flesh Flesh	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	
Trial: JPPA A JPPA Japan, 1988 (Andes) (Protected)	150	300	1	1 3 7	Flesh Flesh Flesh	< 0.005 < 0.005 < 0.005	- - -	< 0.005 < 0.005 < 0.005	Report: R-4309 Study: NN026-03 Study not to GLP Peel was removed 5%SC
Trial: Kochi A JPPA-Kochi Japan, 1988 (W. S. Earl's) (Protected)	175	350	1	1 3 7	Flesh Flesh Flesh	< 0.005 < 0.005 < 0.005	- - -	< 0.005 < 0.005 < 0.005	
Trial: JPPA A JPPA Japan, 1988 (Andes) (Protected)	150	300	1	1 3 7	Flesh Flesh Flesh	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	Report: R-4310 Study: NN026-04 - Study not to GLP -Refer also to 5.7.1/03 Peel was removed 5%SC
Trial: Kochi A Kochi Japan, 1988 (W. S. Earl's) (Protected)	175	350	1	1 3 7	Flesh Flesh Flesh	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	
Trial: S13-03364-01 Fontanars, Valencia SPAIN,2013 (Amarillo de España)	107.6	523	1	0 0 0 3 3 3 7 7 7 14 14 14 21 21 21	Pulp Peel Whole Fruit Pulp Peel Whole Fruit Pulp Peel Whole Fruit Pulp Peel Whole Fruit Pulp Peel Whole Fruit	< 0.01 0.32 0.07 < 0.01 0.25 0.06 0.01 0.35 0.08 < 0.01 0.14 0.03 < 0.01 0.11 0.02	< 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01	< 0.02 0.33 0.08 < 0.02 0.26 0.07 0.02 0.36 0.09 < 0.02 0.15 0.04 < 0.02 0.12 0.03	Report: R-4487 Study: S13-03364 5%SC

Melon	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: S13-03364-02 Budrion, Province of Bologna ITALY, 2013 (Saphir)	108.4	843	1	0 0 0 3 3 3 7 7 7 14 14 14 21 21 21	Pulp Peel Whole Fruit Pulp Peel Whole Fruit Pulp Peel Whole Fruit Pulp Peel Whole Fruit Pulp Peel Whole Fruit	< 0.01 0.03 0.01 < 0.01 0.02 < 0.01 < 0.01 0.02 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 0.04 0.02 < 0.02 0.03 < 0.02 < 0.02 0.03 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 0.02 < 0.02	
Trial: S13-03364-03 Huelva, Andalusia Spain, 2013 (Quisose)	104.3	811	1	0 0 0 3 3 3 7 7 7 14 14 14 21 21 21	Pulp Peel Whole Fruit Pulp Peel Whole Fruit Pulp Peel Whole Fruit Pulp Peel Whole Fruit Pulp Peel Whole Fruit	< 0.01 0.06 0.02 < 0.01 0.06 0.01 < 0.01 0.05 0.01 < 0.01 0.08 < 0.01 < 0.01 0.04 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 0.07 0.03 < 0.02 0.07 0.02 < 0.02 0.06 0.02 < 0.02 0.09 < 0.02 < 0.02 0.05 < 0.02	
Trial: S13-03364-04 Laguanto, Ferrara Italy, 2013 (Sogna)	107.5	627	1	0 0 0 3 3 3 7 7 7 14 14 14 21 21 21	Pulp Peel Whole Fruit Pulp Peel Whole Fruit Pulp Peel Whole Fruit Pulp Peel Whole Fruit Pulp Peel Whole Fruit	< 0.01 0.19 0.05 < 0.01 0.06 0.02 < 0.01 0.04 0.01 < 0.01 0.02 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 0.20 0.06 < 0.02 0.07 0.03 < 0.02 0.05 0.02 < 0.02 0.03 < 0.02 < 0.02 < 0.02 < 0.02	

Cantaloupe

Table 205 Residues in Cantaloupe from supervised trials in USA involving 2 foliar applications of Fenpyroximate 5%EC

Cantaloupe	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: 09022.05-OH*06 Fremont, Ohio USA, 2005 (Aphrodite)	110.9 + 109.7 15 day interval	500 489	2	3	Fruit	< 0.05	-	< 0.05	Report: R-4195 Study: IR-4 PR No. 09022 5%EC
Trial: 09022.05-GA*06 Tifton, Georgia USA, 2005 (Hale's Best Jumbo)	113.1 + 113.1 12 day interval	303 302	2	2	Fruit	< 0.05	-	< 0.05	
Trial: 09022.05-TX*12 Weslaco, Texas USA, 2005 (Mission)	112 + 112 16 day interval	307 326	2	3	Fruit	< 0.05	-	< 0.05	
Trial: 09022.05-TX*13 Weslaco, Texas USA, 2005 (Cruiser)	112 + 112 16 day interval	347 317	2	4	Fruit	< 0.05	-	< 0.05	
Trial: : 09022.05-CA41 Riverside, California USA, 2005 (Laredo)	112 + 113.1 14 day interval	281 283	2	1 3 7 13 21	Fruit Fruit Fruit Fruit Fruit	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	-	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	
Trial: : 09022.05-NM08 Mesilla, New Mexico USA, 2005 (Topmark SR)	114.1 + 112 14 day interval	386 379	2	3	Fruit	< 0.05	-	< 0.05	
Trial: : 09022.05-CA42 Holtville, California USA, 2005 (Hymark)	112 + 112 12 day interval	295 296	2	2	Fruit	< 0.05	-	< 0.05	
Trial: : 09022.05-CA43 Parlier, California USA, 2005 (Topmark)	113.1 + 112 12 day interval	377 370	2	2	Fruit	< 0.05	-	< 0.05	

Watermelon

Table 206 Residues in watermelon from supervised trials in Japan and USA involving 1-2 foliar applications of Fenpyroximate 5%SC or 5%EC.

Watermelon	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: Niigata Niigata JAPAN, 1989 (Wase-Nissho) (Protected)	25	1000	1	1 3 7	Flesh Flesh Flesh	< 0.005 < 0.005 < 0.005	- - -	< 0.005 < 0.005 < 0.005	Report: R-4300 Study not to GLP Fruits (peel removed) 5%SC
Trial: Nara Nara JAPAN, 1989 (Asahikari SR) (Protected)	50	1000	1	1 3 7	Flesh Flesh Flesh	< 0.005 < 0.005 < 0.005	- - -	< 0.005 < 0.005 < 0.005	

Watermelon	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: Niigata Horticulture Experimental Station Niigata Horticulture Experimental Station JAPAN, 1989 (Wase-Nissho) (Protected)	25	1000	1	1 3 7	Flesh Flesh Flesh	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	Report: R-4301 Study not to GLP Fruits (peel removed) 5%SC
Trial: Nara Agricultural Experimental Station Nara Agricultural Experimental Station JAPAN, 1989 (Asahikari SR) (Protected)	50	1000	1	1 3 7	Flesh Flesh Flesh	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	
Trial: Niigata Niigata JAPAN, 1989 (Wase-Nissho) (Protected)	25	1000	1	1 3 7	Flesh Flesh Flesh	< 0.005 < 0.005 < 0.005	- - -	< 0.005 < 0.005 < 0.005	Report: R-4303 Study not to GLP Fruits (peel removed) 5%SC
Trial: Nara Nara JAPAN, 1989 (Asahikari SR) (Protected)	50	1000	1	1 3 7	Flesh Flesh Flesh	< 0.005 < 0.005 < 0.005	- - -	< 0.005 < 0.005 < 0.005	
Trial: Niigata Niigata JAPAN, 1990 (Wase-Nissho) (Protected)	25	1000	1	1 3 7	Flesh Flesh Flesh	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	Report: R-4304 - Study not to GLP Fruits (peel removed) 5%SC
Trial: Nara Nara JAPAN, 1989 (Asahikari SR) (Protected)	50	1000	1	1 3 7	Flesh Flesh Flesh	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	
Trial: JPPA, Kochi JPPA, Kochi JAPAN, 2011 (Yozora) (Protected)	140.5	2810	1	1 3 7 1 3 7	Flesh Flesh Flesh Whole fruit* Whole fruit* Whole fruit*	< 0.005 < 0.005 < 0.005 0.061 0.083 0.069	< 0.005 < 0.005 < 0.005 < 0.005 0.005 0.005	< 0.010 < 0.010 < 0.010 0.066 0.088 0.074	Report: R-4452 - Study not to GLP Refer also to 5.7.3/06 5%SC * calculated value
Trial: JPPA, Miyazaki JPPA, Miyazaki JAPAN, 2011 (Hitorijime HM) (Protected)	115.5	2310	1	1 3 7 1 3 7	Flesh Flesh Flesh Whole fruit* Whole fruit* Whole fruit*	< 0.005 < 0.005 < 0.005 0.030 0.053 0.069	< 0.005 < 0.005 < 0.005 < 0.005 0.006 0.005	< 0.010 < 0.010 < 0.010 0.035 0.059 0.074	
Trial JPPA, Kochi JPPA, Kochi JAPAN, 2011 (Yozora) (Protected)	140.5	2810	1	1 3 7	Peel Peel Peel	0.283 0.393 0.327	< 0.005 0.006 0.006	0.288 0.399 0.333	Report: R-4453- Study not to GLP - Refer also to 5.7.3/05 5%SC
Trial JPPA, Miyazaki JPPA, Miyazaki JAPAN, 2011 (Hitorijime HM) (Protected)	115.5	2310	1	1 3 7	Peel Peel Peel	0.128 0.243 0.126	< 0.005 0.009 0.005	0.133 0.252 0.131	

Watermelon	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Study: N10R060-1 Trial: JPPA, Kochi JPPA, Kochi JAPAN, 2011 (Yozora) (Protected)	140.5	2810	1	1 3 7 1 3 7	Flesh Flesh Flesh Whole fruit* Whole fruit* Whole fruit*	< 0.005 < 0.005 < 0.005 0.042 0.046 0.026	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005	< 0.010 < 0.010 < 0.010 0.047 0.051 0.031	Report: R-4454 - Study not to GLP 5%SC * calculated value
Study: N10R060-1 Trial: JPPA, Miyazaki JPPA, Miyazaki JAPAN, 2011 (Hitorijime HM) (Protected)	115.5	2310	1	1 3 7 1 3 7	Flesh Flesh Flesh Whole fruit* Whole fruit* Whole fruit*	< 0.005 < 0.005 < 0.005 0.017 0.022 0.014	< 0.005 < 0.005 < 0.005 < 0.005 < 0.005 < 0.005	< 0.010 < 0.010 < 0.010 0.022 0.027 0.019	
Trial: JPPA, Kochi JPPA, Kochi JAPAN, 2011 (Yozora) (Protected)	140.5	2810	1	1 3 7	Peel Peel Peel	0.188 0.209 0.112	< 0.005 0.005 < 0.005	0.193 0.214 0.117	Report: R-4455 Study: N10RO60-2 Study not to GLP 5%SC
Trial: JPPA, Miyazaki JPPA, Miyazaki JAPAN, 2011 (Hitorijime HM) (Protected)	115.5	2310	1	1 3 7	Peel Peel Peel	0.064 0.091 0.050	< 0.005 0.005 < 0.005	0.069 0.096 0.055	
Trial: 11182.13-CA121 Parlier, CA USA, 2013 (All Sweet)	112 + 113.1 14 day interval	374 393	2	1	Fruit	< 0.05	< 0.05	< 0.10	Report: IR-4 PR No. 11182 Study: IR-4 PR No. 11182 5%EC
Trial: 11182.13-GA*02 Tifton, GA USA, 2013 (Charleston Grey)	110.9 + 110.9 10 day interval	234 234	2	1	Fruit	< 0.05	< 0.05	< 0.10	
Trial: 11182.13-OH*15 Fremont, OH USA, 2013 (Sangria)	112 + 110.9 13 day interval	430 364	2	1	Fruit	< 0.05	< 0.05	< 0.10	
Trial: 11182.13-SC*438 Charleston, SC USA, 2014 (Micky Lee)	113.1 + 114.2 13 day interval	439 449	2	1	Fruit	< 0.05	< 0.05	< 0.10	

Fruiting vegetables, other than Cucurbits

Peppers

Table 207 Residues in Peppers from supervised trials in EU, Japan and the USA involving foliar applications of Fenpyroximate 5% SC or 5% EC

Peppers	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: AF/6096/NN/2 Montauban, Tarn-et-Garonne France, 2001 (Joselito)	105.4	829	1	7 10	Whole pepper Whole pepper	0.08 0.06	< 0.01 < 0.01	0.09 0.07	Report: R-4176 Study: AF/6096/NN 5%SC
Trial: AF/6096/NN/3 Larbarthe, Tarn-et-Garonne France, 2001 (Joselito)	99.6	972	1	7 10	Whole pepper Whole pepper	0.03 0.02	< 0.01 < 0.01	0.04 0.03	
Trial: AF/6096/NN/4 Zangadas, Thessaloniki Greece, 2001 (RS 912869)	105.5	1037.7	1	0 3 7 10	Whole pepper Whole pepper Whole pepper Whole pepper	0.14 0.15 0.12 0.10	< 0.01 < 0.01 < 0.01 < 0.01	0.15 0.16 0.13 0.11	
Trial Miyagi A Miyagi JAPAN, 1989 Bell Pepper (, Nishiki) (protected)	75	1500	1	1 3 7	Fruit Fruit Fruit	0.130 0.102 0.064	- - -	0.130 0.102 0.064	Report: R-4336 Study: NN027-01 -Study not to GLP Remove the Calyx 5%EC
Trial Nagano A Nagano JAPAN, 1989 Bell Pepper (, Kyonami) (protected)	100	2000	1	1 3 7	Fruit Fruit Fruit	0.092 0.055 0.048	- - -	0.092 0.055 0.048	
	50	2000	1	1 3 7	Fruit Fruit Fruit	0.051 0.036 0.028	- - -	0.051 0.036 0.028	
Trial Miyagi A Miyagi JAPAN, 1989 Bell Pepper (Nishiki) (protected)	75	1500	1	1 3 7	Fruit Fruit Fruit	- - -	0.006 0.005 0.005	0.006 0.005 0.005	Report: R-4337 Study: NN027-02 -Study not to GLP Remove the Calyx 5%EC
Trial Nagano A Nagano JAPAN, 1989 Bell Pepper (Kyonami) (protected)	100	2000	1	1 3 7	Fruit Fruit Fruit	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	
	50	2000	1	1 3 7	Fruit Fruit Fruit	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	
Trial: Miyagi A Miyagi JAPAN, 1989 Bell Pepper (Nishiki) (protected)	75	1500	1	1 3 7	Fruit Fruit Fruit	0.124 0.057 0.071	- - -	0.124 0.057 0.071	Report: R-4339 Study: NN027-03 -Study not to GLP Remove the Calyx 5%EC
Trial: Nagano A Nagano JAPAN, 1989 Bell Pepper (Kyonami) (protected)	100	2000	1	1 3 7	Fruit Fruit Fruit	0.080 0.068 0.038	- - -	0.080 0.068 0.038	
	50	2000	1	1 3 7	Fruit Fruit Fruit	0.044 0.040 0.030	- - -	0.044 0.040 0.030	

Peppers	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: Miyagi A Miyagi JAPAN, 1989 Bell Pepper (Nishiki) (protected)	75	1500	1	1 3 7	Fruit Fruit Fruit	- - -	0.007 < 0.005 0.007	0.007 < 0.005 0.007	Report: R-4340 Study: NN027-04 -Study not to GLP Remove the Calyx 5%EC
Trial: Nagano A Nagano JAPAN, 1989 Bell Pepper (Kyonami) (protected)	100	2000	1	1 3 7	Fruit Fruit Fruit	- - -	< 0.005 0.005 < 0.005	< 0.005 0.005 < 0.005	
	50	2000	1	1 3 7	Fruit Fruit Fruit	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	
Trial: Kochi A Kochi JAPAN, 1991 Bell Pepper (Tosahime) (protected)	75	300	1	1 3 7	Fruit Fruit Fruit	0.126 0.107 0.066	- - -	0.126 0.107 0.066	Report: R-4342 Study: NN027-05 -Study not to GLP Remove the Calyx 5%EC
Trial: Miyazaki A Miyazaki JAPAN, 1991 Bell Pepper (Tosahikari-D) (protected)	50	200	1	1 3 7	Fruit Fruit Fruit	0.100 0.083 0.069	- - -	0.100 0.083 0.069	
Trial: Kochi A Kochi JAPAN, 1991 Bell Pepper (Tosahime) (protected)	75	300	1	1 3 7	Fruit Fruit Fruit	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	Report: R-4343 Study: NN027-06 -Study not to GLP Remove the Calyx 5%EC
Trial: Miyazaki A Miyazaki JAPAN, 1991 Bell Pepper (Tosahikari-D) (protected)	50	200	1	1 3 7	Fruit Fruit Fruit	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	
Trial: Kochi A Kochi JAPAN, 1991 Bell Pepper (Tosahime) (protected)	75	300	1	1 3 7	Fruit Fruit Fruit	0.107 0.086 0.071	- - -	0.107 0.086 0.071	Report: R-4344 Study: NN027-07 -Study not to GLP Remove the Calyx 5%EC
Trial: MiyazakiA Miyazaki JAPAN, 1991 Bell Pepper (Tosahikari-D) (protected)	50	200	1	1 3 7	Fruit Fruit Fruit	0.084 0.078 0.059	- - -	0.084 0.078 0.059	
Trial: Kochi A Kochi JAPAN, 1991 Bell Pepper (Tosahime) (protected)	75	300	1	1 3 7	Fruit Fruit Fruit	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	Report: R-4345 Study: NN027-08 -Study not to GLP Remove the Calyx 5%EC
Trial: Miyazaki A Miyazaki JAPAN, 1991 Bell Pepper (Tosahikari-D) (protected)	50	200	1	1 3 7	Fruit Fruit Fruit	- - -	< 0.005 < 0.005 < 0.005	< 0.005 < 0.005 < 0.005	
Study: NN027-09 Trial: Iwate A Iwate	100	200	1	1 3 7	Fruit Fruit Fruit	0.170 0.138 0.094	- - -	0.170 0.138 0.094	Report: R-4346 -Study not to

Peppers	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
JAPAN, 1991 Bell Pepper (Tosa-green B)	50	200	1	1 3 7	Fruit Fruit Fruit	0.084 0.070 0.040	- - -	0.084 0.070 0.040	GLP Remove the Calyx 5%EC
Trial: NJ09 Bridgeton, NJ USA, 2005 Bell Pepper (King Authur)	117.6 + 113.1 14 day interval	715 740	2	1	Fruit	0.068,0.069 0.07	-	0.068,0.069 0.07	Report: R-4194 Study: IR-4 PR No. 08617 5%EC * rain within 1 hour, so application 2 repeated
Trial-WI04 Arlington,WI USA, 2005 Bell Pepper (Bellboy)	115.3 + 112 14 day interval	440 432	2	1	Fruit	< 0.05	-	< 0.05	
Trial –OH*05 Freemont, OH USA, 2005 Non Bell Pepper (Sahuaro)	112 + 112 14 day interval	462 459	2	1	Fruit	< 0.05	-	< 0.05	
Trial –TX09 Weslaco, TX USA, 2005 Non Bell Pepper (Tam Veracruz)	114.2 + 115.3 13 day interval	398 402	2	1	Fruit	< 0.05	-	< 0.05	
Trial –TX10 Weslaco, TX USA, 2005 Bell Pepper (Capistrano)	113.1 + 113.1 14 day interval	433 436	2	1	Fruit	< 0.05	-	< 0.05	
Trial FL14 Citra, FL USA, 2005 Non Bell Pepper (Mitla)	114.2 + 116.5 13 day interval	426 437	2	1	Fruit	0.11,0.12 0.12	-	0.11,0.12 0.12	
Trial FL15 Citra, FL USA, 2005 Bell Pepper (Capistrano)	110.9 + 115.3 14 day interval	413 434	2	1	Fruit	< 0.05	-	< 0.05	
Trial TX*11 Weslaco, TX USA, 2005 Bell Pepper (Capistrano)	112 + 115.3 16 day interval	393 396	2	1	Fruit	0.056,0.075 0.07	-	0.056,0.075 0.07	
Trial –NC04 Clinton, North Carolina USA, 2005 Bell Pepper (Heritage)	112 + 109.7* + 112 14 day interval	403 396	2	0 1 3 7 12	Fruit Fruit Fruit Fruit Fruit	0.093,0.0950.09 0.133,0.1200.13 0.098,0.1100.10 0.099,0.0960.10 0.094,0.0700.08	-	0.093,0.095,0.09 0.133,0.120,0.13 0.098,0.110,0.10 0.099,0.096,0.10 0.094,0.070,0.08	
Trial –NC05 Clinton, North Carolina USA, 2005 Non Bell Pepper (Aruba)	112 + 113.1 14 day interval	403 408	2	1	Fruit	0.057,0.050 0.05	-	0.057,0.050, 0.05	
Trial-TN05 Crossville, TN USA, 2005 Bell Pepper (California Wonder Sweet)	115.3 + 114.2 + 120.9 14 day interval	402 400 409	3	1	Fruit	< 0.05	-	< 0.05	
Trial-FL16 Citra, FL USA, 2005 Bell Pepper (Capistrano)	116.5 + 110.9 14 day interval	435 416	2	1	Fruit	0.074,0.067 0.07	-	0.074,0.067 0.07	

Peppers	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial-NM09 Mesilla, NM USA, 2005 Non Bell Pepper (Joe E. Parker)	112 + 112 14 day interval	467 467	2	1 3 7 14	Fruit Fruit Fruit Fruit	< 0.05 < 0.05 < 0.05 < 0.05	-	< 0.05 < 0.05 < 0.05 < 0.05	
Trial-CA39 Holtville, CA USA, 2005 Bell Pepper (Wizard)	114.2 + 110.9 13 day interval	547 536	2	1	Fruit	< 0.05	-	< 0.05	
Trial-CA40 Parlier, CA USA, 2005 Bell Pepper (Indria)	112 + 114.2 14 day interval	426 424	2	1	Fruit	< 0.05	-	< 0.05	
Trial-CO08 Ft. Collins, CO USA, 2005 (protected) Non Bell Pepper (DRH 7118 F1)	113.1 + 115.3 13 day interval	471 480	2	1	Fruit	0.056,0.052 0.054	-	0.056,0.052 0.054	

Tomato

Table 208 Residues in Tomatoes from supervised trials in Brazil, EU and Japan involving foliar applications of Fenpyroximate 5% SC or 5% EC

Tomatoes	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Study A48130 Cheste Spain, 1990 (Alex) (protected)	103	2000	1	0 3 7 14	Fruit Fruit Fruit Fruit	0.04 0.03 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.05 0.04 0.04 0.03	Report: R-4038 -Study not to GLP 5%SC
Study A48131 Cheste Spain, 1990 (Alex) (protected)	154.5	2000	1	0 3 7 14	Fruit Fruit Fruit Fruit	0.04 0.04 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.05 0.05 0.03 0.03	Report: R-4039 -Study not to GLP 5%SC
Trial: AF/6094/NN/1 Akrolimni, Pella, Greece, 2001 (Titano)	102.9	1004	1	0 3 7 10	Fruit Fruit Fruit Fruit	0.01 0.02 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.02 0.03 0.03 0.03	Report: R-4178 Study: AF/6094/NN 5%SC
Trial: AF/6094/NN/2 Anachoma, Thessaloniki Greece, 2001 (Volcano)	100.5	980	1	7 10	Fruit Fruit	0.02 0.02	< 0.01 < 0.01	0.03 0.03	
Trial: AF/6094/NN/3 Camino De la Reina, Seville Spain, 2001 (Malpica)	103.1	1006	1	0 3 7 10	Fruit Fruit Fruit Fruit	0.08 0.04 0.04 0.04	< 0.01 < 0.01 < 0.01 < 0.01	0.09 0.05 0.05 0.05	

Tomatoes	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: AF/6094/NN/4 La Puebla del Rio, Seville Spain, 2001 (Avalon)	107.4	1048	1	7 10	Fruit Fruit	0.05 0.05	< 0.01 < 0.01	0.06 0.06	
Trial: AF/6095/NN/1 Charlton, Shropshire UK, 2001 (Solution)(protected)	104.1	1016	1	0 3 7 10	Fruit Fruit Fruit Fruit	0.03 0.04 0.02 0.03	< 0.01 < 0.01 < 0.01 < 0.01	0.04 0.05 0.03 0.04	Report: R-4179 Study: AF/6095/NN 5%SC
Trial: AF/6095/NN/2 Spondon, Derbyshire UK, 2001 (Cussack)(protected)	102.3	998	1	7 10	Fruit Fruit	0.10 0.08	< 0.01 < 0.01	0.11 0.09	
Trial: AF/6095/NN/3 Montauban, Tarn-et-Garonne France, 2001 (Petula) (protected)	101.8	993	1	0 3 7 10	Fruit Fruit Fruit Fruit	0.03 0.04 0.06 0.03	< 0.01 < 0.01 < 0.01 < 0.01	0.04 0.05 0.07 0.04	
Trial: AF/6095/NN/4 Orgueil, Tarn-et-Gronne, France, 2001 (Cecilia) (protected)	100.6	981	1	7 10	Fruit Fruit	0.02 0.04	< 0.01 < 0.01	0.03 0.05	
Trial: AF/6095/NN/5 Argelato, Emilia Romagna Italy, 2001 (Incas) (protected)	105.3	1027	1	7 10	Fruit Fruit	0.08 0.09	< 0.01 < 0.01	0.09 0.10	
Trial: AF/6781/NN/1 Charlton, Shropshire UK, 2002 (Solution)(protected)	97.4	950	1	0 3 7 10	Fruit Fruit Fruit Fruit	0.06 0.04 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01	0.07 0.05 0.04 0.04	Report: R-4180, Study: AF/6781/NN 5%SC
Trial: AF/6781/NN/2 Valle Niza, Malaga Spain, 2002 (Josefina)(protected)	104.9	1023	1	0 3 7 10	Fruit Fruit Fruit Fruit	< 0.01 0.09 0.06 0.07*	< 0.01 < 0.01 < 0.01 < 0.01*	< 0.02 0.10 0.07 0.08*	
Trial: AF/6781/NN/3 Spondon, Derbyshire UK, 2002 (Shirley) (protected)	94.9	926	1	7 10	Fruit Fruit	0.06 0.07	< 0.01 < 0.01	0.07 0.08	
Trial: AF/6781/NN/4 Valle Niza, Malaga, Spain, 2002 (Josefina)(protected)	111.7	1090	1	7 10	Fruit Fruit	0.08 0.11	< 0.01 < 0.01	0.09 0.12	
Trial: AF/6782/NN/1 Sevilla Spain, 2002 (Mina)	102.5	1000	1	0 3 7 10 14	Fruit Fruit Fruit Fruit Fruit	0.01 0.02 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.03 < 0.02 < 0.02 0.02	Report: R-4181 Study: AF/6782/NN 5%SC
Trial: AF/6782/NN/3 Funes, Zaragoza Spain, 2002 (H-9036 DG)	102.5	995	1	0 3 7 10 14	Fruit Fruit Fruit Fruit Fruit	0.07 0.04 0.05 0.03 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.08 0.05 0.06 0.04 0.05	

Tomatoes	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: AF/6782/NN/4 Barboles, Zaragoza Spain, 2002 (H-9036)	102.5	997	1	7 10	Fruit Fruit	0.04 0.03	< 0.01 < 0.01	0.05 0.04	
Trial: AF/6782/NN/5 Lavilledieu du Temple, Tarn-et-Garonnne France, 2002 (Rio Grande)	102.5	1003	1	7 10	Fruit Fruit	0.02 0.01	< 0.01 < 0.01	0.03 0.02	
Trial: .Ushiku A Ushiku Japan, 1995 (Houryu) (protected)	60	300	1	1 3 7	Fruit Fruit Fruit	0.104 0.061 0.064	< 0.005 < 0.005 < 0.005	0.109 0.066 0.069	Report: R-4400 Study: NN031-01 Calyxes removed 5%EC
	120	300	1	1 3 7	Fruit Fruit Fruit	0.120 0.112 0.062	0.006 0.006 0.005	0.126 0.118 0.067	
Trial: . Miyazaki A Miyazaki Japan, 1995 (Momotaro) (protected)	60	300	1	1 3 7	Fruit Fruit Fruit	0.097 0.053 0.062	0.006 0.005 0.006	0.103 0.058 0.068	
	120	300	1	1 3 7	Fruit Fruit Fruit	0.095 0.087 0.083	0.007 0.008 0.012	0.102 0.095 0.094	
Trial: Ibaraki Ibaraki Japan, 1995 (Houryu) (protected)	120	300	1	1 3 7	Fruit Fruit Fruit	0.122 0.127 0.106	< 0.005 0.008 0.008	0.127 0.135 0.114	Report: R-4401 Study: NN031-02 Calyxes removed 5%EC
	60	300	1	1 3 7	Fruit Fruit Fruit	0.111 0.076 0.080	0.006 < 0.005 < 0.005	0.117 0.081 0.085	
Trial: . Miyazaki Miyazaki Japan, 1995 (Momotaro) (protected)	120	300	1	1 3 7	Fruit Fruit Fruit	0.107 0.112 0.110	0.010 0.012 0.009	0.117 0.124 0.119	
	60	300	1	1 3 7	Fruit Fruit Fruit	0.096 0.058 0.062	0.010 0.008 0.006	0.106 0.066 0.068	
Study A48132 Santo Antonio de posse Brazil, 1990 (Santa Clara)	108.2	1400 1400	2	7 14	Fruit	0.02 0.01	< 0.01 < 0.01	0.03 0.02	Report: R-4040 -Study not to GLP
Study A48133 Santo Antonio de posse Brazil, 1990 (Santa Clara)	216.3	1400 1400	2	7 14	Fruit	0.03 0.02	< 0.01 < 0.01	0.04 0.03	Report: R-4041 -Study not to GLP 5%SC

Table 209 Residues in Tomatoes from supervised trials in USA involving foliar applications of Fenpyroximate 5%EC

Tomatoes	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Study: IR-4 No. 09027 Trial: .NY03 Freeville, NY USA, 2005 (Mariana)	102 + 118 12 day interval	Treatment 02: 192, 202 Treatment 03: 189, 199	2	1	Fruit	0.04,0.04 <u>< 0.05</u>	0.03,0.03 < 0.05	0.07,0.07 <u>< 0.10</u>	Report: R-4196 (Processing study) 5%EC
Study: IR-4 No. 09027 Trial: .OH*04 Fremont, OH USA, 2005 (Cupid)	113.1, 114.2, 112 12 and 23 day interval	455 478 468	3	1	Fruit	<u>< 0.05</u>	< 0.05	<u>< 0.10</u>	Report: R-4196 (Small fruited trial) 5%EC
Trial: .FL11 Citra, FL USA, 2005 (Solarsett)	113.1, 116.5 13 day interval	285 295	2	1 3 7 14 21	Fruit Fruit Fruit Fruit Fruit Fruit	0.07,0.07,<u>0.07</u> < 0.05 < 0.05 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	<u>0.12</u> < 0.10 < 0.10 0.10 < 0.10	Report: R-4196, Study: IR-4 No. 09027 5%EC
Trial: .GA*05 Tifton, GA USA, 2005 (Amelia)	113.1, 112 12 day interval	455 450	2	1	Fruit	0.11,0.06 <u>0.09</u>	0.02,0.02,< 0.05	0.13,0.08 <u>0.14</u>	
Trial: FL13 Citra, FL USA, 2005 (FL47) (protected)	109.7, 108.6 14 day interval	278 275	2	1	Fruit	0.14,0.07 <u>0.11</u>	< 0.05	0.19,0.12 <u>0.16</u>	
Trial: .FL12 Citra, FL USA, 2005 (FL47)	115.3, 113.1 14 day interval	293 286	2	1	Fruit	0.09,0.08 <u>0.09</u>	< 0.05	0.11,0.10 <u>0.14</u>	
Trial: .TX08 Weslaco, TX USA, 2006 (Mariachi RZ) (protected)	113.1, 113.1 13 day interval	434 433	2	1	Fruit	0.08,0.07 <u>0.08</u>	< 0.05	0.13,0.12 <u>0.13</u>	
Trial: .CO07 Fort Collins, CO USA, 2005 (Trust F1) (protected)	118.7, 114.2 13 day interval	295 285	2	1	Fruit	0.08,0.07 <u>0.08</u>	< 0.05	0.13,0.12 <u>0.13</u>	
Trial: .NM06 Las Cruces, NM USA, 2005 (Celebrity VFN)	112, 109.7 14 day interval	234 231	2	1 3 7 13	Fruit Fruit Fruit Fruit	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	<u>< 0.10</u> < 0.10 < 0.10 < 0.10	
Trial: .CA30 Medera, CA USA, 2005 (Ace 55 VF)	112, 112 14 day interval	281 282	2	1	Fruit	<u>< 0.05</u>	< 0.05	<u>< 0.10</u>	
Trial: .NM07 Las Cruces, NM USA, 2005 (Cal-Ace)	138.9, 138.9 12 day interval	639 639	2	1	Fruit	0.06,0.05 <u>0.09</u>	< 0.05	0.11, 0.10, <u>0.14</u>	Report: R-4196, Study: IR-

Tomatoes	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: .CA31 Irvine, CA USA, 2005 (Bobcat)	113.1, 113.1 15 day interval	378 377	2	1	Fruit	0.05	< 0.05	0.10	4 No. 09027 5%EC
Trial: .CA32 Irvine, CA USA, 2005 (Bobcat)	112, 114.2 15 day interval	376 383	2	1	Fruit	< 0.05	< 0.05	< 0.10	
Trial: .CA33 Holtville, CA USA, 2005 (#9997)	113.1, 112 12 day interval	258 261	2	1	Fruit	0.08,0.08 0.08	< 0.05	0.08	
Trial: .CA34 Holtville, CA USA, 2005 (#9997)	113. 114.2 13 day interval	252 259	2	1	Fruit	< 0.05	< 0.05	< 0.05	
Trial: .CA35 Davis, CA USA, 2005 (Shady Lady)	112, 115.3 13 day interval	279 289	2	1	Fruit	< 0.05	< 0.05	< 0.05	
Trial: .CA36 Davis, CA USA, 2005 (AB-2)	112, 112 14 day interval	Treatment 02: 280, 280 Treatment 03: 279, 283	2	1 1 1	Fruit	< 0.05	< 0.05	< 0.05	
Trial: .CA37 Parlier, CA USA, 2005 (Cherry Grande)	110.9, 112 14 day interval	372 387	2	1	Fruit	0.12,0.11 0.12	0.06,0.05 0.05	0.158,0.16 0.17	
Trial: .CA38 Parlier, CA USA, 2005 (Quality 21)	114.2, 115.3 14 day interval	241 250	2	1	Fruit	< 0.05	< 0.05	< 0.05	

Beans

Table 210 Residues in Beans from supervised trials in the EU and the USA involving foliar applications of Fenpyroximate 5% SC or 5% EC

Beans	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Study: A48191 Cheste SPAIN, 1990 (Kidney, Garrafal Oro) (protected)	321.9	2500	1	0 3 7 14	Beans Beans Beans Beans	0.55 0.30 0.14 0.04	0.02 0.01 0.01 < 0.01	0.57 0.31 0.15 0.04	R-4068, -Study not to GLP 5%SC
Trial:-AF/6098/NN/1 Sanlucar de Barrameda, Seville SPAIN, 2001 (Brasilena) (protected)	102.7	1002	1	0 3 7 10	Whole pods Whole pods Whole pods Whole pods	0.30 0.09 0.08 0.05	< 0.01 < 0.01 < 0.01 < 0.01	0.31 0.10 0.09 0.06	Report: R-4158 Study: AF/6098/N

Beans	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial:-AF/6098/NN/2 Los Palacios Villafranca, Seville SPAIN, 2001 (Helda) (protected)	108.4	1058	1	7 10	Whole pods Whole pods	0.10 0.06	< 0.01 < 0.01	0.11 0.07	N 5%SC
Trial:-AF/6098/NN/3 Coria del Rio, Seville SPAIN, 2001 (Festival RZ) (protected)	103.9	1014	1	7 10	Whole pods Whole pods	0.02 0.03	< 0.01 < 0.01	0.03 0.04	
Trial:-AF/6098/NN/4 Akrolimni, Pella Greece, 2001 (Trebona) (protected)	97.7	954	1	0 3 7 10	Whole pods Whole pods Whole pods Whole pods	0.18 0.13 0.05 0.06	< 0.01 < 0.01 < 0.01 < 0.01	0.19 0.14 0.06 0.07	
Trial: AF/6099/NN/1 Montauban, Tarn-et-Garonne, France,2001 Bean, Green (Anger)	106.3	1037	1	0 3 7 10	Whole pods Whole pods Whole pods Whole pods	0.23 0.25 0.19 0.23	< 0.01 < 0.01 < 0.01 < 0.01	0.24 0.26 0.20 0.24	Report: R-4159 Study: AF/6099/NN 5%SC
Trial: AF/6099/NN/2 St Jory, Haute-Garonne Frence, 2001 Bean, Green (Bouster)	102.3	1000	1	7 10	Whole pods Whole pods	0.08 0.06	< 0.01 < 0.01	0.09 0.07	
Trial: AF/6099/NN/3 Palefyto, Pella Greece, 2001 Bean, Green (-)	107.1	1045	1	7 10	Whole pods Whole pods	0.06 0.07	< 0.01 < 0.01	0.07 0.08	
Trial: AF/6099/NN/4 Profitis, Thessaloniki Greece, 2001 Bean, Green (Zargana)	101.9	994	1	0 3 7 10	Whole pods Whole pods Whole pods Whole pods	0.24 0.20 0.14 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.25 0.21 0.15 < 0.02	
Trial AF/6778/NN/01 Sanlucar de Barrameda, Cadiz Spain, 2002 (Encano Dulce) (protected)	101.9	994	1	0 3 7 10	Whole pods Whole pods Whole pods Whole pods	0.19 0.08 0.03 0.02	< 0.01 < 0.01 < 0.01 < 0.01	0.20 0.09 0.04 0.03	Report: R-4160 Study AF/6778/NN 5%SC
Trial AF/6778/NN/02 Santa Engracia, Spain, 2002 (Oriente) (protected)	101.2	987	1	0 3 7 10	Whole pods Whole pods Whole pods Whole pods	0.18 0.11 0.03 0.03	< 0.01 < 0.01 < 0.01 < 0.01	0.19 0.12 0.04 0.04	
Trial AF/6778/NN/03 Sanlucar de Barrameda, Cadiz Spain, 2002 (Brisilena) (protected)	100.5	980	1	7 10	Whole pods Whole pods	0.03 0.01	< 0.01 < 0.01	0.04 0.02	
Trial AF/6778/NN/04 Los Palacios y Villafrance, Sevilla Spain, 2002 (Helba) (protected)	98.2	958	1	7 10	Whole pods Whole pods	0.03 0.02	< 0.01 < 0.01	0.04 0.03	
Trial: AF/6769/NN/1 Malause, Meauzac France, 2002 Bean, Green (Booster)	103.63	1011	1	0 3 7 10	Whole pods Whole pods Whole pods Whole pods	0.43 0.37 0.40 0.41	< 0.01 < 0.01 0.01 0.01	0.44 0.38 0.41 0.42	Report: R-4164 Study:

Beans	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: AF/6769/NN/2 Milagro, Zaragoza Spain, 2002 Bean, Green (Antea)	103.53	1010	1	0 3 7 10	Whole pods Whole pods Whole pods Whole pods	0.08 0.07 0.05 0.06	< 0.01 < 0.01 < 0.01 < 0.01	0.09 0.08 0.06 0.07	AF/6769/NN 5%SC
Trial: AF/6769/NN/3 St Porquier, Meauzac France, 2002 Bean, Green (Arcalia)	104.14	1016	1	7 10	Whole pods Whole pods	0.06 0.04	< 0.01 < 0.01	0.07 0.05	
Trial: AF/6769/NN/4 Funes, Zaragoza, Spain, 2002 Bean,Green Moncayo)	102.60	1001	1	7 10	Whole pods Whole pods	0.13 0.06	< 0.01 < 0.01	0.14 0.07	
Trial CA38 Davis, CA USA, 2008 Snap Bean (Blue Lake Bush 274)	110 + 109 14 day interval	299 290	2	1 3 8 10	Whole pods Whole pods Whole pods Whole pods	0.19,0.17,0.18 0.12,0.08,0.10 0.06,0.07,0.07 0.08,0.06,0.07	< 0.05 < 0.05 < 0.05 < 0.05	0.24,0.22, 0.23 0.17,0.23, 0.15 0.11,0.12, 0.12 0.13,0.11, 0.12	Report: R-4458 Study IR-4 PR No. 09942 5%EC
5.9.1/06 Study: IR-4 PR No. 09942 Trial FL37 Citra, FL USA, 2008 Snap Bean (Dusky)	110 + 109 5 day interval	374 364	2	1	Whole pods	0.07,0.1, 0.09	< 0.05	0.12,0.17 0.14	
Trial GA*13 Tifton, GA USA, 2008 Snap Bean (Blue Lake Bush 274)	112 + 113 13 day interval	449 449	2	1 1	Whole pods	0.13,0.16 0.15	0.04,0.03 < 0.05	0.17,0.19 0.20	
Trial NY24 Freeville, NY USA, 2008 Snap Bean (Hystyle)	111 + 111 13 day interval	318 318	2	1	Whole pods	0.08,0.10 0.09	< 0.05	0.13,0.15 0.14	
Frrmont, OH09 USA, 2008 Snap Bean (Sea Biscuit)	110 + 108 14 day interval	374 364	2	1	Whole pods	< 0.05	< 0.05	< 0.10	
Trial OH*10 Fremont, OH USA, 2011 Snap Bean (Brio)	106 + 111 14 day interval	364 374	2	1	Whole pods	< 0.05	< 0.05	< 0.10	
Trial WA*07 Moxee, WA USA, 2011 Snap Bean (Jade)	114 + 111 14 day interval	374 364	2	1	Whole pods	0.19,0.18 0.19	< 0.05	0.24,0.23 0.24	
Trial WI17 Arlington, WI USA, 2011 Snap Bean (Hystyle)	113, 111 13 day interval	393 383	2	1	Whole pods	0.10,0.07 0.09	< 0.05	0.15,0.12, 0.,14	

Potato

Table 211 Residues in Potato from supervised trials in USA involving 2 foliar applications of Fenpyroximate 5%EC

Potato	Application			DALA	Commodity	Residues (mg/kg)			Reference &
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	Comments
Trial 09-NY10	111 + 112	321	2	6	Tubers	< 0.05	< 0.05	< 0.10	Unpublished
Freeville, NY		333							
USA, 2009									Study IR-4 PR No. 10173
(Yukon Gold)									
Trial 09-NY11	115 + 114	376	2	7	Tubers	< 0.05	< 0.05	< 0.10	5%EC
North Rose, NY		375							
USA, 2009									
(Superior)									
Trial 09-FL07	111 + 111	375	2	7	Tubers	< 0.05	< 0.05	< 0.10	
Citra, FL		375							
USA, 2009									
(Red Pontiac)									
Trial 09-OH*09	125 + 115	353	2	6	Tubers	< 0.05	< 0.05	< 0.10	
Wooster, OH		356							
USA, 2009									
(Norland Red)									
Trial 09-WI12	112 + 114	394	2	6	Tubers	< 0.05	< 0.05	< 0.10	
Arlington, WI		399							
USA, 2009									
(Superior)									
Trial 09-WI16	113 + 114	200	2	7	Tubers	< 0.05	< 0.05	< 0.10	
Arlington, WI		212							
USA, 2009									
(Superior)									
Trial 09-NM17	115 + 110	288	2	7	Tubers	< 0.05	< 0.05	< 0.10	
Las Cruces, NM		325							
USA, 2009									
(Red Pontiac)									
Trial 09-ID12	112 + 114		2	7	Tubers	< 0.05	< 0.05	< 0.10	
Kimberley, ID									
USA, 2009									
(Russet Burbank)	565 + 567	422	2	7	Flakes	< 0.05	< 0.05	< 0.10	
		424			Chips	< 0.05	< 0.05	< 0.10	
					Wet Peels	< 0.05	< 0.05	< 0.10	
Trial 09-ID13	113 + 113	468	2	7	Tubers	< 0.05	< 0.05	< 0.10	
Kimberley, ID		470							
USA, 2009									
(Russet Burbank)									
Trial 09-WA05	113 + 113	341	2	7	Tubers	< 0.05	< 0.05	< 0.10	
Prosser, WA		328							
USA, 2009									
(Yukon Gold)									
Trial 09-	116 +	274	2	7	Tubers	< 0.05	< 0.05	< 0.10	

Potato	Application			DALA	Commodity	Residues (mg/kg)			Reference &
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	Comments
WA06	114								
Prosser, WA		256							
USA, 2009									
(Russet Burbank)									
Trial 09-WA*07	118 + 117	388	2	7	Tubers	< 0.05	< 0.05	< 0.10	
Moxee, WA		384							
USA, 2009									
(Red Norland)									
Trial 09-WA*08	113 + 112	377	2	7	Tubers	< 0.05	< 0.05	< 0.10	
Moxee, WA		370							
USA, 2009									
(Russet (Dillon))									
Trial 09-CA97	111 + 111	294	2	7	Tubers	< 0.05	< 0.05	< 0.10	
Holtville, CA		293							
USA, 2009									
(Cal White)									
Trial 09-NC18	110 + 112	292	2	0	Tubers	< 0.05	< 0.05	< 0.10	
Clinton, NC		298		3	Tubers	< 0.05	< 0.05	< 0.10	
USA, 2009				6	Tubers	< 0.05	< 0.05	< 0.10	
(Atlantic)				9	Tubers	< 0.05	< 0.05	< 0.10	
				12	Tubers	< 0.05	< 0.05	< 0.10	
Trial 09-MI11	112 + 111	281	2	0	Tubers	< 0.05	< 0.05	< 0.10	
Laingsburg, MI		279		3	Tubers	< 0.05	< 0.05	< 0.10	
USA, 2009				7	Tubers	< 0.05	< 0.05	< 0.10	
(FL 1879)				11	Tubers	< 0.05	< 0.05	< 0.10	
				14	Tubers	< 0.05	< 0.05	< 0.10	

Maize

Table 212 Residues in maize from supervised trials in EU and USA involving 1-2 foliar applications of Fenpyroximate 5 %SC or 5%EC

Maize	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: S11-02946-01	51.4	305	1	0	Cobs	< 0.01	< 0.01	< 0.02	Report: R-4470
Bolho, Somogy				7	Cobs	< 0.01	< 0.01	< 0.02	
HUNGARY, 2011				16	Cobs	< 0.01	< 0.01	< 0.02	Study: S11-02946
(Royalty)				21	Cobs	< 0.01	< 0.01	< 0.02	
				28	Cobs	< 0.01	< 0.01	< 0.02	5%SC
Study: S12-02436	49.5	386	1	0	Cobs	< 0.01	< 0.01	< 0.02	Report: R-4480
Mezohek, Josz-Naggkun-Szolnok				7	Cobs	< 0.01	< 0.01	< 0.02	
				14	Cobs	< 0.01	< 0.01	< 0.02	5%SC
HUNGARY, 2011				21	Cobs	< 0.01	< 0.01	< 0.02	
(Royalty)				28	Cobs	< 0.01	< 0.01	< 0.02	

Maize	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: 01 Stafford, KS USA, 2010 (P0541HR)	109 + 108 14 day interval	94 94	2	13	Grain	< 0.003	< 0.003	< 0.003	Report: R-4447 Study: TCI-10-270 5%EC
Trial: 02 Hinton, OK USA, 2010 Field corn(Syngenta NK-N72K)	112 + 115 14 day interval	94 94	2	7 14 21 28	Grain Grain Grain Grain	< 0.003 < 0.003 < 0.003 < 0.003	< 0.003 < 0.003 < 0.003 < 0.003	< 0.003 < 0.006 < 0.006 < 0.003	
Trial: 03 Hinton, OK USA, 2010 Field corn(Pioneer 33B54)	110 + 115 15 day interval	94 94	2	13	Grain	< 0.003	< 0.003	< 0.003	
Trial: 04 Uvalde, TX USA, 2010 (D16 6721)	112 + 111 14 day interval	94 94	2	14	Grain	< 0.01	< 0.01	< 0.02	
Trial: 05 Raymondville, TX USA, 2010 Field corn(H6284162)	115 + 114 13 day interval	94 94	2	14	Grain	< 0.003	< 0.003	< 0.003	
Trial: 06 Dill City, OK USA, 2010 (SyngentaNK-N72K)	114 + 114 14 day interval	94 94	2	13	Grain	< 0.003	< 0.003	< 0.003	
Trial: 07 Levelland, TX USA, 2010 (Agventure R350VBW)	111 + 110 14 day interval	94 94	2	14	Grain	< 0.003	< 0.003	< 0.006	
Trial: 08 Larned, KS USA, 2010 Field corn(33Y75)	111 + 112 14 day interval	94 94	2	14	Grain	< 0.003	< 0.003	< 0.006	
Trial: 09 Belpre, KS USA, 2010 (Pioneer 33D49)	109 + 111 14 day interval	94 94	2	14	Grain	< 0.003		< 0.003	
Trial: 10 Pierce, CO USA, 2010 (LG 2407)	109 + 109 14 day interval	94 94	2	14	Grain	< 0.003	< 0.003	< 0.003	

Table 213 Residues in Maize from supervised trials in EU involving one foliar application of Fenpyroximate 5%SC.

Maize	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: S11-02947-01 HUNGARY EU, 2011 (DKC 4490)	56.4	329	1	0 7 14 21 28	Grain Grain Grain Grain Grain	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Report: R-4471 Study: S11-02947

Maize	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: S11-02947-02 HUNGARY EU, 2011 (DK 4590)	54.4	318	1	0	Grain	< 0.01	< 0.01	< 0.02	5%SC
				7	Grain	< 0.01	< 0.01	< 0.02	
				14	Grain	< 0.01	< 0.01	< 0.02	
				21	Grain	< 0.01	< 0.01	< 0.02	
				28	Grain	< 0.01	< 0.01	< 0.02	

Tree nuts

Almonds

Table 214 Residues in almond from supervised trials in USA involving one foliar application of Fenpyroximate 5%EC.

Almond	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: CA1 Porterville, CA USA, 2001 (Mission)	451	944	1	14	Nut meat	< 0.05	< 0.05	< 0.10	Report: R-4155 Study: AA010709 5%EC
Trial: CA2 Terra Bella, CA USA, 2001 (Carmel)	448	935	1	14	Nut meat	< 0.05	< 0.05	< 0.10	
Trial: CA3 Hanford, CA USA, 2001 (Carmel)	449	925	1	14	Nut meat	< 0.05	< 0.05	< 0.10	
Trial: CA4 Hanford, CA USA, 2001 (Mission)	450	925	1	14	Nut meat	< 0.05	< 0.05	< 0.10	
Trial: CA5 Hanford, CA USA, 2001 (Prices)	447	935	1	0	Nut meat	< 0.05	< 0.05	< 0.10	
				7	Nut meat	< 0.05	< 0.05	< 0.10	
				14	Nut meat	< 0.05	< 0.05	< 0.10	
				21	Nut meat	< 0.05	< 0.05	< 0.10	
				28	Nut meat	< 0.05	< 0.05	< 0.10	

Walnut

Table 215 Residues in walnut from supervised trials in USA involving one foliar application of Fenpyroximate 5%EC

Walnut	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: CA6 Farmersville, CA USA, 2001 (Serr)	453	925	1	14	Nut meat	< 0.05	< 0.05	< 0.10	Report: R-4155 Study: AA010709 5%EC
Trial: CA7 Poplar, CA USA, 2001 (Tulare)	445	944	1	14	Nut meat	< 0.05	< 0.05	< 0.10	
Trial: CA8 Porterville, CA USA, 2001 (Chandler)	448	953	1	14	Nut meat	< 0.05	< 0.05	< 0.10	

Pecan

Table 216 Residues in Pecan from supervised trials in USA involving one foliar application of Fenpyroximate 5% EC

Pecan	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: FL1 Monticello, FL USA, 2001 (Stuart)	449	916	1	14	Nut meat	< 0.05	< 0.05	< 0.10	Report: R-4155 Study: AA010709 5%EC
Trial: GA1 Chula, GA USA, 2001 (Sumner)	449	935	1	14	Nut meat	< 0.05	< 0.05	< 0.10	
Trial: GA2 Nashville, GA USA, 2001 (Stuart)	451	953	1	14	Nut meat	< 0.05	< 0.05	< 0.10	
Trial: LA1 Shreveport, LA USA, 2001 (Melrose)	460	972	1	14	Nut meat	< 0.05	< 0.05	< 0.10	
Trial: TX1 Uvalde, TX USA, 2001 (Stuart)	449	916	1	14	Nut meat	< 0.05	< 0.05	< 0.10	

Coffee

Table 217 Residues in Coffee Bean from supervised trials in Brazil involving two foliar applications of Fenpyroximate (5%SC)

Coffee Bean	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: 018.034.184.08 Londrina/PR Brazil, 2008 (Red Catuai)	100 + 100 30 day interval	2500 2500	2	15	Beans	< 0.025	-	< 0.025	Report: R-4499 5%SC
Trial:0018.034.190.08 Espirito Santo do Pinhal/SP. Brazil, 2008 (New world)	100 + 100 30 day interval		2	15	Beans	< 0.025	-	< 0.025	Report: R-4500 5%SC
Trial:0018.034.240.08 Uberlândia / MG Brazil, xxxx (Red Catuai)	100 + 100 30 day interval		2	7 15 25 35	Beans Beans Beans Beans	< 0.025 < 0.025 ND ND	- - - -	ND < 0.025 ND ND	Report: R-4501 5%SC
Trial:0018.034.251.08 Pereiras/SP Brazil, 2008 (Red Catuai)	108 + 108 30 day interval		2	7 15 25 35	Beans Beans Beans Beans	< 0.025 < 0.025 < 0.025 < 0.025	- - - -	< 0.025 < 0.025 < 0.025 < 0.025	Report: R-4502 5%SC
Trial: RA 121 001 13B Santa Mariana/PR Brazil, 2014 (Catuara amarela)	100 + 100 30 day interval		2	5 15 25 35	Beans Beans Beans Beans	0.05 0.02 < 0.01 0.03	- - - -	0.05 0.02 < 0.01 0.03	Report: R-4490 Study: RA 121 001 13B
Trial: Bandeirantes/PR Brazil, 2014 (Catuai Vermelho)	100 + 100 30 day interval		2	5 15 25 35	Beans Beans Beans Beans	0.05 0.03 0.04 0.02	- - - -	0.05 0.03 0.04 0.02	5%SC
Trial: Cornélio Procópio/ PR Brazil, 2014 (Catuai Vermelho)	100 + 100 30 day interval		2	5 15 25 35	Beans Beans Beans Beans	0.04 < 0.01 0.02 0.02	- - - -	0.04 < 0.01 0.02 0.02	
Trial: Iracemápolis/SP Brazil, 2014 (Catuai Vermelho)	100 + 100 30 day interval		2	15	Beans	< 0.01	-	< 0.01	

Hops

Table 218 Residues in Hops from supervised trials in EU, Japan and the USA involving 1-2 foliar applications of Fenpyroximate 5% SC or 5% EC

Hops	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: 01 Gambach, Germany, 1989 (Perle)	375	3000	1	0	Green hops	5.2	< 0.5	5.7	Report: R-4032
				7	Green hops	1.6	< 0.5	2.1	
				14	Green hops	0.9	< 0.5	1.4	Study: ER89DEU804
				21	Green hops	0.8	< 0.5	1.3	
				21	Dried hops	6.4	<1.0	7.4	5%SC
Trial: 02 Gambach, Germany, 1989 (North Brewers)	375	3000	1	0	Green hops	7.6	< 0.5	8.1	
				7	Green hops	3.8	< 0.5	4.3	
				14	Green hops	3.1	< 0.5	3.6	
				21	Green hops	3.2	< 0.5	3.7	
				21	Dried hops	4.7	<1.0	5.7	
Trial: 03 Oberrussenried, Germany, 1989 (Hallertauer)	375	4000	1	0	Green hops	2.7	< 0.5	3.2	
				7	Green hops	1.6	< 0.5	2.1	
				14	Green hops	1.5	< 0.5	2	
				21	Green hops	0.5	< 0.5	1	
				21	Dried hops	2.1	<1.0	3.1	
Trial: 04 Oberrussenried, Germany, 1989 (Tettnanger)	375	4000	1	0	Green hops	2.6	< 0.5	3.1	
				7	Green hops	1.1	< 0.5	1.6	
				14	Green hops	1.1	< 0.5	1.6	
				21	Green hops	0.8	< 0.5	1.3	
				21	Dried hops	3	<1.0	4	
Trial: 01 Wolnzach, Germany, 1998 (Perle)	269.2	3526	1	21	Fresh hops	1.59	-	1.59	Report: R-4111
				21	Dried hops	4.38	-	4.38	Study: NHH 094/984984 5%SC
	262	3431	1	21	Fresh hops	1.94	-	1.94	
				21	Dried hops	4.98	-	4.98	
Trial: 02 Wolnzach, Germany, 1998 (Magnum)	280.7	3677	1	21	Fresh hops	1.66	-	1.66	
				21	Dried hops	5.89	-	5.89	
	278	3641	1	21	Fresh hops	1.76	-	1.76	
				21	Dried hops	8.24	-	8.24	
Trial: 03 Wolnzach, Germany, 1998 (Hersbrucker)	263.1	3446	1	21	Fresh hops	1.89	-	1.89	
				21	Dried hops	7.42	-	7.42	
	270.4	3542	1	21	Fresh hops	1.87	-	1.87	
				21	Dried hops	6.22	-	6.22	
Trial: 01 Gambach Germany, 1990 (Brewers Gold)	375	2000	1	0	Green hops	3.1	< 0.5	3.6	Report: R-4063
				7	Green hops	3.7	< 0.5	4.2	
				14	Green hops	3.7	< 0.5	4.2	Study: ER90DEU804
				21	Green hops	1.1	< 0.5	1.6	
				21	Dried hops	6.8	<1.0	7.8	5%SC
Trial: 02 Gambach Germany, 1990 (North Brewers)	375	2000	1	0	Green hops	1.8	< 0.5	2.3	
				7	Green hops	2.1	< 0.5	2.6	
				14	Green hops	2.5	< 0.5	3	
				21	Green hops	< 0.5	< 0.5	<1.0	

Hops	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
				21	Dried hops	8.2	< 1.0	9.2	
Trial: 03	375	2500	1	0	Green hops	5.3	< 0.5	5.8	
Tettnang-Ried				7	Green hops	2.5	< 0.5	3	
Germany, 1990				14	Green hops	2.4	< 0.5	2.9	
(Tettnanger)				21	Green hops	2.1	< 0.5	2.6	
				21	Dried hops	7	< 1.0	8	
Trial: 04	375	4000	1	0	Green hops	4.7	< 0.5	5.2	
Lindau-Bodenegg				7	Green hops	3.5	< 0.5	4	
Germany, 1990				14	Green hops	13.7	< 0.5	14.2	
(Tettnanger)				21	Green hops	4.9	< 0.5	5.4	
				21	Dried hops	<1.0	< 1.0	< 2.0	
Trial: 0301	232	3095	1	0	Green hops	11.3	< 0.5	11.8	Report: R-4064
Gambach				7	Green hops	1.5	< 0.5	2	
Germany, 1991				14	Green hops	< 0.5	< 0.5	< 1	Study: ER91DEU804
(Brewers gold)				21	Green hops	< 0.5	< 0.5	< 1	
				21	Dried hops	1.2	< .0	2.2	5%SC
Trial: 0302	231.5	3088	1	0	Green hops	6.6	< 0.5	7.1	
Gambach				7	Green hops	2.3	< 0.5	2.8	
Germany, 1991				14	Green hops	1.2	< 0.5	1.7	
(North Brewers)				21	Green hops	0.7	< 0.5	1.2	
				21	Dried hops	3.7	< 1.0	4.7	
Trial: 0303	210	2800	1	0	Green hops	< 0.5	< 0.5	< 1	
Lindau-Bodenegg				7	Green hops	2.6	< 0.5	3.1	
Germany, 1991				14	Green hops	1.7	< 0.5	2.2	
(Tettnanger)				21	Green hops	1.6	< 0.5	2.1	
				21	Dried hops	4.3	< 1.0	5.3	
Trial: OR29	178 + 188	949	2	13	Cones	1.2,1.3	-	1.2, 1.3	Report: R-4197
Hubbard, OR	15 day interval	1015				1,25		1.25	
USA, 2002									Study: A8087
(Nugget)									
Trial: WA43	177 + 178	1850	2	15	Cones	1.5,1.2	-	1.5,1.2	5%EC
Prosser, WA USA, 2002 (Nugget)	14 day interval	1836				1.35		1.35	
Trial: ID17	183 + 177	968	2	13	Cones	4.2,3.4	-	4.2,3.4	
Parma, ID USA, 2002 (Zeus)	14 day interval	941				3.8		3.8	
Trial: Iwate	200	40	1	14	Dried cones	4.34	0.11	4.45	Report: R-4375
Iwate				28	Dried cones	1.48	0.05	1.53	
Japan, 1992				42	Dried cones	0.16	< 0.05	0.21	Study not to GLP
(Shinshu-Wase)				56	Dried cones	0.07	< 0.05	0.12	5%SC
Trial: Yamagata	350	70	1	14	Dried cones	7.21	0.16	7.37	
Yamagata				28	Dried cones	7.68	0.31	7.99	

Hops	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Japan, 1992 (Shinshu-Wase)				42	Dried cones	0.44	< 0.05	0.49	
				56	Dried cones	0.15	< 0.05	0.2	
Trial: Iwate Iwate Japan, 1992 (Shinshu-Wase)	200	40	1	14	Dried cones	3.86	0.3	4.16	Report: R-4376
				28	Dried cones	1.47	0.14	1.61	
				42	Dried cones	0.18	< 0.08	0.26	- Study not to GLP
				56	Dried cones	0.12	< 0.08	0.2	5%SC
Trial: Yamagata Yamagata Japan, 1992 (Shinshu-Wase)	350	70	1	14	Dried cones	7.66	0.72	8.38	
				28	Dried cones	7.53	0.74	8.27	
				42	Dried cones	0.48	< 0.08	0.56	
				56	Dried cones	0.19	< 0.08	0.27	

Tea

Table 219 Residues in Tea from supervised trials in India and Japan involving 1-2 foliar applications of Fenpyroximate 5%EC

Tea	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: Ibaraki Ibaraki Japan, 1989 (Kanaya midori)	200	40	1	7	Plucked leaves	13.2	-	13.2	Report: R-4133
				14	Plucked leaves	3.98	-	3.98	
				21	Plucked leaves	1.06	-	1.06	Study not to GLP
				30	Plucked leaves	0.482	-	0.482	5%EC
				7	Tea extract	0.091	-	0.091	
				14	Tea extract	0.027	-	0.027	
				21	Tea extract	0.014	-	0.014	
				30	Tea extract	0.009	-	0.009	
Trial: Kyoto Kyoto Japan, 1989 (Oku midori)	200	40	1	7	Plucked leaves	10.2	-	10.2	
				14	Plucked leaves	2.98	-	2.98	
				21	Plucked leaves	1.48	-	1.48	
				30	Plucked leaves	0.104	-	0.104	
				7	Tea extract	0.07	-	0.07	
				14	Tea extract	0.021	-	0.021	
				21	Tea extract	0.013	-	0.013	
				30	Tea extract	< 0.005	-	< 0.005	
Trial: Kyoto	500	10000	1	7	Dried leaves	21	0.68	21.68	Report: R-4211

Tea Trial Location Country, year (Variety)	Application Rate (g ai/ha)	 Water (L/ha)	 No.	DALA (days)	Commodity	Residues (mg/kg) Fenpyroximate	 M-1	 Sum of Fenpyroximate and M-1	Reference & Comments
Japan, 2008				14	Dried leaves	10.4	0.66	11.06	Study: 24-226
(Yabukita)				21	Dried leaves	3.44	0.2	3.64	Study not to GLP
Trial: Kumamoto	500	10000	1	7	Dried leaves	17.8	0.76	18.56	5%EC
Japan, 2008				14	Dried leaves	5.66	0.33	5.99	
(Oku midori)				21	Dried leaves	1.3	0.07	1.37	
Trial: Saitama Japan, 2010	500+200	10000	2	7	Dried leaves	11.6	1.34	12.94	Report: R-4432
(Fukumidori)		4000		14	Dried leaves	1.8	0.32	2.12	-Study not to GLP
				21	Dried leaves	30.21	0.05	30.26	5%EC
Trial: Mie	500+200	10000	2	7	Dried leaves	31.2	1.86	33.06	
Japan, 2010		4000		14	Dried leaves	11.2	0.75	11.95	
(Yabukita)				21	Dried leaves	3.2	0.23	3.43	
Trial: N1 Jorhat, Assam India, 2004	30		1	7	Green tea leaf	1.15	0.19	1.34	Report: R-4182
(-)				7	Black tea leaf	0.98	0.19	1.17	
				14	Green tea leaf	ND	ND	ND	Study: 3865/04
				14	Black tea leaf	ND	ND	ND	
	60		1	7	Green tea leaf	1.22	0.19	1.41	5%EC
				7	Black tea leaf	1.2	0.15	1.35	
				14	Green tea leaf	0.02	ND	ND	
				14	Black tea leaf	0.02	ND	ND	
Trial: N2 Jorhat, Assam India, 2004	30		1	7	Green tea leaf	0.68	0.03	0.71	
(-)				7	Black tea leaf	0.61	0.04	0.65	
				14	Green tea leaf	< 0.02	ND	ND	
				14	Black tea leaf	< 0.02	ND	ND	
	60		1	7	Green tea leaf	1.23	0.06	1.29	
				7	Black tea leaf	1.29	0.06	1.35	
				14	Green tea leaf	0.16	0.02	0.18	
				14	Black tea leaf	0.16	0.02	0.18	
Trial: N3 Jorhat, Assam India, 2004	30		1	7	Green tea leaf	1.44	0.1	1.54	
(-)				7	Black tea leaf	0.78	0.05	0.83	
				14	Green tea leaf	ND	ND	ND	
				14	Black tea leaf	ND	ND	ND	
	60		1	7	Green tea leaf	1.99	0.12	2.11	
				7	Black tea leaf	1.27	0.07	1.34	
				14	Green tea leaf	0.05	< 0.02	ND	
				14	Black tea leaf	0.03	ND	ND	
Trial: N4	30		1	7	Green tea	0.3	0.03	0.33	

Tea Trial Location Country, year (Variety)	Application Rate (g ai/ha)	 Water (L/ha)	 No.	DALA (days)	Commodity	Residues (mg/kg) Fenpyroximate	 M-1	 Sum of Fenpyroximate and M-1	Reference & Comments
Jorhat, Assam India, 2004 (-)					leaf				
				7	Black tea leaf	0.27	0.03	0.3	
				14	Green tea leaf	< 0.02	ND	ND	
				14	Black tea leaf	0.02	ND	ND	
	60		1	7	Green tea leaf	0.31	0.04	0.35	
				7	Black tea leaf	0.3	0.05	0.35	
				14	Green tea leaf	0.04	< 0.02	ND	
				14	Black tea leaf	0.02	< 0.02	ND	
Trial: S1 UPASI, Valparai, Tamili Nadu India, 2004 (-)	30		1	7	Green tea leaf	3.89	0.16	4.05	
				7	Black tea leaf	2.8	0.21	3.01	
				14	Green tea leaf	0.44	0.03	0.47	
				14	Black tea leaf	0.2	0.02	0.22	
	60		1	7	Green tea leaf	4.1	0.26	4.36	
				7	Black tea leaf	3.97	0.34	4.31	
				14	Green tea leaf	1.25	0.08	1.33	
				14	Black tea leaf	0.79	0.1	0.89	
Trial: S2 UPASI, Gudalur, Tamili Nadu India, 2004 (-)	30		1	7	Green tea leaf	3.7	0.14	3.84	
				7	Black tea leaf	2.97	0.14	3.11	
				14	Green tea leaf	1.21	0.08	1.29	
				14	Black tea leaf	0.95	0.07	1.02	
	60		1	7	Green tea leaf	3.87	0.14	4.01	
				7	Black tea leaf	3.44	0.16	3.6	
				14	Green tea leaf	1.94	0.13	2.07	
				14	Black tea leaf	1.45	0.11	1.56	
Trial: S3 UPASI, Valparai, Tamili Nadu India, 2004 (-)	30		1	7	Green tea leaf	0.95	0.03	0.98	
				7	Black tea leaf	0.67	0.03	0.7	
				14	Green tea leaf	0.12	ND	0.12	
				14	Black tea leaf	0.11	< 0.02	0.13	
	60		1	7	Green tea leaf	0.99	0.04	1.03	
				7	Black tea leaf	1.33	0.06	1.39	
				14	Green tea leaf	0.15	< 0.02	0.17	
				14	Black tea leaf	0.11	< 0.02	0.13	
Trial: S4 UPASI, Gudalur,	30		1	7	Green tea leaf	0.8	0.02	0.82	
				7	Black tea leaf	0.93	0.05	0.98	

Tea	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Tamili Nadu									
India, 2004 (-)				14	Green tea leaf	0.15	< 0.02	0.17	
				14	Black tea leaf	0.09	< 0.02	0.11	
	60		1	7	Green tea leaf	2.72	0.06	2.78	
				7	Black tea leaf	2.39	0.11	2.5	
				14	Green tea leaf	0.69	0.04	0.73	
				14	Black tea leaf	0.67	0.06	0.73	
Trial: UPASI	25	400	1	0	Black tea	4.76	-	4.76	Report: R-4461
India, 2005				7	Black tea	1.78	-	1.78	- Study not to GLP
(mix of cultivars)				10	Black tea	0.73	-	0.73	5%EC
				14	Black tea	0.48	-	0.48	
Trial: UPASI	25	400	1	0	Black tea	17.29	-	17.29	
India, 2004				1	Black tea	15.74	-	15.74	
(mix of cultivars)				3	Black tea	8.56	-	8.56	
				5	Black tea	6.33	-	6.33	
				7	Black tea	3.01	-	3.01	
				10	Black tea	1.01	-	1.01	
				14	Black tea	0.21	-	0.21	
	50	400	1	0	Black tea	32.01	-	32.01	
				1	Black tea	20.71	-	20.71	
				3	Black tea	12.45	-	12.45	
				5	Black tea	11.59	-	11.59	
				7	Black tea	4.85	-	4.85	
				10	Black tea	2.78	-	2.78	
				14	Black tea	0.59	-	0.59	

Animal feed

Bean forage

Table 220 Residues in Snap Beans forage from supervised trials in USA involving foliar applications of Fenpyroximate 5% EC

Beans	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial CA38 Davis, CA USA, 2008 (Blue Lake Bush 274)	110 + 109 14 day interval	299 290	2	1 3 8 10	Foliage Foliage Foliage Foliage	2.07,1.76, 1.92 1.69,1.61, 1.65 0.88,0.99, 0.93 0.83,0.74, 0.79	1.28, 1.15, <1.22 1.61,1.43, 1.52 0.79,0.84, 0.82 0.62,0.52, 0.57	3.35, 2.91, <u>3.14</u> 3.3,3.04, 3.17 1.67,1.83, 1.75 1.45,1.26, 1.36	Report: R-4458 Study IR-4 PR No. 09942 5%EC
Trial FL37 Citra, FL USA, 2008 (Dusky)	110 + 109 5 day interval	374 364	2	1	Foliage	0.96,0.83, 0.90	0.37,0.31 0.34	1.33,1.14 <u>1.24</u>	
Trial GA*13 Tifton, GA USA, 2008 (Blue Lake Bush 274)	112 + 113 13 day interval	449 449	2	1	Foliage	2.16,2.73 2.45	0.55,0.53 0.54	2.71,3.26 <u>2.99</u>	
Trial NY24 Freeville, NY USA, 2008 (Hystyle)	111 + 111 13 day interval	318 318	2	1	Foliage	3.38,3.45 3.42	1.01,1.05 1.03	4.39,4.5 <u>4.45</u>	
Frrmont, OH09 USA, 2008 (Sea Biscuit)	110 + 108 14 day interval	374 364	2	1	Foliage	0.02,0.02 < 0.05	< 0.05, < 0.05 < 0.05	0.07,0.07, <u>< 0.07</u>	
Trial OH*10 Fremont, OH USA, 2011 (Brio)	106 + 111 14 day interval	364 374	2	1	Foliage	< 0.05, < 0.05 < 0.05	< 0.05, < 0.05 < 0.05	< 0.10, < 0.10 <u>< 0.10</u>	
Trial WA*07 Moxee, WA USA, 2011 (Jade)	114 + 111 14 day interval	374 364	2	1	Foliage	1.70,2.04 1.87	0.62,0.77 0.70	2.32,2.81 <u>2.57</u>	
Trial WI17 Arlington, WI USA, 2011 (Hystyle)	113, 111 13 day interval	393 383	2	1	Foliage	5.1,6.5, 5.80	0.93,1.01 0.97	6.03,7.51 <u>6.77</u>	

Maize forage and stover

Table 221 Residues in forage and stover of field maize from supervised trials in USA involving 2 foliar applications of Fenpyroximate 5%EC

Maize	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: 01 Stafford, KS USA, 2010 (P0541HR)	113 + 113 14 day interval	94 94	2	14	Forage	0.312, 0.274 0.29	0.0878, 0.0829 0.09	0.3998, 0.356 0.38	Report: R-4447 Study: TCI-10-270 5%EC
	109 + 108 14 day interval	94 94	2	13	Stover	0.617,1.49, 1.05	0.193,0426, 0.310	0.81,1.916 , 1.36	
Trial: 02 Hinton, OK USA, 2010 (Syngenta NK-N72K)	112 + 113 14 day interval	94 94	2	7 14 21 28	Forage Forage Forage Forage	0.720,0.77, 0.75 0.556,0.76, 0.66 0.698,0.67 0,0.68 0.284,0.35 0,0.32	0.520,0.56 , 0.54 0.352,0.51 ,0.43 0.447,0.42 ,0.44 0.173,0.21 ,0.19	1.24,1.335, 1.29 0.908,1.272, 1.09 1.145,1.094, 1.12 0.457,0.563, 0.51	
	112 + 115 14 day interval	94 94	2	7 14 21 28	Stover Stover Stover Stover	2.54,2.08 , 2.31 1.38,1.92 , 1.65 2.18,1.31 , 1.75 0.906,107 0.99	1.41,1.19 , 1.30 0.917,122 1.07 1.57,0.939 1.25 0.749,0.85 0.80	3.95,3.27, 3.61 2.297,3114 , 2.72 3.75,2.249 3.0 1.655,1.919 1.79	
Hinton, OK USA, 2010 (Pioneer 33B54)	111 + 110 14 day interval	94 94	2	14	Forage	0.432,0.552 0.49	0.269,0.370 0.32	0.701,0.918 0.81	
	110 + 115 15 day interval	94 94	2	13	Stover	1.67,1.60 1.64	1.65,1.61 1.63	3.32,3.21, 3.27	
Trial: 04 Uvalde, TX USA, 2010 (D16 6721)	111 + 109 14 day interval	94 94	2	13	Forage	0.228,0.396 0.31	0.118,0.210 0.16	0.346,0.604 0.47	
	112 + 111 14 day interval	94 94	2	14	Stover	1.33,1.73 1.53	0.574,0.82 0.698	2.98,2.552 2.228	
Trial: 05 Raymondville, TX USA, 2010 (H6284162)	110 + 115 14 day interval	94 94	2	14	Forage	0.214,0.172 0.19	0.143,0.120 0.13	0.357,0.291 0.32	
	115 + 114 13 day interval	94 94	2	14	Stover	2.08,2.30 2.19	1.65,1.72 1.685	3.73,4.02, 3.875	
Trial: 06 Dill City, OK USA, 2010 (Syngenta NK-N72K)	113 + 112 14 day interval	94 94	2	14	Forage	0.262,0.176 0.22	0.133,0.080 0.11	0.395,0.2566 0.33	
	114 + 114 14 day interval	94 94	2	13	Stover	0.877,1.17 1.02	0.799,1.00 0.900	1.676,2.17 1.92	
Trial: 07 Levelland, TX USA, 2010 Field corn (Agventure R350VBW)	111 + 110 14 day interval	94 94	2	14	Stover	1.83,1.37 1.600	1.07,0.808 0.939	2.9,2.178 2.539	
Trial: 08 Larned, KS USA, 2010 Field corn(33Y75)	109 + 112 14 day interval	94 94	2	13	Forage	0.488,0.398, 0.44	0.157,0.130 0.14	0.645,0.524 0.58	
	111 + 112 14 day interval	94 94	2	14	Stover	1.007	0.499,0.370 0.435	1.619,1.265 1.442	
Trial: 09 Belpre, KS USA, 2010 (Pioneer 33D49)	111 + 112 14 day interval	94 94	2	13	Forage	0.24, 0.24 0.24	0.134,0.120 0.13	0.382,0.362 0.37	
	109 + 111 14 day interval	94 94	2	14 14	Stover	1.04,1.10 1.070	0.44,0.56 0.498	1.477,1.658 1.568	

Maize	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of fenpyroximate and M-1	
Trial: 10 Pierce, CO USA, 2010 Field corn(LG 2407)	112 + 113 14 day interval	94 94	2	14	Forage	0.168,0.184 0.18	0.117,0.140.13	0.285,0.322 0.31	
	109 + 109 14 day interval	94 94	2	14	Stover	0.160,0.184 0.172	0.115,0.140.126	0.275,0.32 0.298	

Maize silage

Table 222 Residues in Maize silage from supervised trials in EU involving one foliar application of Fenpyroximate 5% SC

Maize	Application			DALA	Commodity	Residues (mg/kg)			Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: S11-02947-01 HUNGARY EU, 2011 (DKC 4490)	56.4	329	1	0 7 14 21 28	Silage Silage Silage Silage Silage	1.19 0.93 1.1 0.71 0.81	0.02 0.06 0.09 0.08 0.09	1.21 0.99 1.19 0.79 0.9	Report: R-4471 Study: S11-02947 5%SC
Trial: S11-02947-02 HUNGARY EU, 2011 (DK 4590)	54.4	318	1	0 7 14 21 28	Silage Silage Silage Silage Silage	0.79 0.89 0.5 0.69 0.21	< 0.01 0.06 0.04 0.06 0.03	0.8 0.95 0.54 0.75 0.24	

Almond hull

Table 223 Residues in almond hull from supervised trials in USA involving one foliar application of Fenpyroximate 5% EC

Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: CA1 Porterville, CA USA, 2001 (Mission)	451	944	1	14	Hulls	0.547,0.559 0.553	0.215,0.191 0.203	0.762, 0.75 0.756	Report: R-4155 Study: AA010709 5%EC
Trial: CA2 Terra Bella, CA USA, 2001 (Carmel)	448	935	1	14	Hulls	0.998,1.13 1.064	0.277,0.312 0.295	1.275,1.442 1.359	
Trial: CA3 Hanford, CA USA, 2001 (Carmel)	449	925	1	14	Hulls	0.889,1.14 1.015	0.178,0.224 0.201	1.067,1.364 1.216	

Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1	
Trial: CA4 Hanford, CA USA, 2001 (Mission)	450	925	1	14	Hulls	0.871,1.14 1.006	0.256,0.285 0.271	1.127,1.425 1.277	
Trial: CA5 Hanford, CA USA, 2001 (Prices)	447	935	1	0 7 14 21 28	Hulls Hulls Hulls Hulls Hulls	1.12,1.10, 1.11 1.16,0.961, 1.061 0.355,0.305 0.330 0.642,0.740 0.691 0.248,0.296 0.272	0.0895, 0.0788, 0.084 0.286,0.247, 0.267 0.129,0.118, 0.124 0.232,0.225, 0.229 0.117,0.135, 0.126	1.2095, 1.1788, 1.194 1.446,1.208, 1.328 0.484,0.423, 0.454 0874,0.965, 0.920 0.365, 0.431, 0.398	

FATE OF RESIDUES IN STORAGE AND PROCESSING

Hydrolysis

Hydrolysis of fenpyroximate under simulated processing conditions was studied with [pyrazole-3-^{14}C]fenpyroximate to investigate nature of degradation in aqueous buffered solutions at a nominal concentration of 1 mg/L at pH of 4, 5 and 6 (Penketh, S., 2008, Report No R-4475). Fenpyroximate and degradates were determined by liquid scintillation counting and analysed by reversed phase HPLC. The principal degradate was M-3 (41.0–88.7% after incubation). Degradates M-1 and M-6 were found at ≤ 3.0%. An unidentified breakdown product was found in the pH 4 and 5 samples that was not M-11. The results are summarised below table and are expressed as a percentage of applied radioactivity (% AR).

Table 224 Results of hydrolysis of fenpyroximate under simulated pasteurization, brewing/baking/boiling and sterilization

	pH 4, 90 °C, 20 minutes representing pasteurisation	pH5, 100 °C, 60 minutes representing brewing, baking and boiling	pH 6, 120 °C, 20 minutes representing sterilisation
Total recovery	92.3	94.9	94.5
Fenpyroximate	47.2	3.1	7.2
M-3	41.0	86.2	88.7
M-6	1.6	3.0	0.5
M-1	1.4	nd	nd
Others*	6.4	3.9	3.6

nd - Not detected

*: not associated with specific components

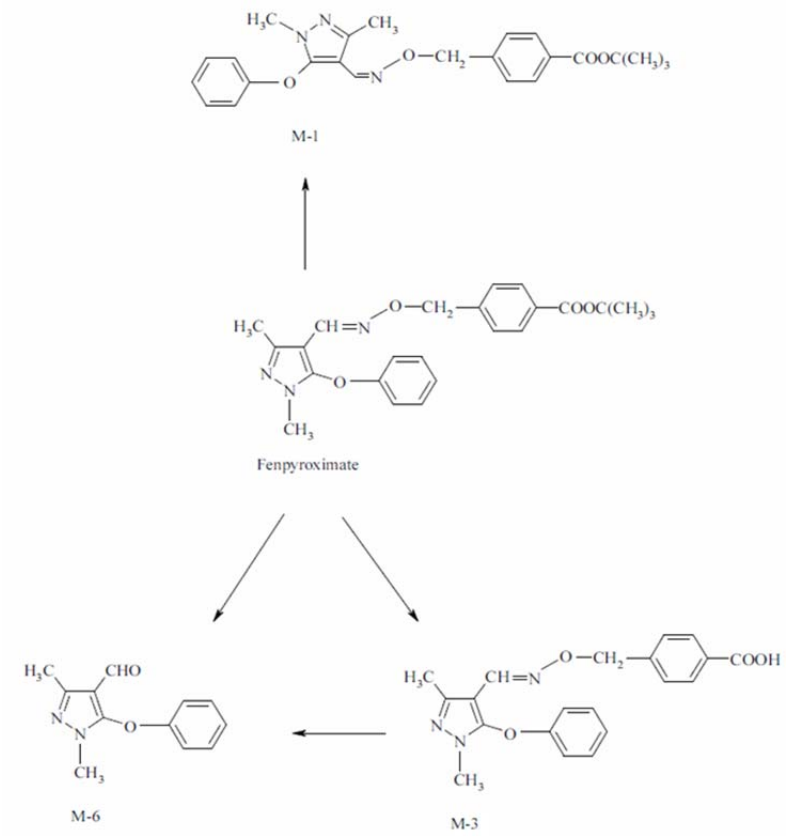


Figure 6 Proposed degradation pathway for fenpyroximate under simulated processing conditions

Fresh beans

Two processing Studies for Beans with Pods after one application of fenpyroximate at rate of 0.5 kg ai/ha (5× exaggerated rate) in France and Italy was conducted (Jullian E., 2015. report No.: R-4516). Beans with pod were sampled by hand 7 days after application. Fresh beans and the processed fractions (washed beans, cooked beans and canned beans) were analysed for residues of fenpyroximate, M-1 and M-3 using the multi-residue method QuEChERS by LC-MS/MS, the LOQ was 0.01 mg/kg.

Table 225 Residue trials with fenpyroximate 51.2 g/L SC in fresh beans performed for processing

Trial Ref Location Crop Year	Application		Process tested -	Portion analysed	Residue (mg/kg)			Total residue (mg/kg, fenpyroximate equivalents)	Processing factor (matrix / RAC)*	Reference and comments
	No.	kg a.s./ha			Fenpyroximate	M-1	M-3			
82000, Montauban, France 2015	1	0.526	-	Fresh beans (RAC)	1.36	0.03	n/a	1.39	-	R-4516
			Washing	Washed beans	1.70	0.02	< 0.01	<1.73	1.2	
			Cooking	Cooked beans	0.97	0.03	< 0.01	<1.01	0.73	
			Canning	Canned beans	0.47	0.04	0.07	0.58	0.42	
48012, Bagnacavallo, Italy 2015	1	0.480	-	Fresh beans (RAC)	0.89	0.03	n/a	0.92	-	
			Washing	Washed beans	0.79	0.02	< 0.01	< 0.82	0.89	
			Cooking	Cooked beans	0.40	0.02	< 0.01	< 0.43	0.47	
			Canning	Canned beans	0.36	0.03	0.03	0.42	0.46	

* Processing factor calculated as the sum of fenpyroximate + M-1 in the RAC divided by the sum of fenpyroximate + M-1 + M-3 in the processed commodity.
Residues <LOQ have been assumed to be at the LOQ for the purposes of this calculation

Apples

Processing studies for apples from two trials after one application of fenpyroximate at rate of 0.5 kg ai/ha were conducted in Poland and Germany (Jullian E. 2016, report No.: R-4515) during 2015. Samples from the untreated and treated plots were taken by hand 21 days after application. After processing the specimens were immediately stored deep frozen (approximately -20 °C) in a freezer at the processing test site until shipping for analytical phase. Apples prior to processing, were analysed for residues of fenpyroximate and its metabolite M-1 using the multi-residue method QuEChERS with an LOQ of 0.01 mg/kg. Apple processed fractions (washed apples, wet pomace, dry pomace, pasteurised apple juice, pasteurised apple sauce and dried apples) were analysed for residues of fenpyroximate, M-1 and M-3 using the multi-residue method QuEChERS with an LOQ of 0.01 mg/kg. The residue levels of fenpyroximate, M-1 and M-3 found in apple fruits, prior to processing and processed fractions are summarised in Table 226.

Table 226 Residue trials with fenpyroximate 51.2 g/L SC in apples performed for processing

Doc. No. Trial Ref Location Crop Year	Application		Process tested -	Portion analysed	Residue (mg/kg)			Total residue (mg/kg, fenpyroximate equivalents)	Processing factor (matrix / RAC)*	Reference and comments
	No.	kg a.s./ha			Fenpyroximate	M-1	M-3			
S15-03109-01, 64-530, Zapust, Poland 2015	1	0.476	- Washing Pressing Pressing Pasteurisation Pasteurisation Drying	Fresh apple (RAC) Washed fruits Wet Pomace Dry Pomace Pasteurised juice Pasteurised sauce Dried apples	0.16 0.10 0.36 0.81 < 0.01 < 0.01 0.61	< 0.01 < 0.01 0.01 0.04 < 0.01 < 0.01 0.03	n/a < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.17 < 0.12 < 0.38 < 0.86 < 0.03 < 0.03 < 0.65	- 0.71 2.2 5.1 0.18 0.18 3.8	R-4515
S15-03109-02, 21683, Stade, Germany 2015	1	0.556	- Washing Pressing Pressing Pasteurisation Pasteurisation Drying	Fresh apple (RAC) Washed fruits Wet Pomace Dry Pomace Pasteurised juice Pasteurised sauce Dried apples	0.21 0.21 1.11 2.45 < 0.01 0.02 1.03	0.01 0.02 0.08 0.17 < 0.01 < 0.01 < 0.07	n/a < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.22 < 0.24 <1.20 <2.63 < 0.03 < 0.04 <1.11	- 1.1 5.5 12.0 0.14 0.18 5.0	

* Processing factor calculated as the sum of fenpyroximate + M-1 in the RAC divided by the sum of fenpyroximate + M-1 + M-3 in the processed commodity.
Residues <LOQ have been assumed to be at the LOQ for the purposes of this calculation.

Tomatoes

Two processing studies for tomatoes after one application of fenpyroximate at rate of 0.5 kg ai/ha in Italy and Spain (Jullian E., 2016, report No.: R-4518) were received. Fruits were sampled by hand 7 days after application. The tomato fruits and processed fractions (washed tomatoes, tomato juice, canned tomatoes and tomato puree) were analysed for residues of fenpyroximate, M-1 and M-3 using the multi-residue method QuEChERS by LC-MS/MS, LOQ was 0.01 mg/kg.

Table 227 Residue trials with fenpyroximate 51.2 g/L SC in tomatoes performed for processing

Trial Ref Location Crop Year	Application		Process tested -	Portion analysed	Residue (mg/kg)			Total residue (mg/kg, fenpyroximate equivalents)	Processing factor (matrix / RAC)*	Reference and comments
	No.	kg a.s./ha			Fenpyroximate	M-1	M-3			
S15-03111-01,44023, Lagosanto, Emilia Italy 2015	1	0.519	- Washing Juicing Canning Pureeing	Whole tomato (RAC) Washed tomatoes Tomato juice Canned tomatoes Puree	0.13 0.10 0.09 0.05 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	n/a < 0.01 < 0.01 < 0.01 < 0.01	< 0.13 < 0.12 < 0.11 < 0.07 < 0.10	- 0.92 0.85 0.54 0.76	R-4518
S15-0311-	1	0.510	-	Whole tomato	0.11	< 0.01	n/a	< 0.12	-	

Trial Ref Location Crop Year	Application		Process tested	Portion analysed	Residue (mg/kg)			Total residue (mg/kg, fenpyroximate equivalents)	Processing factor (matrix / RAC)*	Reference and comments
	No.	kg a.s./ha	-		Fenpyroximate	M-1	M-3			
02, 41790, Santiponce, Spain 2015				(RAC)						
			Washing	Washed tomatoes	0.08	< 0.01	< 0.01	< 0.10	0.83	
			Juicing	Tomato juice	0.03	< 0.01	< 0.01	< 0.05	0.42	
			Canning	Canned tomatoes	0.01	< 0.01	< 0.01	< 0.03	0.25	
			Pureeing	Puree	0.06	< 0.01	< 0.01	< 0.08	0.67	

* Processing factor calculated as the sum of fenpyroximate + M-1 in the RAC divided by the sum of fenpyroximate + M-1 + M-3 in the processed commodity.
Residues <LOQ have been assumed to be at the LOQ for the purposes of this calculation.

Grapes

Two processing studies for grapes after one application of fenpyroximate at rate of 0.25 kg ai/ha in France and Spain (Jullian E., 2016, report No.: R-4517) were received. Fruits were sampled by hand 28 days after application. Grapes, and processed fractions (wet pomace, wine, pasteurised juice, washed and destemmed grapes and raisins) were analysed for residues of fenpyroximate, M-1 and M-3 using the multi-residue method QuEChERS by LC-MS/MS, LOQ was 0.01 mg/kg. The residue levels of fenpyroximate, M-1 and M-3 found in grapes (RAC) and processed fractions are summarised in Table 228.

Table 228 Residues of fenpyroximate, M-1 and M-3 in grapes RAC and processed fractions

Trial Ref Location Crop Year	Application		Process tested	Portion analysed	Residue (mg/kg)			Total residue (mg/kg, fenpyroximate equivalents)	Processing factor (matrix / RAC)*	Reference and comments
	No.	kg a.s./ha			Fenpyroximate	M-1	M-3			
S15-03110-01，66200, Elne, France, 2015	1	0.255	-	Whole fruit (RAC)	0.10	< 0.01	n/a	0.11	-	R-4517
				Wet pomace						
			Pomace	Wine	0.48	0.03	< 0.01	< 0.52	4.7	
			Wine	Pasteurised juice	< 0.01	< 0.01	< 0.01	< 0.03	0.27	
			Juicing	Washed grapes	< 0.01	< 0.01	< 0.01	< 0.03	0.27	
			Washing	Raisins	0.07	< 0.01	< 0.01	< 0.09	0.82	
			Drying		0.29	0.02	< 0.01	< 0.32	2.9	
S15-03110-02，50549, El Buste, Spain, 2015	1	0.248	-	Whole fruit (RAC)	0.67	0.01	n/a	0.68	-	
				Wet pomace						
			Pomace	Wine	3.37	0.09	< 0.01	<3.47	5.10	
			Wine	Pasteurised juice	< 0.01	< 0.01	< 0.01	< 0.03	0.04	
			Juicing	Washed grapes	< 0.01	< 0.01	< 0.01	< 0.03	0.04	
			Washing	Raisins	0.39	< 0.01	< 0.01	< 0.41	0.60	
			Drying		0.70	0.02	< 0.01	< 0.73	1.1	

* Processing factor calculated as the sum of fenpyroximate + M-1 in the RAC divided by the sum of fenpyroximate + M-1 + M-3 in the processed commodity.
Residues <LOQ have been assumed to be at the LOQ for the purposes of this calculation

Strawberry

Two processing studies for strawberries after one application of fenpyroximate at rate of 0.5 kg ai /ha in Italy and Spain were conducted (Jullian E., 2016 report No.: R-4512). Samples of fruit (RAC) were taken 7 days after application for processing. RAC samples were transferred to the laboratory for processing according to simulated commercial practices with samples taken for washing, canning and jam. All samples were then stored frozen until required for analysis. Strawberry fruits and processed fractions (washed strawberry, canned strawberry and pasteurised jam) were analysed for residues of fenpyroximate, M-1 and M-3 according to the validated method RES/RAM/004. LOQ for fenpyroximate, M-1 and M-3 in strawberry fruit and processed matrices was 0.01 mg/kg. The residue levels of fenpyroximate, M-1 and M-3 found in strawberry (RAC) and processed fractions are summarised in Table 229.

Table 229 Residue of fenpyroximate, M-1 and M-3 in strawberry and processed fractions (data from 5× exaggerated rate)

Trial Ref Location Crop Year	Application		Process tested	Portion analysed	Residue (mg/kg)			Processing factor (matrix / RAC)*	Reference and comments
	No.	kg a.s./ha			Fenpyroximate	M-1	M-3		
S15-03112-01 Bologna Italy, 2016	1	0.508	- - Canning Jam	Whole fruit (RAC) Washed fruit Canned fruit Jam	0.58 0.31 0.03 0.11	< 0.01 < 0.01 < 0.01 < 0.01	ND < 0.01 < 0.01 < 0.01	- 0.53 0.05 0.19	R-4512
S15-03112-02 Cadiz Spain, 2015	1	0.498	- - Canning Jam	Whole fruit (RAC) Washed fruit Canned fruit Jam	0.82 0.47 0.16 0.30	0.03 0.02 < 0.01 0.02	ND < 0.01 < 0.01 0.02	- 0.58 0.19 0.38	

* Processing factor calculated as sum of individual processing factors for fenpyroximate and M-1

Orange

Processing studies for orange after 2 applications of fenpyroximate at rate of 0.5 kg ai/ha were conducted in the USA during 1994/95 (Jullian E. 2016, Report No. R-4107). Samples from the untreated and treated plots were taken 14 days after application. After processing the specimens were immediately stored deep frozen (approximately -20 °C) in a freezer at the processing test site until shipping for analytical phase. Oranges prior to processing and processed fractions (molasses, oil, dried pulp and juice) were analysed for residues of fenpyroximate and M-1. The residue levels of fenpyroximate, M-1 in orange fruits and processed fractions are summarised in Table 230.

Table 230 Residue trials with fenpyroximate 5% SC in oranges performed for processing

Trial Location Year (Variety)	Application		Process tested -	Portion analysed	Residue (mg/kg)		Total residue (mg/kg, fenpyroximate equivalents)	Processing factor *	Reference and comments
	No.	kg ai/ha			Fenpyroximate	M-1			
Trial: CA4 Tulare, CA USA, 1994- 1995 (Navel)	2	0.5+0.5 14 days interval	- Juice Molasses Oil Dried	Fresh fruits (RAC) Juice Molasses Oil Dried fruit	0.420 ND 0.032 30.9 2,22	< 0.008 < 0.008 < 0.008 < 0.008 < 0.046	0.428 < 0.008 0.040 30.908 2.266	- < 0.019 0.093 72.2 5.3	Report No. R-4107; Study: AA940422
Trial: FL2 Palm Beach FL; USA, 1994- 1995 (Hamlin)	1	0.5+0.5 14 days interval	- Juice Molasses Oil Dried	Fresh fruits (RAC) Juice Molasses Oil Dried fruit	0.357 < 0.008 0.009 4.67 1.69	0.01 < 0.008 < 0.008 0.220 0.146	0.367 < 0.016 0.017 4.89 1.836	- < 0.044 0.046 13.3 5.0	

* Processing factor calculated as the sum of fenpyroximate + M-1 in the RAC divided by the sum of fenpyroximate + M-1 in the processed commodity.
Residues <LOQ have been assumed to be at the LOQ for the purposes of this calculation.

Maize

One processing study for maize after 2 application of fenpyroximate at rate of 570 g ai /ha in the USA (report No. R-4447). Maize grain was sampled at 14 DAT. The grain was used to generate the aspirated grain fraction and process whole grain into grifts, meal, flour, starch, and refined oil (wet and dry milled).

Table 231 Residue trials with fenpyroximate in maize grains performed for processing

Trial Location Year (Variety)	Application		Portion analysed	Residue (mg/kg)		Total residue (mg/kg, fenpyroximate equivalents)	Processing factor *	Reference and comments
	No.	kg ai/ha		Fenpyroximate	M-1			
Trial: TCI-10-270-04 Uvalde,TX USA, 2010		1.14	Grains (RAC) Grits Meal Flour Refined oil (dry milling) Starch Refined oil (wet milling)	0.206 0.00332 0.0266 0.066 0.175 - 0.0563	0.00527 - 0.00574 0.0129 0.0336 - 0.00901	0.211 0.00332 0.03234 0.0789 0.2086 - 0.06531	- 0.016 0.15 0.37 0.99 0.31	Report No. R-4447;
Trial: TCI-10-270-04 Uvalde,TX USA, 2010		0.223	Grain (RAC) Aspirated grain fraction	0.01255 1.23	0.00617 0.387	0.01872 1.617	- 86	

Potato

The Meeting received one processing study on potato from the USA. A potato crop received 2 applications of fenpyroximate at a rate of 570 g ai/ha (Study IR-4 PR No.10173). The potato tubers were sampled at 7 DAT, and processed to flakes, chips and wet peel.

Table 232 Processing study for potato

Potato	Application			DALA		Residues (mg/kg)			Processing factor	Reference & Comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)	Commodity	Fenpyroximate	M-1	Sum of Fenpyroximate and M-1		
Trial 09-ID12 Kimberley, ID USA, 2009 (Russet Burbank)	565 + 567	422 424	2	7	Tubers (RAC)	< 0.05	< 0.05	<u>≤ 0.1</u>	-	
					Flakes Chips Wet Peels	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1	- - -	Study IR-4 PR No.10173

Tea

Two processing studies for tea after one application of fenpyroximate at rate of 200 g ai /ha in Ibaraki and Kyoto Prefectures in Japan (report No. R-4133). Tea leaves was sampled at 7, 14, 21 and 30 DAT for processing. The dried tea leaves were soaked with 100 °C of water, and after standing to cool, the extract was filtered. Then the solution was added with sodium chloride, and then extracted with hexane. The hexane phase was dried *in vacuo*, and cleaned up by using silica-gel column and C18 cartridge column chromatography. The analytes (*E* and *Z* isomers of fenpyroximate) were determined by using a High Performance Liquid Chromatography (HPLC) equipped with UV detector. For both analytes, the limit of detection was 0.005 mg/kg and the average recoveries at 0.2 mg/kg fortified to control samples of *E* and *Z* isomers were 83% and 80%, respectively.

Table 233 Processing study for Tea

Tea	Application			DALA	Commodity	Residues (mg/kg)			Processing factor	Reference & comments
Trial Location Country, year (Variety)	Rate (g ai/ha)	Water (L/ha)	No.	(days)		Fenpyroximate	M-1	Sum of Fenpyroximate and M-1		
Trial: Ibaraki Ibaraki Japan, 1989 (Kanaya midori)	200	40	1	7	Plucked leaves	13.2	-	13.2		R-4133
				14	Plucked leaves	3.98	-	3.98		
				21	Plucked leaves	1.06	-	1.06		
				30	Plucked leaves	0.482	-	0.482		
				7	Tea extract	0.091	-	0.091	0.0069	
				14	Tea extract	0.027	-	0.027	0.0068	
				21	Tea extract	0.014		0.014	0.013	
				30	Tea extract	0.009		0.009	0.019	
Trial: Kyoto Kyoto Japan, 1989 (Oku midori)	200	40	1	7	Plucked leaves	10.2	-	10.2		R-4133
				14	Plucked leaves	2.98	-	2.98		
				21	Plucked leaves	1.48		1.48		
				30	Plucked leaves	0.104	-	0.104		
				7	Tea extract	0.07	-	0.07	0.0069	
				14	Tea extract	0.021	-	0.021	0.007	
				21	Tea extract	0.013		0.013	0.0088	
				30	Tea extract	< 0.005	-	< 0.005	0.048	

LIVESTOCK FEEDING STUDIES

The Meeting received the information on a lactating dairy cow feeding study (Baker F.C., Bacher R. and Gibson N., 1999, report No. R-4113). Holstein dairy cows were treated orally with gelatine capsules fortified with fenpyroximate once daily for 29 consecutive days. Groups of cows were dosed at three dose levels (3 cows/dose group) equal to 1.0, 3.0 and 10.0 ppm fenpyroximate equivalents in the diet (dry weight basis) for the low (1×), medium (3×) and high (10×) dose groups, corresponded to 0.03, 0.10 and 0.35 mg ai/kg body weight. Milk samples were collected twice a day (morning and evening) on dose days 0, 1, 3, 7, 11, 14, 18, 21, 24 and 28. Milk samples were composited as a proportion of the level of morning and evening milk production, and stored frozen prior to analysis. Cows were sacrificed between 15 and 22 hours after their last dose. Animals were examined for gross tissue abnormalities and the tissue samples of liver, kidney, composite muscle (round and loin) and composite fat (perirenal and omental) were collected, homogenised in a frozen state with dry ice and stored frozen prior to analysis. Samples were extracted with acetone, purified and subsequently analysed by GC/NPD or LC-MS/MS (liver, kidney). The limit of quantification for each analyte (fenpyroximate/Fen-OH, M-21, M-22, M-3, N-desmethyl-M-3) was 0.005 ppm in milk and 0.01 ppm in all tissues. The limit of detection for milk, muscle and fat method (GC/NPD) was approximately 0.1 µg/mL. The limit of detection for the LC/MS/MS analysis of liver and kidney was also approximately 0.005 µg/mL. All residue values were corrected for recovery of concurrent fortification samples under 100%. In addition storage stability studies were conducted to evaluate the stability of the analytes when stored frozen up to 82 days before extraction.

Milk results indicate that the mean daily fenpyroximate/Fen-OH residues in high dose milk reached a maximum level of 0.017 ppm 1 day and 3 days after the first dose. Mean daily residues of fenpyroximate/Fen-OH appeared to decline gradually over the remainder of the study except for an outlier at day 21 (0.016 ppm). High dose milk contained 0.010 ppm at day 28. Medium dose milk contained < 0.005 ppm fenpyroximate/Fen-OH at day 3, 0.008 ppm at day 14 and 0.005 ppm at day 21. Metabolite M-21 was detected below the method LOQ (< 0.005 ppm) in some high dose milk samples throughout the study. M-21 was not detected in day 3 medium dose milk and was < 0.005 ppm in day 14 and day 21 milk. Low dose milk was not analysed because of the low, or undetectable, residues observed in medium dose samples. Result are summarised in Table 234.

Muscle from animals receiving a high dose of fenpyroximate contained an average of 0.038 ppm fenpyroximate/Fen-OH and < 0.010 ppm of M-3. Muscle from medium dose animals

contained 0.015 ppm fenpyroximate/Fen-OH; M-3 was undetectable. The latter result was confirmed in one sample by LC-MS/MS analysis, which allowed a lower limit of detectability. Low dose muscle contained < 0.010 ppm fenpyroximate/Fen-OH.

Fat from high dose animals contained an average of 0.105 ppm fenpyroximate/Fen-OH. Residues of M-3 were < 0.01 ppm. Fat from animals receiving a medium dose of fenpyroximate contained an average of 0.056 ppm fenpyroximate/Fen-OH and < 0.020 ppm of M-3. The absence, or low level, of M-3 in medium dose fat was confirmed in one sample by LC/MS/MS analysis. Fat from animals receiving a low dose of fenpyroximate contained 0.015 ppm fenpyroximate/Fen-OH.

Liver from high dose animals contained an average of < 0.010 ppm fenpyroximate, N-desmethyl-M-3 and M-22. M-3 residues averaged 0.80 ppm. Liver from animals receiving a medium dose of fenpyroximate contained 0.37 ppm M-3; other metabolite residues were undetectable. Low dose liver contained 0.19 ppm M-3 and undetectable levels of other metabolites.

Kidney from high dose animals also contained a significant residue of M-3 (0.40 ppm), a lesser amount of fenpyroximate (0.014 ppm) and undetectable residues of N-desmethyl-M-3 and M-22. Kidney from medium dose animals contained 0.29 ppm M-3, < 0.01 ppm fenpyroximate, and undetectable levels of N-desmethyl-M-3 and M-22. Low dose kidney samples contained 0.20 ppm M-3; other residues were not detected.

Recovery of M-21 was low in some milk samples, as was M-3 in muscle, fenpyroximate/Fen-OH and M-3 in fat and N-desmethyl-M-3 in kidney. However, all residues were corrected for recovery %. The storage stability studies indicated that analytes were stable in milk and tissues when stored frozen, except for M-21 in milk (66% relative recovery) and Fen-OH in fat (60% relative recovery).

During the in-life phase of the study, the cows appeared normal and healthy. Residues of fenpyroximate and metabolites found in the respective samples of milk and tissues are summarised in Table 235.

Table 234 Residue Levels (Corrected for Recovery) in High Dose (10 ppm in the Diet) Milk Samples from the Fenpyroximate Dairy Cow Feeding Study

Collection Interval	Animal Code	PTRL West No.	Fenpyroximate/Fen-OH[a] (mg/kg)		M-21[b] (mg/kg)	
			Residue Level	Mean	Residue Level	Mean
Control	1179-1A	744W-004,5	< 0.005		< 0.005	
Day 0	1179-4A 1179-4B 1179-4C	744W-006 744W-007 744W-008	< 0.005 ND [c] < 0.005	< 0.005	< 0.005 < 0.005 ND	< 0.005
Control	1179-1A	744W-009,10,26	ND		ND	
Day 1	1179-4A 1179-4B 1179-4C	744W-011 744W-012 744W-013	0.017 0.014 0.021	0.017	ND ND ND	ND
Control	1179-1A	744W-016,26	ND		ND	
Day 3	1179-4A 1179-4B 1179-4C	744W-018 744W-019 744W-020	0.022 0.015 0.014	0.017	ND ND ND	ND
Control	1179-1A	744W-021,-014,-015	ND		ND	
Day 7	1179-4A 1179-4B 1179-4C	744W-023 744W-024 744W-025	0.007 0.019 0.015	0.014	< 0.005 ND ND	< 0.005
Control	1179-1A	744W-026,28	ND		ND	
Day 11	1179-4A 1179-4B 1179-4C	744W-030 744W-031 744W-032	0.010 0.010 0.017	0.012	ND ND ND	ND
Control	1179-1A	744W-033,34	ND[3]		< 0.005	
Day 14	1179-4A 1179-4B 1179-4C	744W-035 744W-036 744W-037	0.012 0.012 0.014	0.013	ND ND ND	ND
Control	1179-1A	744W-040,41	ND		ND	

Collection Interval	Animal Code	PTRL West No.	Fenpyroximate/Fen-OH[a] (mg/kg)		M-21[b] (mg/kg)	
			Residue Level	Mean	Residue Level	Mean
Day 18	1179-4A 1179-4B 1179-4C	744W-042 744W-043 744W-044	0.007 0.013 0.014	0.011	< 0.005 ND ND	< 0.005
Control	1179-1A	744W-045,46	ND		< 0.005	
Day 21	1179-4A 1179-4B 1179-4C	744W-047 744W-048 744W-049	0.013 0.019 0.015	0.016	< 0.005 < 0.005 ND	< 0.005
Control	1179-1A	744W-151	ND		< 0.005	
Day 24	1179-4A 1179-4B 1179-4C	744W-096 744W-097 744W-098	0.007 0.006 0.008	0.007	< 0.005 < 0.005 < 0.005	< 0.005
Control	1179-1A	744W-079	ND		< 0.005	
Day 28	1179-4A 1179-4B 1179-4C	744W-102 744W-103 744W-104	0.015 0.008 0.007	0.010	< 0.005 < 0.005 < 0.005	< 0.005

[a] Fenpyroximate and Fen-OH converted to M-3 and detected as methyl M-3; expressed as fenpyroximate mg/kg corrected for recoveries of fortifications <100%.

[b] M-21 detected as methyl M-21; expressed as M-21 mg/kg corrected for recoveries of fortifications <100%.

[c] ND=Not detected, no peak

< 0.005 ppm is below limit of quantification

Table 235 Mean and range of recovery corrected residue levels found in milk and tissues of cows fed fenpyroximate at dose levels of 0.003, 0.10 and 0.35 mg/kg bw over 29 consecutive days

Analyte	Mean and (range) residue level (ppm)				
	Milk	Liver	Kidney	Muscle	Fat
High dose (10 ppm)					
Fenpyroximate/Fen-OH	0.013* (0.006-0.022) [101.5%]	< 0.010 (< 0.010-0.011) [87%]	0.014 (0.009-0.019) [77%]	0.038 (0.024-0.049) [76.1%]	0.105 (0.046-0.159) [53.5%]
M-21	< 0.005 (ND-< 0.005) [73.7%]	NA	NA	NA	NA
M-3	NA	0.80 (0.70-0.90) [103%]	0.40 (0.35-0.44) [90%]	< 0.010 (ND-< 0.010) [56.3%]	< 0.010 (ND-< 0.010) [55.2%]
N-desmethyl-M-3	NA	< 0.010 (ND-< 0.010) [83%]	ND	NA	NA
M-22	NA	< 0.010 (ND-0.010) [94%]	ND	NA	NA
Medium dose (3 ppm)					
Fenpyroximate/Fen-OH	0.005 (< 0.005-0.011) [101.5%]	ND	< 0.01 (ND-< 0.01) [77%]	0.015 (0.012-0.017) [76.1%]	0.056 (0.025-0.073) [53.5%]
M-21	< 0.005 (ND-< 0.005) [73.7%]	NA	NA	NA	NA
M-3	NA	0.37 (0.28-0.42) [103%]	0.29 (0.23-0.35) [90%]	ND	< 0.010 (ND-< 0.01) [55.2%]
N-desmethyl-M-3	NA	ND	ND	NA	NA
M-22	NA	ND	ND	NA	NA
Low dose (1 ppm)					
Fenpyroximate/Fen-OH	-	ND	ND	< 0.010 (all < 0.010) [76.1%]	0.015 (0.010-0.018) [53.5%]
M-21	-	NA	NA	NA	NA

Analyte	Mean and (range) residue level (ppm)				
	Milk	Liver	Kidney	Muscle	Fat
M-3	NA	0.19 (0.16-0.22) [103%]	0.20 (0.18-0.23) [90%]	-	-
N-desmethyl-M-3	NA	ND	ND	NA	NA
M-22	NA	ND	ND	NA	NA

NA–Not applicable;

ND–Not detected ;

- Not analysed ;

* - Does not include day 0

Recovery % is in [ ].

Table 236 Fortification Recoveries of fenpyroximate and M-21obtained from Milk Analyses

Fenpyroximate Concurrent Fort in Milk Recovery				
Sample	Dose Level	Fortification Standard	Fort Level (μg/g)	Recovery %
Day 0 Milk	High	Fenpyroximate	0.05 0.05	109.1 120.1
Day 1 Milk	High	Fenpyroximate	0.05 0.05	85.4 143.4
Day 3 Milk	High Medium	Fenpyroximate Fenpyroximate	0.05 0.05 0.05 0.05	117.5 109.9 87.0 97.3
Day 7 Milk	High	Fenpyroximate	0.05 0.05	124.4 108.3
Day 11 Milk	High	Fenpyroximate	0.05 0.05	80.7 117.5
Day 14 Milk	High Medium	Fenpyroximate Fenpyroximate	0.05 0.05 0.05 0.05	100.1 143.8 80.5 65.5
Day 18 Milk	High	Fenpyroximate	0.05 0.05	116.7 113.7
Day 21 Milk	High Medium	Fenpyroximate Fenpyroximate	0.05 0.05 0.05 0.05	99.9 106.1 83.4 74.6
Day 24 Milk	High	R-UL-1/Fen-OH	0.05 0.05	97.5 83.6
Day 28 Milk	High	Fenpyroximate	0.05 0.05	68.9 104.0
			Mean R.S.D.	101.4 (20.3%)
M21 Concurrent Fort in Milk Recovery				
Sample	Dose Level	Fortification Standard	Fort Level (μg/g)	Recovery %
Day 0 Milk	High	M-21	0.05 0.05	85.8 83.4
Day 1 Milk	High	M-21	0.05 0.05	52.6 40.9
Day 3 Milk	High Medium	M-21 M-21	0.05 0.05 0.05 0.05	52.6 40.9 89.8 95.2
Day 7 Milk	High	M-21	0.05 0.05 0.05 0.05	48.0 48.4 44.1 48.2
Day 11 Milk	High	M-21	0.05 0.05	52.6 40.9
Day 14 Milk	High	M-21	0.05 0.05	37.7 49.6

Fenpyroximate Concurrent Fort in Milk Recovery				
Sample	Dose Level	Fortification Standard	Fort Level (µg/g)	Recovery %
	Medium	M-21	0.05 0.05	111.8 116.9
Day 18 Milk	High	M-21	0.05 0.05	55.0 75.8
Day 21 Milk	High Medium	M-21 M-21	0.05 0.05 0.05 0.05	55.8 48.5 126.4 100.8
Day 24 Milk	High	M-21	0.05 0.05	141.0 170.4
Day 28 Milk	High	M-21	0.05 0.05	76.3 73.6
			Mean R.S.D.	73.7 (46.9%)

Table 237 Fortification Recoveries of fenpyroximate/Fen-OH and M-3 obtained from Muscle Analyses

Fenpyroximate/Fen-OH Concurrent Fortification Recovery in Muscle				
Sample	Dose Level	Fortification Standard	Fort Level (µg/g)	Recovery %
Muscle (Set 1)	High	Fenpyroximate	0.05 0.05	69.0 81.3
	Mid	Fenpyroximate	0.05 0.05	70.0 73.4
	Low	Fen- OH	0.05 0.05	75.8 87.3
			Mean R.S.D.	76.1 (9.2%)
M-3 Concurrent Fortification Recovery in Muscle				
Sample	Dose Level	Fortification Standard	Fort Level (µg/g)	Recovery %
Muscle (Set 1) (Set 2)	High High	M-3 M-3	0.05 0.05 0.05 0.05 0.01 0.01	60.1 57.9 89.3 52.1 41.3 55.1
(Set 1) (Set 2)	Mid Mid	M-3 M-3	0.05 0.05 0.05 0.05 0.05	46.9 49.0 54.8 57.3 55.1
			Mean R.S.D.	56.3 (21.7%)

Table 238 Fortification Recoveries of fenpyroximate/Fen-OH and M-3 obtained from Fat Analyses

Fenpyroximate/Fen-OH Concurrent Fortification Recovery in Fat				
Sample	Dose Level	Fortification Standard	Fort Level (µg/g)	Recovery %
Fat	High	Fenpyroximate	0.05 0.05 0.05 0.05	(Set 1) 45.7 45.8 (Set 2) 49.2 63.4
	Mid	Fen- OH	0.05 0.05	33.9 47.1
	Low	Fen- OH	0.05 0.05	81.8 61.3
			Mean R.S.D.	53.5 (24.1%)

Fenpyroximate/Fen-OH Concurrent Fortification Recovery in Fat				
Sample	Dose Level	Fortification Standard	Fort Level (μg/g)	Recovery %
M-3 Concurrent Fortification Recovery in Fat				
Sample	Dose Level	Fortification Standard	Fort Level (μg/g)	Recovery %
Fat	High	M-3	0.05 0.05 0.05 0.05 0.01 0.01 0.05 0.05	(Set 1) 31.9 51.4 (Set 2) 34.9 70.1 (Set 3) 66.7 82.1 (Set 3) 69.7 47.4
	Mid	M-3	0.05 0.05	47.0 50.8
			Mean R.S.D.	55.2 (29.6%)

Table 239 Fortification Recoveries of fenpyroximate and other metabolites obtained from Liver Analyses

Fortification Recoveries Obtained from Liver Analyses				
Sample Set	% Recoveries of Analyte Fortified			
	N-Desmethyl M-3	M-3	M-22	Fenpyroximate
High Dose A	65	103	83	92
High Dose B	62	111	97	82
High Dose C	87	113	112	103
Mid Dose A	88	94	89	84
Mid Dose B	119	85	76	56
Mid Dose C	103	97	86	83
Low Dose A	71	104	99	95
Low Dose B	66	108	101	94
Low Dose C	84	114	102	97
Mean	83	103	94	87
R.S.D.	(23.1%)	(9.4%)	(11.9%)	(15.7%)

Table 240 Fortification Recoveries of fenpyroximate and other metabolites obtained from Kidney Analyses

Fortification Recoveries Obtained from Kidney Analyses				
Sample Set	% Recoveries of Analyte Fortified			
	N-Desmethyl M-3	M-3	M-22	Fenpyroximate
High Dose A	33	66	70	87
High Dose B	48	89	74	69
High Dose C	32	92	78	74
Mid Dose A	86	73	80	64
Mid Dose B	73	84	85	70
Mid Dose C	105	83	87	75
Low Dose A	-*	-*	-*	-*
Low Dose B	64	119	94	90
Low Dose C	99	114	102	87
Mean	68	90	84	77
R.S.D.	(42.0%)	(20.5%)	(12.6%)	(12.6%)

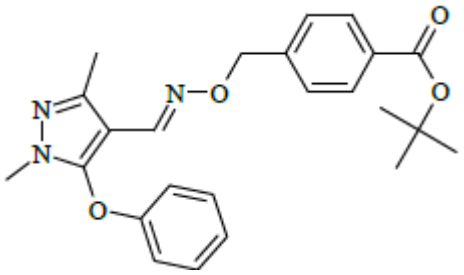
* The low dose fortification sample was spilled.

APPRAISAL

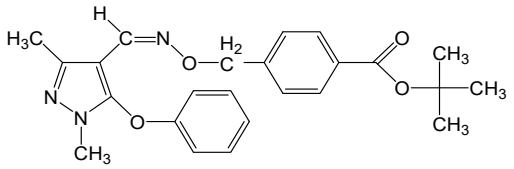
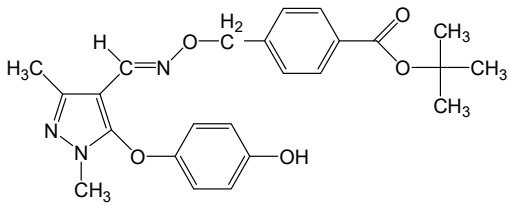
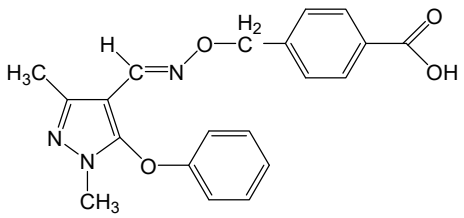
Fenpyroximate is a pyrazole non-systemic selective acaricide/insecticide for the control of mites and hoppers in a wide range of crops including fruits and vegetables. It was first evaluated by JMPR in 1995 and then in 1999 and 2010 for maximum residue levels, and in 2004 and 2007 for toxicology.

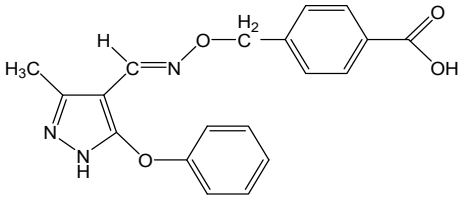
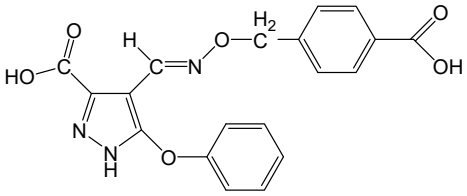
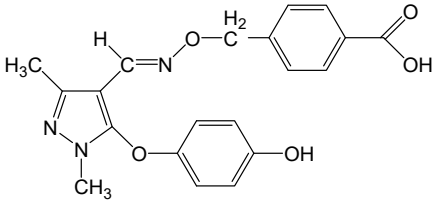
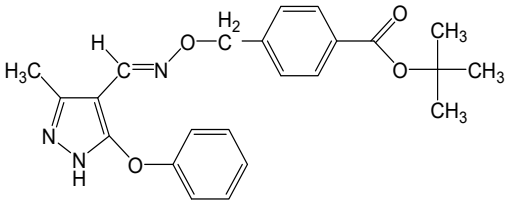
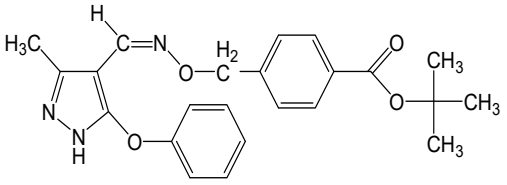
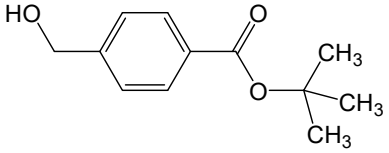
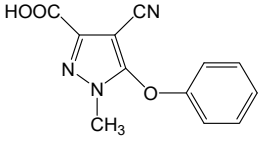
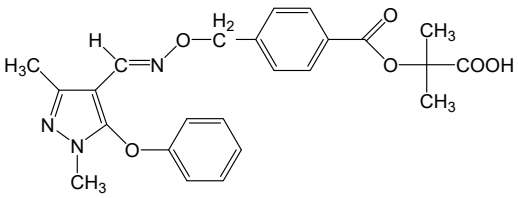
Fenpyroximate was scheduled at the 48th Session of the CCPR for Periodic Re-evaluation for residues and toxicology by 2017 JMPR. The meeting received information from manufacturer on the metabolism of fenpyroximate in citrus, apple, grape, snap beans, cotton, Swiss chard and lactating goat, as well as rotational crop studies, environmental fate in soil and water, method of residue analysis, stability in stored analytical samples, use patterns, supervised residue trials, fate of residue during storage and processing, and livestock feeding studies.

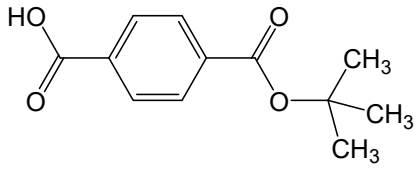
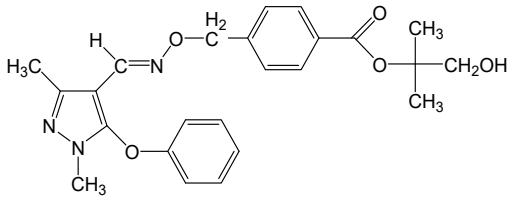
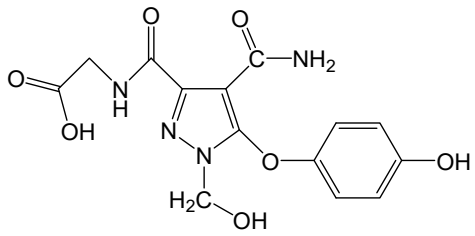
The IUPAC name of fenpyroximate is tert-butyl (E)-alpha-(1,3-dimethyl-5-phenoxypyrazol-4-ylmethyleneamino-oxy)-*p* toluate.



The following abbreviations are used for the metabolites discussed in the appraisal:

Code Number (Synonyms)	Description	Structure
M-1 (*Z*-isomer)	IUPAC: tert-butyl (*Z*)-α-(1,3-dimethyl-5-phenoxypyrazol-4-ylmethyleneamino-oxy)-*p*-toluate CAS: 1,1-dimethylethyl (*Z*)-4-[[[[(1,3-dimethyl-5-phenoxy-1*H*-pyrazol-4-yl)methylene]amino]oxy]methyl]benzoate	
M-2	Tert-butyl (*E*)-4-{[1,3-dimethyl-5-(4-hydroxyphenoxy)pyrazol-4-yl]-methyleneaminooxymethyl}benzoate	
M-3	(*E*)-4-[(1,3-dimethyl-5-phenoxypyrazol-4-yl)methyleneaminooxymethyl]benzoic acid	

Code Number (Synonyms)	Description	Structure
N-desmethyl M-3	(*E*)-4-[(3-methyl-5-phenoxypyrazol-4-yl)methyleneaminooxymethyl]benzoic acid	
N-desmethyl M-3 acid	(*E*)-4-[(3-carboxy-5-phenoxypyrazol-4-yl)methyleneaminooxymethyl]benzoic acid	
M-5	(*E*)-4-{[(1,3-dimethyl-5-(4-hydroxyphenoxy)pyrazol-4-yl]methyleneaminooxymethyl}benzoic acid	
M-12	Tert-butyl (*E*)--4-[(3-methyl-5-phenoxypyrazol-4-yl)methyleneaminooxymethyl]benzoate	
M-12 isomer	Tert-butyl (*Z*)-4-[(3-methyl-5-phenoxypyrazol-4-yl)methyleneaminooxymethyl]benzoate	
M-15	Tert-butyl 4-hydroxymethylbenzoate	
M-21	4-cyano-1-methyl-5-phenoxypyrazole-3-carboxylic acid	
M-22	(*E*)-2-[4-[(1,3-dimethyl-5-phenoxypyrazol-4-yl)methyleneaminooxymethyl]benzoyloxy]-2-methylpropanoic acid	

Code Number (Synonyms)	Description	Structure
MTBT	Mono-(tert-butyl) terephthalate	
Fen-OH	2-hydroxymethyl-2-propyl (*E*)-4-[(1,3-dimethyl-5-phenoxypyrazol-4-yl)-methylenaminooxymethyl]benzoate	
Metabolite 2	2-(5-(4-hydroxyphenoxy)-1-(hydroxymethyl)-1H-pyrazole-4-carbamoyl-3-carbonylamino)acetic acid	

Studies on the metabolism in plants, livestock and environmental fate utilised either [pyrazole-3-^{14}C]-fenpyroximate or [benzyl-(U)-^{14}C]-fenpyroximate.

Plant metabolism

The meeting received plant metabolism studies with fenpyroximate following foliar application (representative use patterns) to citrus, apples, grapes (Fruit crop group), snap beans and cotton (Pulses and Oilseeds crop group) and Swiss chard (Leafy crop group).

Citrus

In a study, outdoor mandarin tangerine trees were treated with [pyrazole-^{14}C] fenpyroximate at a rate of 22.4 ± 1.5 mg/tree or 33.5 ± 0.5 mg /tree. The residues in pulp were less than 0.03 mg/kg (LOD) at 0–28 DAT or less than 0.01 mg/kg at maturity (137 DAT), therefore no characterization or identification was conducted. 67–89% radioactive residues in leaves and more than 92% in rind were extracted with acetone/methanol. Fenpyroximate (33% TRR, 0.12 mg eq/kg), M-12 (17% TRR, 0.06 mg eq/kg) and M-1 (6% TRR, 0.02 mg eq/kg) were identified as major residues in rind at harvest.

Fenpyroximate (18% TRR, 0.24 mg eq/kg), M-1 (9% TRR, 0.13 mg eq/kg) and M-12 (6% TRR, 0.08 mg eq/kg) were the major components of radioactive residues in leaves.

The polar metabolites were enzyme hydrolysed with β-glucosidase or cellulose, and M-20 glucoside and several other glucosides were found in leaves and the fruit rind.

In the second study, outdoor mandarin trees were treated with [benzyl-^{14}C]-fenpyroximate at a rate of 21 mg/tree. Citrus leaves and fruits were sampled from two trees at 0, 3, 7, 14, 28 and 98 DAT. The radioactive residues in pulps were all less than 0.01 mg/kg, and no further characterizations or identifications were conducted. The radioactive residues in rinds were 0.21 mg eq/kg at maturity

(98 DAT) and 81–99% radioactive residues in rind were extracted with acetone/methanol (1:1). The residue of fenpyroximate in the rind was 1.1 mg eq/kg (99% TRR) just after application and 0.09 mg eq/kg (43% TRR) at maturity (98 DAT). The major metabolites in rind were M-1 and M-12. M-1 in rind were less than 4% TRR (0.01–0.04 mg eq/kg) in all samples. M-12 level in rind was 11–12% TRR (0.10–0.12 mg eq/kg) at 7–28 DAT, and 19% TRR (0.04 mg eq/kg) at maturity.

The radioactive residues in leaves were 4.2, 2.5 and 0.86 mg eq/ kg at 14, 28 and 98 DAT (maturity). 72–100% of radioactive residues in leaves were extracted with acetone/methanol (1:1). The residues of fenpyroximate in the leaves were 9.8 mg eq/kg (100% TRR) just after application, 1.1 mg eq/kg (43% TRR) at 28 DAT and 0.21 mg eq/kg (24% TRR) at maturity (98 DAT). The major metabolites in leaves were M-1 and M-12. M-1 level in leaves was 20–32% TRR (0.49–2.03 mg eq/kg) from 7 to 28 DAT, and was 16% TRR (0.14 mg eq/kg) at 98 DAT. M-12 level in leaves was less than 5% TRR (0.09–0.21 mg eq/kg) at 3–28 DAT, and 8% TRR (0.07 mg eq/kg) at maturity. Two new metabolites, M-15 in leaves was at maximum of 0.04 mg eq/kg (< 1% TRR) at 7 DAT, and M-17 in leaves was at maximum of 0.16 mg eq/kg (4% TRR) at 14 DAT.

The polar metabolites were enzyme hydrolysed with β-glucosidase or cellulose, M-15 glucoside and several unknown glucosides were detected at a level of less than 0.01 mg eq/kg in the leaves and rind at 98 DAT.

In the third study, outdoor Dancy tangerine trees were treated with [pyrazole-^{14}C] fenpyroximate at the rates of 21 mg/tree (harvest at day 0–28） or 32 mg/tree (harvest at day 65). No radioactive residues were detected in pulps from trees treated with 21 mg/tree at 0–28 DAT (LOD was 0.13 mg eq/kg). Radioactive residue levels in pulps were 0.022 mg eq/kg at harvest (65 DAT).

The radioactive residue levels were 0.96–1.4 mg eq/kg in rind from trees treated with 21 mg/tree. Radioactive residue levels in rind were 1.0 mg eq/kg at the time of harvest (65 DAT). 90% of radioactivity from rind were extracted with acetone/methanol. The residues of fenpyroximate, M-1, M-12 and M-3 in rind were 0.60 mg eq/kg (58% TRR), 0.13 mg eq/kg (13% TRR), 0.04 mg eq/kg (4% TRR) and 0.04 mg eq/kg (4%TRR) at harvest (65 DAT). M-6 was detected at 0.02 mg eq/kg (2%TRR) and other metabolites (M-3, M-8, M-9, M-11, M-13 and M-19) were < 0.02 mg eq/kg (<2% TRR).

The radioactive residue levels were 11–14 mg eq/kg in leaves from trees treated with 21 mg/tree (0–28 DAT). Radioactive residue levels in leaves were 14 mg eq/kg from trees treated with 32 mg eq/tree (65 DAT). 71–96% of radioactivity from leaves were extracted with acetone/methanol. Residues of fenpyroximate in leaves were 3.6–12 mg eq/kg from 0–28 DAT. The residues of fenpyroximate, M-1 and M-12 in leaves at harvest (65 DAT) were 4.4 mg eq/kg (31% TRR), 1.7 mg eq/kg (12% TRR) and 0.41 mg eq/kg (3% TRR). Polar Radioactive residues accounted for 1.93 mg eq/kg (14% TRR) and other metabolites were < 0.02 mg eq/kg (< 2% TRR).

Apple

The meeting received information on 2 metabolism studies of fenpyroximate in/on apple.

In the studies, apple trees were treated once by foliar spraying with [pyrazole-^{14}C] or [benzyl-^{14}C]-fenpyroximate at rate of 7.5 g ai/100 L. The residues in apple fruits were 0.12–0.13 mg eq/kg at day 0 and 0.032–0.036 mg eq/kg at harvest. More than 97% radioactive residues in fruit was extracted with acetone/water (1:1). The major components of radioactive residues in fruits were fenpyroximate and M-1. The residues of fenpyroximate and M-1 in fruit were 0.015–0.017 mg eq/kg (47% TRR) and 0.005–0.007 mg eq/kg (18–19% TRR) at harvest. The other metabolites were all below 0.01 mg eq/kg in fruits.

Radioactive residues in leaves amounted to 10–12 mg eq/kg at day 0 and decreased to 0.51–0.63 mg eq/kg at harvest (57 DAT). More than 94% radioactive residues in leaves was extracted with acetone/water (1:1). The major components of radioactive residues in leaves were fenpyroximate and M-1. The residues of fenpyroximate and M-1 in leaves were 1.3–1.6 mg eq/kg (53–59% TRR) and 0.50–0.61 mg eq/kg (19–25% TRR) at 28 DAT, and decreased to 0.22 mg eq/kg (35–43% TRR) and

0.091–0.16 mg eq/kg (18–26% TRR) at harvest. The other metabolites were all below 0.01 mg eq/kg in leaves.

Grape

The meeting received information on 2 metabolism studies of Fenpyroximate in/on grape.

In the studies, grapevines was once treated by hand spraying with [pyrazole-^{14}C] or [benzyl-^{14}C]-fenpyroximate at rate of 7.5 g ai/100L.The radioactive residues in grape juice were lower than 0.01mg eq/kg over the whole sampling period, and no further characterization or identification was conducted. The radioactive residues in grape berries were 0.086–0.097 mg eq/kg at day 0 and 0.060–0.081 mg eq/kg at harvest. More than 93% of radioactive residues in berries were extracted with acetone/methanol (1:1). The major components of radioactivity in berries were fenpyroximate and M-1. The residues of fenpyroximate and M-1 were 0.027–0.031 mg eq/kg (38–45% TRR) and 0.004–0.006 mg eq/kg (5–10% TRR) in grape berry at harvest. Other metabolites identified in grape berry were less than 0.01 mg eq/kg.

The radioactive residues in leaves amounted to 6.2–7.5 mg eq/kg at day 0 and decreased to 0.97–1.2 mg eq/kg at harvest (57 DAT). 83–99% of radioactive residues in leaves was extracted with acetone/methanol (1:1). The major components of radioactivity were fenpyroximate and M-1. The residues of fenpyroximate and M-1 were 0.33–0.64 mg eq/kg (34–56% TRR) and 0.052–0.054 mg eq/kg (5% TRR) in leaves at harvest (57 DAT). Minor metabolites identified were M-9 (0.01 mg eq/kg), M-12 (0.01 mg eq/kg) and M-19 (0.02 mg eq/kg) in leaves at harvest.

Snap bean

The snap bean plants (*Phaseolus vulgaris*, field grown) were treated with [Pyrazole-^{14}C] - fenpyroximate at rate of 104 g/ha or [Benzyl-^{14}C]-fenpyroximate at rate of 105 g/ha. Snap beans were harvested 7 days after application. The total radioactive residues in beans were 0.11 to 0.12 mg eq/kg. More than 99% of the radioactive residues was extracted with acetonitrile: water. The primary components of the residues were fenpyroximate and M-1. The residues of fenpyroximate accounted for 86 to 89% TRR (0.095–0.106 mg eq/kg). The residues of M-1 accounted 4.0 to 4.7% TRR (0.005 mg eq/kg). Other minor components were less than 10% TRR or 0.01 mg eq/kg.

Swiss chard

Swiss Chard plants were treated once with [pyrazole-^{14}C] or [benzyl-^{14}C]-fenpyroximate at rate of 102 g ai/ha. Samples of stem, leaves from 0, 14 and 35 DAT and root at 35 DAT were collected. Radioactive residues in stems and leaves at 0–35 DAT were up to 6.5 mg eq/kg for [pyrazole-^{14}C] fenpyroximate and up to 7.7 mg eq/kg for [benzyl ring -^{14}C] fenpyroximate. The majority of the radioactivity (96 to 100% of TRR) was extracted with acetonitrile/distilled water (4/1, v/v) and acetonitrile/ 0.1N hydrochloric acid (4/1, v/v). The most prominent residue in stems and leaves was fenpyroximate (0.34–7.6 mg eq/kg, 37–99% TRR). High polar radioactivity (retained at TLC origin) accounted for 31 to 49% TRR (0.43–0.68 mg eq/kg), which consisted of a number of individual metabolites with each individually below 8.5% TRR. . M-1 was less than 3.8% TRR (0.04 mg eq/kg).

Cotton

The cotton plants were treated once with [pyrazole-^{14}C] fenpyroximate as a foliar spray at a rate of 194 g ai/ha. The cotton forage of immature plants was harvested 1 DAT. Mature plants were harvested after boll opening (30 DAT) and were separated into seed, lint and gin trash. The major parts of the treated radioactivity were recovered from rinses and extracts (68–126% of TRR) in the forage, seed kernel, lint/hull, leaves and gin trash. The radioactive residues in post-extraction solid (PES) was < 10% of TRR except in seed kernels (28% TRR) and lint/hulls (15% TRR). However, the levels of radioactivity in PES of seed kernel and lint/hull were very low (0.002–0.003 mg eq/kg). The radioactive residues were 9.2 mg eq/kg in gin trash, 3.6 mg eq/kg in forage, 0.021 mg eq/kg in lint/hull, and 0.008 mg eq/kg in seed kernel.

Fenpyroximate and M-1 (approximately equal amounts) were the major components in leaves, the immature cotton (forage), cotton seed, and the cotton gin trash. . The residues of fenpyroximate and M-1 were 2.5 mg eq/kg (70% of TRR) and 1.2 mg eq/kg (32% of TRR), respectively, in immature cotton (forage), and for both fenpyroximate and M1, 0.003 mg eq/kg (38% TRR) in cottonseed (kernel), and 3.4 mg eq/kg (37% of TRR) in Gin trash. A number of other metabolites detected were less than 10% TRR or 0.01 mg/kg.

In summary, following foliar spray application of fenpyroximate, radioactive residues mainly remained on plant surfaces, and the parent fenpyroximate and M-1 were the major components of residues. All metabolites above 0.01 mg/kg in plants were also found in rats. Primary metabolic pathways of fenpyroximate in plants included conversion of fenpyroximate to Z-isomer (M-1) and hydrolysis of ester, oxime ether cleavage, N-demethylation, oxidation or conjugation to polar metabolites

Confined rotational crop studies

The meeting received information on two confined rotational crop studies.

In the first study, radish, lettuce and wheat were sown as rotational crops at intervals of 30, 120 and 365 days following treatment of sandy loam soil with [pyrazole-^{14}C] fenpyroximate at a rate of 224 g ai/ha. Lettuce and radish were harvested at maturity. Wheat was sampled at the forage and hay growth stages, and at maturity for collection of grain, straw and chaff. Residues in lettuce were ≤0.002 mg eq/kg. Residues in radish root and leaves ranged from 0.001 to 0.008 mg eq/kg. Residues wheat forage and hay ranged from 0.005 to 0.047 mg eq/kg. Residues in straw and chaff taken at maturity were 0.018 to 0.11 mg eq/kg and residues in grain were 0.004 to 0.01 mg eq/kg.

All individual metabolite residues were < 0.01 mg eq/kg. The residues of fenpyroximate in radish root was 0.001 mg eq/kg, and other two metabolites were less than 0.002 mg eq/kg. The residues of three metabolites in radish foliage were 0.001–0.002 mg eq/kg.

Wheat forage from the 120 day plant back contained M-5, M-3 and 4 other metabolites (0.001–0.005 mg eq/kg). All metabolites in hay extracts from all plant back groups were 0.001–0.007 mg eq/kg. M-21 in the 30 day plant back hay, and M-8, M-5 and M-3 in hay extracts from 120 and 365 day plant back were identified. Residues of metabolites in wheat chaff were 0.001–0.006 mg eq/kg.

The extracted residues in wheat straw from the 30, 120 and 365 day plant back intervals were 0.052, 0.081 and 0.025 mg eq/kg. Straw from the 30 day plant back contained a polar unknown (0.022 mg eq/kg, 32% TRR), M-8 (0.008 mg eq/kg, 12% TRR), and 5 other metabolites. Straw from the 120 day plant back contained M-8 (0.011 mg eq/kg, 14% TRR) and other 3 metabolites above 0.01 mg eq/kg. These included a polar unknown and 2 mid polarity metabolites.

In another study, radish, spinach, and wheat were sown as rotational crops at intervals of 30, 120 and 270 days following treatment of sandy loam soil with [benzyl-^{14}C]-fenpyroximate at a rate of 111 g ai/ha. The plants were harvested at immaturity and maturity. Total radioactive residues in all crop matrices were very low. Wheat hay, straw and grain from Day 31, and wheat straw and grain from Day 118 had TRR equal to or more than 0.01 mg/kg. TRRs in all crops at 270 days were below 0.01 mg/kg.

Extraction of radioactivity accounted for between 69–87% TRR (0.010–0.033 mg/kg) for Day 31 (grain, hay and straw) and 24–39% TRR (0.002–0.005 mg/kg) for Day 118 grain and straw. All individual metabolites in wheat straw at the 31 day plant-back interval were less than 0.001 mg eq/kg.

In summary, fenpyroximate related residues in soil are unlikely to result in significant levels in rotational crops following application at maximum seasonal rates of up to 224 g ai/ha for non-permanent crops.

Animal metabolism

Lactating goats

Two studies on lactating goat metabolism were available for the meeting. In the first study, a lactating goat was orally administered [pyrazole-^{14}C]-fenpyroximate by capsule twice daily for 3 consecutive days at a dose of 10 ppm in the diet, corresponding to 0.5 mg/kg bodyweight/day. Milk samples were collected twice daily during the dosing period. The treated goat was sacrificed approximately 22 hours after the final dose.

The majority of radioactive residues was recovered in the excreta (urine 32% AD, faeces 33% AD) and in the gastrointestinal tract (11% AD). For tissues, radioactive residues were highest in liver (1.2 mg eq/kg), followed by kidney (1.1 mg eq/kg), muscle (0.021 mg eq/kg) and fat (0.082 mg eq/kg). TRR in milk reached 0.037 mg eq/kg before the end of dosing.

More than 67% of the TRR in milk and tissues was extracted with solvent. The residues of fenpyroximate in milk were 0.001–0.008 mg eq/kg. The major metabolites in milk were M-21 (0.011–0.015 mg eq/kg, 37–55% TRR) and Fen-OH (0.001–0.003 mg eq/kg, 3–12% TRR). The residues of fenpyroximate were highest in fat (0.035 mg/kg, 42% TRR), followed by muscle (0.006 mg eq/kg, 25% TRR) and kidney (0.005 mg eq/kg, 1% TRR). N-desmethyl M-3 (0.27–0.31 mg eq/kg, 21–28% TRR) and M-3 (0.46–0.61 mg eq/kg, 42–50% TRR) were the major metabolites detected in kidney and liver, Fen-OH (0.014–0.029 mg eq/kg, 35–58% TRR) was the major metabolite detected in fat and muscle, while other metabolites were less than 10% TRR in tissues. No M-1 was detected in tissues and only a very low level in milk (0.001 mg eq/kg, 3% TRR).

In another study, a lactating goat was orally administered by capsule with [benzyl -^{14}C]-fenpyroximate for 3 consecutive days at a rate of 10 ppm in diet, corresponding to 0.3 mg/kg bodyweight/day. Milk samples were collected twice daily during the dosing period. The treated goat was sacrificed approximately 22 hours after the final dose.

The majority of the radioactive residues were recovered in the excreta (urine 12% AD, faeces 45% AD) and in the gastrointestinal tract (22% AD, unexcreted). For tissues, radioactive residues were highest in kidney (2.1 mg eq/kg), followed by liver (1.3 mg eq/kg) with fat (0.14 mg eq/kg) and muscle (0.027 mg eq/kg). TRR in milk reached the plateau of 0.031 mg eq/kg at 24–32 hours after first dosing.

More than 85% of the TRR in milk and tissues was extracted with solvent. The residues of fenpyroximate in milk were 0.003–0.008 mg eq/kg (13–26% TRR). The major metabolites in milk were M-22 (0.003–0.007 mg eq/kg, 16–27% TRR). Fen-OH was detected only in milk (0.002 mg eq/kg, 8% TRR) at 23–32 hour and M-21 (0.005 mg eq/kg, 38% TRR) was only detected in milk at 8–24 hours. The residues of fenpyroximate was highest in fat (0.049 mg eq/kg, 36% TRR), followed by kidney (0.022 mg eq/kg, 1% TRR) and muscle (0.002 mg eq/kg, 7% TRR). M-5 Glucuronide (0.25–0.55 mg eq/kg, 20–26% TRR), Fen-OH (0.741–0.98 mg eq/kg, 47–59% TRR) were the major metabolites in kidney and liver, M-22 (0.020 mg eq/kg, 74% TRR), Fen-OH (0.009 mg eq/kg, 33% TRR) were major metabolites in muscle. Fen-OH (0.019 mg eq/kg, 14% TRR) and M-22 (0.024 mg eq/kg, 17% TRR) were detected in fat. M-1 was detected in kidney (0.140 mg eq/kg, 5% TRR) and liver (0.073 mg eq/kg, 6% TRR). Other metabolites detected were less than 10% TRR in tissues.

Environmental fate

Aerobic metabolism in soil

The Meeting received information on soil aerobic metabolism, photolysis and aqueous hydrolysis properties of [^{14}C]-fenpyroximate.

The degradation of fenpyroximate in soil incubated under dark aerobic conditions was moderate to slow with DT_{50} values of 23–254 days, indicating moderate persistence in soil. The parent fenpyroximate was the predominant radioactive residue in soil from day 0 to day 219. The major

degradation products formed in soil treated with [benzyl (U)-^{14}C]-fenpyroximate were M-3 with maximum value of 29% AR, while other minor degradation products including M-1 and M-15 were less than 5% AR. The major degradation products formed in soil treated with [pyrazole-3-^{14}C]-fenpyroximate were M-3 (maximum 17% AR), M-8 (maximum 16% AR), and M-11 (maximum 9.6% AR), with less than 5% AR of other minor degradation products including M-1, M-6 and M-13.

In summary, fenpyroximate degraded steadily to major metabolites M-3, M-8 and M-11, minor metabolites M-1 and M-6, bound residue and carbon dioxide.

Soil photolysis

The soil photolysis of fenpyroximate in wet soils was investigated. Irradiation enhanced the degradation of fenpyroximate on wet soils. The DT_{50} values for the irradiated soil samples (equivalent to natural sunlight at 30–50°N) were 43 and 35 days for [benzyl-^{14}C] and [pyrazole-^{14}C] fenpyroximate, respectively. While the DT_{50} values for the dark soil samples were 91 and 69 days for [benzyl-^{14}C] and [pyrazole -^{14}C] fenpyroximate, respectively. Major degradation products observed in irradiated soil samples were M-1 (maximum 17% AR), M-12 (maximum 5.5% AR), M-12 isomer (maximum 5.1% AR) and MTBT (maximum 8.3% AR). M-3 (maximum 14% AR) was the only degradation product observed in the dark soil sample.

Hydrolysis

Fenpyroximate degradation was very slow at pH 5, 7, and 9 at 25 °C. The half-life for the hydrolytic degradation of [pyrazole-^{14}C]-fenpyroximate was calculated to be 180 days for pH 5, 226 days for pH 7 and 221 days for pH 9, indicating hydrolysis plays a negligible role in its degradation under environmental conditions. M-1 (maximum 7% AR) was only observed at pH 5 and pH 9. The principal hydrolysis product observed at each pH was M-3 (maximum 10% AR).

Methods of analysis

The Meeting received description and validation data for analytical methods for residues of fenpyroximate, M-1, M-3 and de-methyl fenpyroximate in plant matrices, livestock matrices and soil. The recoveries were within 70–120% with RSD less than 20%, the validated methods were considered suitable for data generation.

For most data gathering methods, residues are extracted with acetonitrile, acetone or methanol. Extracted residues undergo clean-up by silica gel/alumina column, GPC or dispersive SPE with primary/secondary amine (PSA). The residue separation and analysis is by GC-FTD, GC-N-FID, LC-UV, GC-MS, LC-MS, or LC-MS/MS, using internal standards or matrix matched standards. For most matrices, the limits of quantification (LOQ) for each analyte is 0.01 mg/kg.

For livestock matrices, the residues are extracted with acetone: water (2:1, v/v), acetonitrile, or acetonitrile: water (8:2. v/v), cleaned up by SPE or GPC, and analysed with GC or LC-MS. The LOQs for fenpyroximate are 0.01 mg/kg in milk and tissues (liver, kidney, muscle and fat).

Multi-residue methods (QuEChERS) for enforcement are also validated for fenpyroximate and M-1 in plant matrices, fenpyroximate and M-3 in livestock matrices.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the stability of fenpyroximate 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.

Residues of fenpyroximate and M-1 were stable in lettuce at least 9 months, in apple at least 18 months, in orange at least 9 months, in hops at least 24 months, in sunflower at least 9 months and in potato at least 9 months. Residues of fenpyroximate were stable in grape for at least 12 months. Residues of M-1 were stable in grape for at least 36 months

The stability of fenpyroximate in animal commodities was studied in the lactating goat residue transfer study with fortified samples stored for the same intervals as in studies. The residues of fenpyroximate and M-1 were stable in muscle at least 56 days, kidney at least 53 days, fat at least 54 days, milk at least 79 days and liver at least 53 days.

Definition of the residue

Following application of fenpyroximate to crops, the parent compound and its Z isomer (M-1) were the major residues from the day of application until harvest (7–137 days after treatment). With the exception of M-12 in citrus rind (19% TRR, 0.04 mg eq/kg), other metabolites were less than 10% TRR or less than 0.01 mg eq/kg. Residues of fenpyroximate were consistently two to ten times greater than M-1 across all crops in the metabolism studies, and residues of M-1 were frequently below the LOQ in crop field trials. Therefore, the Meeting decided that fenpyroximate is a suitable marker for enforcement in plants.

In deciding if 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 those compounds. M-1 is found in metabolism studies and crop field trials at levels that are not negligible relative to parent compound in some samples and is considered to have equal toxicity to parent fenpyroximate. Therefore, M-1 should be included for risk assessment. The only other candidate compound is M-3, which formed under conditions simulating pasteurization, brewing/baking/boiling, and sterilisation. M-3 comprised from 41% (pasteurisation) to 89% (sterilisation) of the applied radioactivity in that study. M-3 was observed in the rat metabolism study and its toxicity is considered to be covered by that of fenpyroximate. Processing studies that included these conditions were conducted with exaggerated residues of fenpyroximate (relative to field trial residues) in RAC commodities of apple, bean, grape, strawberry, maize, and tea. Residues of M-3 were < 0.01 mg/kg in all processed commodities except canned beans (up to 0.07 mg/kg) and strawberry jam (0.02 mg/kg). These levels of M-3 were less than 15% of the residues of parent compound. Therefore, the Meeting decided that M-3 did not contribute significantly to the total exposure of fenpyroximate through the diet. The residue definition for assessing dietary exposure from plant commodities is the sum of fenpyroximate and M-1, expressed as fenpyroximate.

In goats, the residue profile varied by matrix. The Fen-OH metabolite was consistently observed as a major residue across all matrices (12% TRR milk, 58% TRR muscle, 35% TRR fat, 59% TRR liver, and 47% TRR kidney). Additional residues observed across multiple matrices at greater than 10% TRR were fenpyroximate (26% TRR milk, 43% TRR fat), M-5 glucuronide (20% TRR liver, 26% TRR kidney), M-3 (50% TRR liver, 42% TRR kidney), M-21 (26–55% TRR in milk, and M-22 (74% TRR muscle, 17% TRR fat). A QuEChERS method was validated for analysis of fenpyroximate and M-3 in milk, fat, muscle, and offal. Based on structural similarities between fenpyroximate, M-3 and Fen-OH, the Meeting noted that the method is likely to be suitable for analysis of Fen-OH. Given the toxicity of Fen-OH is considered to be covered by that of fenpyroximate, the Meeting decided that the sum of fenpyroximate, Fen-OH and M-3, expressed as fenpyroximate is a suitable marker for compliance in livestock commodities.

In addition to the residues for compliance, dietary exposure from consumption of livestock commodities may occur for M-5-glucuronide for liver (20% TRR, 0.25 mg eq/kg) and kidney (26% TRR, 0.55 mg eq/kg), for M-21 in milk (37% TRR, 0.011 mg eq/kg), and M-22 in muscle (74% TRR, 0.02 mg eq/kg). The toxicity of M-5- glucuronide, M-21 and M-22 are covered by parent fenpyroximate. Since M-21 and M-22 were not detected in the feeding study, the Meeting decided that they do not need to be included for assessing dietary exposure. The Meeting decided that definition for dietary assessment is the sum of fenpyroximate, Fen-OH, M-3, and M-5 (free and conjugated), expressed as fenpyroximate, in livestock commodities.

The Log Pow of fenpyroximate is above 5. The ratio of residues of fenpyroximate in fat and muscle in metabolism studies is 5.8 to 24.5, and the total residues in fat is 3.4–5.1 times higher than residues in muscle. On the weight of evidence, the Meeting decided the residue is fat soluble.

The residue definition of fenpyroximate for plant commodities for compliance with the MRL is fenpyroximate, and for dietary assessment is the sum of fenpyroximate and M-1 (Z isomer).

Residue definition of fenpyroximate for animal commodities for compliance with MRL is sum of fenpyroximate, Fen-OH and M-3, expressed as fenpyroximate, for dietary assessment is sum of fenpyroximate, Fen-OH, M-3, and M-5 (free and conjugated), expressed as fenpyroximate.

The residues of fenpyroximate, sum of fenpyroximate, Fen-OH and M-3 are fat soluble.

Results of supervised trials on crops

Supervised residue trial data were available for fenpyroximate on citrus (oranges, mandarin, lemons, grapefruit, natsudaidai, tangor), pome fruit (apples, pears), stone fruit (cherries, peaches, apricot, plums), berries and other small fruits (grape, raspberries, strawberries), assorted tropical and subtropical fruits-inedible peel (avocado, papaya), cucurbits (cucumber, melon, courgette, watermelon, cantaloupe), fruit vegetables other than cucurbits (tomatoes, pepper), legume vegetables (beans), root and tuber vegetables (potatoes), cereal grain (maize), tree nuts (almond, pecan, walnut), hops, coffee and tea.

Citrus fruits

The critical GAP for citrus in the USA is two foliar applications at 235 g ai/ha with a PHI of 14 days. The maximum rate per growing season is 471 g ai/ha. The Meeting received supervised residue trial data for fenpyroximate on citrus fruit from the USA, EU and Japan.

In trials approximating critical GAP in the USA, residues in citrus fruit were:

Lemons: residues of fenpyroximate (n=4) 0.017, 0.15, 0.17 and 0.18 mg/kg, residues of fenpyroximate and M-1 (n=4) 0.16, 0.18, 0.19 and 0.2 mg/kg.

Grapefruit: residues of fenpyroximate (n=4) < 0.01, 0.017, 0.017 and 0.073 mg/kg, residues of fenpyroximate and M-1 (n=4) 0.025, 0.047, 0.077 and 0.081 mg/kg

Oranges: residues of fenpyroximate (n=8) 0.011, 0.018, 0.039, 0.066, 0.13, 0.1332, 0.15 and 0.26 mg/kg, the residues of fenpyroximate and M-1 (n=8) 0.074, 0.089, 0.14, 0.15, 0.16, 0.18, 0.19 and 0.27 mg/kg.

The Meeting noted that median residues following two foliar applications to lemon, grapefruit and orange are within a 5-fold range and a Kruskal-Wallis H-test suggest the residues in lemon, grapefruit and orange are from similar populations. The Meeting decided to combine the data to estimate a maximum residue level for the citrus group. The combined residue data of fenpyroximate (n=16) are: < 0.01, 0.011, 0.017(3), 0.018, 0.039, 0.066, 0.073, 0.13, 0.13, 0.15, 0.15, 0.17, 0.18 and 0.26 mg/kg. The combined residue data of fenpyroximate and M-1 (n=16) 0.025, 0.047, 0.074, 0.077, 0.081, 0.089, 0.17, 0.15, 0.16, 0.16, 0.18, 0.18, 0.19, 0.19, 0.20 and 0.27 mg/kg.

In trials conducted on tangor, satsuma, Chin citron natsudaidai and orange with approximate application rate, the residues in flesh was 13% of that of fruit. The Meeting recommended a maximum residue level of 0.6 mg/kg, and STMR of 0.020 mg/kg and HR of 0.0364 (highest individual) mg/kg respectively for citrus group, and replace the previous recommendation of 0.5 mg/kg for citrus fruits.

Pome fruits

Apple

The critical GAP for apple in Greece is one foliar application at 106 g ai/ha with a PHI of 7 days.

In trials conducted in EU member states approximating critical GAP, the residues of fenpyroximate in apples were: (n=10) 0.04, 0.06, 0.07(2), 0.08, 0.11(2), 0.12(2) and 0.14 mg/kg, the residues of fenpyroximate and M-1 in apples were: (n=10) 0.05, 0.07, 0.08(2), 0.09, 0.12(2), 0.13, 0.14 and 0.19 mg/kg.

The meeting noted that the cGAP gave rise to residues that exceeded the ARfD (110% for children in Canada) and therefore a public health concern could not be excluded. For this reason, the alternative GAP from Belgium was considered. The GAP in Belgium is one foliar application at 76.5 g ai/ha with a PHI of 7 days. Ten trials conducted in the EU are available at over dosed rates compared to the GAP and where scaled using the proportionality principle.

Residues of fenpyroximate in apple in rank order (n=10) were: 0.01, 0.04, 0.06, 0.07, 0.08, 0.09, 0.10, 0.12, 0.14 and 0.19 mg/kg.

Residues of fenpyroximate in apple (scaled using factors ranging from 1.42–2.35) in rank order (n=10) were: 0.028, 0.039, 0.049, 0.055, 0.062, 0.068, 0.073, 0.077, 0.081 and 0.095 mg/kg.

Residues of fenpyroximate and M-1 in apples were: (n=10) 0.038, 0.049, 0.059, 0.065, 0.072, 0.078, 0.083, 0.087, 0.1 and 0.15 mg/kg.

The Meeting recommended maximum residue level, STMR and HR of 0.2, 0.075 and 0.15 mg/kg respectively for apples.

Pears

The critical GAP for pears in US (pome fruit) is one foliar application at 117 g ai/ha with a PHI of 14 days. In trials conducted in USA approximating critical GAP in USA, France and New Zealand, residues of fenpyroximate in pears were: (n=6) 0.04, < 0.05, 0.07, 0.086, 0.051 and 0.14 mg/kg. The residues of fenpyroximate an M-1 in pears were: (n=6): 0.05, 0.07, < 0.10, 0.101, 0.136 and 0.14 mg/kg.

In trials conducted in USA with nominal rate of 450 g ai/ha with a PHI of 14 days were considered, the residues of fenpyroximate in pears were: (n=6) 0.073, 0.13, 0.16, 0.18, 0.24 and 0.28 mg/kg. The residues of fenpyroximate and M-1 in pears were: (n=6) 0.12, 0.18, 0.22, 0.23, 0.30 and 0.35 mg/kg.

The scaled residues (117/450=0.26) of fenpyroximate were: (n=6) 0.019, 0.033, 0.043, 0.047, 0.063, 0.072 mg/kg. The scaled residues (0.26) of fenpyroximate and M-1 were: (n=6) 0.032, 0.048, 0.056, 0.060, 0.077 and 0.090 mg/kg

Taking the proportionality approach into consideration, the Meeting agreed to make estimation based on combined data. The Meeting recommended maximum residue level of 0.2 mg/kg, STMR of 0.078 mg/kg and HR of 0.14 mg/kg for pears.

The Meeting withdrew the previous recommendations of 0.3 mg/kg for pome fruits.

Stone fruits

The critical GAP in USA for stone fruits is two applications at 117 g ai/ha with a PHI of 7 days.

Cherry

In trials conducted in the USA approximating critical GAP, the residues of fenpyroximate in cherries were: (n=8) 0.26, 0.34, 0.36, 0.46, 0.66, 0.79, 0.93 and 0.99 mg/kg. The residues of fenpyroximate and M-1 were: 0.31, 0.39, 0.41, 0.51, 0.66, 0.79, 0.93 and 0.99 mg/kg. The Meeting recommended maximum residue level, STMR and HR of 2, 0.585 and 0.99 mg/kg respectively for Cherry. The Meeting noted that the GAP in USA is for group of stone fruits, and decided to extrapolate to subgroup of cherries.

Short term dietary exposure assessment showed that residues in cherry exceed the acute reference dose (ARfD) of 0.01 mg/kg bw, at 110% of ARfD for children (Denmark, Germany). No alternative GAP with sufficient data for cherry was available.

Peach

In trials conducted in the USA approximating critical GAP, the residues of fenpyroximate in peaches were (n=10): 0.073, 0.075, 0.086, 0.098, 0.11, 0.13, 0.13, 0.15, 0.18 and 0.20 mg/kg. The residues of

fenpyroximate and M-1 were: 0.12, 0.12, 0.14, 0.15, 0.16, 0.18, 0.18, 0.20, 0.23, 0.25 mg/kg. The Meeting recommended maximum residue level, STMR and HR of 0.4, 0.17 and 0.25 mg/kg respectively for peaches.

Short term dietary exposure assessment showed that residues in peach exceed the acute reference dose (ARfD) of 0.01 mg/kg bw, at 130% of ARfD for children (Japan, Canada). No alternative GAP with sufficient data for peach was available.

However, as there was no exceedance of the ARfD for apricot, therefore, the Meeting estimated the maximum residue level, STMR and HR of 0.4, 0.17 and 0.25 mg/kg respectively for apricot.

Plum

In trials conducted in the USA approximating critical GAP, residues of fenpyroximate in plums were (n=6): < 0.05(2), 0.08 0.13, 0.20 and 0.27 mg/kg. The residues of fenpyroximate and M-1 were: < 0.1(2), 0.13, 0.18, 0.25 and 0.32 mg/kg. The Meeting estimated the maximum residue level, STMR and HR of 0.8, 0.155 and 0.33 mg/kg respectively for plums. The Meeting noted that the GAP in USA is for group of stone fruits, and decided to extrapolate to peaches.

Short term dietary exposure assessment showed that residues in plum dried exceed the acute reference dose (ARfD) of 0.01 mg/kg bw, at 270% of ARfD for children (Australia). No alternative GAP with sufficient data for plum was available.

The Meeting withdrew the previous recommendations of 0.4 mg/kg for stone fruits.

Berries and other small fruits

Raspberry

The critical GAP in the USA on raspberries is two applications at 117 g ai/ha with a PHI of 1 days. No trials were provided matching the critical GAP.

The registered use of fenpyroximate for raspberry in Austria allows one application at rate of 76.5 g ai/ha with a PHI of 14 days. In trials conducted in matching GAP, the residues of fenpyroximate in raspberry were: 0.01 0.04, 0.08 and 0.10 mg/kg. The residues of fenpyroximate and M-1 were: 0.02, 0.05, 0.09 and 0.11 mg/kg. The Meeting estimated a maximum residue level, STMR and HR of 0.2, 0.07 and 0.11 mg/kg respectively for raspberry.

Grape

The critical GAP in USA for grape is two applications at 117 g ai/ha with a PHI of 14 days.

In trials conducted in the Germany (two applications at rate of 135 g ai/ha with PHI of 14 days) approximating critical GAP, the residues of fenpyroximate in grapes were (n=2): 0.11, 0.40 mg/kg. The residues of fenpyroximate and M-1 were: (n=2) 0.12 and 0.41 mg/kg.

The registered use of fenpyroximate for vine in Spain allows one application at rate of 50 g ai/ha with a PHI of 28 days. In trials conducted in EU matching the GAP, the residues of fenpyroximate in grape were: (n=12) < 0.01(2), 0.01(2), 0.02(2), 0.03, 0.04(3), and 0.05(2) mg/kg. The residues of fenpyroximate and M-1 were: (n=12) < 0.02(2), 0.02(2), 0.03(2), 0.04(3) 0.05 and 0.06(2) mg/kg.

The Meeting recommended maximum residue level of 0.1 mg/kg, STMR of 0.035 mg/kg and HR of 0.06 mg/kg for grapes.

Strawberries

The Critical GAP in the USA on low growing berries including strawberries (USA subgroup 13-07G) is two applications at a rate of 117 g ai/ha with a PHI of 1 days.

In trials conducted in USA approximating critical GAP, the residues of fenpyroximate in strawberries were: (n=8) 0.06, 0.07, 0.19(2), 0.24, 0.24, 0.28 and 0.53 mg/kg. The residues of fenpyroximate and M-1 were: (n=8) 0.06, 0.08, 0.20, 0.22, 0.24, 0.27, 0.33 and 0.56 mg/kg.

The meeting noted that the cGAP gave rise to residues that lead to an exceedance of the ARfD (130% for children in France) and therefore a public health concern could not be excluded. The Meeting considered the alternative GAP for strawberries in Germany and Austria. The GAP in Germany and Austria is one foliar application at 102 g ai/ha with a PHI of 7 days. Sixteen trials support the GAP.

Residues of fenpyroximate in strawberries in rank order (n=16) were: < 0.01 (2), 0.01 (2), 0.02, 0.03, 0.05 (3), 0.06, 0.07 (2), 0.08, 0.10, 0.13, 0.19 mg/kg

Residues of fenpyroximate and M1 in strawberries in rank order (n=16) were: 0.02 (4), 0.03, 0.04, 0.06 (3), 0.07, 0.08 (2), 0.09, 0.11, 0.14, 0.20 mg/kg.

The Meeting recommended maximum residue level of 0.3 mg/kg, STMR of 0.06 mg/kg and HR of 0.2 mg/kg for strawberries.

Assorted tropical and sub-tropical fruit–inedible peel

Avocado

The Critical GAP in the USA on avocado is two applications at a rate of 117 g ai/ha with a PHI of 1 days.

In trials conducted in USA approximating critical GAP, the residues in avocado were: (n=5) < 0.05, < 0.05, < 0.05, 0.06 and 0.10 mg /kg. The Meeting noted that the avocado is minor crop, and agreed to estimate a maximum residue level, STMR and HR of 0.2, 0.05 and 0.1 mg/kg respectively.

Papaya

The critical GAP in Brazil is three applications at rates of up to 40 g ai/ha with a PHI of 3 days.

The Meeting was was unable to estimate a maximum residue level as no trials were provided matching the critical GAP.

Cucurbit vegetable

Cucumber

The critical GAP in the USA on cucumber is two applications at a rate of 117 g ai/ha with a PHI of 1 days. In trials conducted in USA matching the cGAP, residues of fenpyroximate in cucumber were: (n=7) < 0.05, 0.08 (2), 0.063, 0.065, 0.11 and 0.17 mg/kg. The residues of fenpyroximate and M-1 were: < 0.10, 0.113, 0.115, 0.13(2), 0.16 and 0.22 mg/kg. The Meeting estimated the maximum residue level, STMR and HR of 0.3, 0.13 and 0.24 (highest individual) mg/kg for cucumber, to replace previous recommendation of 0.3 mg/kg.

Summer Squash

The registered uses of fenpyroximate for zucchini in Germany allows one application at rate of 46–92 g ai/ha with PHI of 3 days, In trials conducted in France and Italy matching the GAP, residues of fenpyroximate in courgettes were: (n=6) < 0.01, 0.01(2), 0.02(2) and 0.03 mg/kg. The residues of fenpyroximate and M-1 were: < 0.02, 0.02(2), 0.03(2) and 0.04 mg/kg. The Meeting estimated the maximum residue level, STMR and HR of 0.06, 0.025 and 0.04 mg/kg for summer squash.

Melons, except Watermelon

The GAP in the USA on cantaloupe (included in USA crop subgroup 9A) is two applications at rate of 117 g ai/ha with a PHI of 3 days.

In trials conducted in USA matching the GAP, the residues of fenpyroximate, fenpyroximate and M-1 in cantaloupe were: (n=8) < 0.05 (8) mg ai/ha.

In trials conducted in Spain and Italy (one application of 110 g ai/ha, 3 days PHI), the residues of fenpyroximate in melons were : (n=4) < 0.01, 0.01, 0.02 and 0.08 mg/kg, residues of fenpyroximate and M-1 were: < 0.02, 0.02, 0.03 and 0.09 mg/kg.

The Meeting noted the application in trials on cantaloupe was 12–12 days, and decided to combine the data to estimate a maximum residue level, STMR and HR of 0.2, 0.05 and 0.09 mg/kg respectively for melon except watermelon, replaced the previous recommendation of 0.05 mg/kg).

Watermelon

The GAP in the USA on watermelon (included in USA crop subgroup 9A) is two applications at rate of 117 g ai/ha with a PHI of 3 days. In trials conducted in USA (2 application at rate of 110 g ai/ha, PHI of 1day), the residues of fenpyroximate in watermelon fruit were: (n=4) < 0.05 (4). The residues of fenpyroximate and M-1 were < 0.1 (4).

The Meeting agreed to estimate the maximum residue level, STMR and HR of 0.05, 0.1 and 0.1 mg/kg.

Short term dietary exposure assessment showed that residues in watermelon exceed the acute reference dose (ARfD) of 0.01 mg/kg bw, at 190% of ARfD for children (Canada). No alternative GAP with sufficient data for watermelon was available.

Fruiting vegetables other than Cucurbit

Peppers

The critical GAP in USA on pepper (included in US crop group 8–10) is two applications at rate of 117 g ai/ha with a PHI of 1 day.

In trials conducted in USA matching the cGAP, residues of fenpyroximate, fenpyroximate and M-1 in peppers were: (n=16) < 0.05(9), 0.05, 0.054, 0.07(2), 0.012 and 0.13 mg/kg. The Meeting estimated the maximum residue level, STMR and HR of 0.2, 0.05 and 0.13 mg/kg for bell and non-bell pepper. The Meeting noted that the GAP in USA is for US crop group 8–10, The Meeting agreed to extrapolate to subgroup of peppers except martynia, okra and roselle.

The Meeting withdraw the previous recommendation for chill pepper, dry of 1 mg/kg.

Tomato

The Critical GAP in the USA on tomato (for USA crop group 8-10) is two applications at rate of 117 g ai/ha with a PHI of 1 days.

In trials conducted in USA approximating critical GAP, the residues of fenpyroximate in tomato were: (n=19) < 0.05 (9), 0.05, 0.07, 0.08 (3), 0.09 (3), 0.11 and 0.12 mg/kg. The residues of fenpyroximate and M-1 were: < 0.1(9), 0.1, 0.12, 0.13(3), 0.14(3), 0.16 and 0.17 mg/kg. As residues in cherry tomato is normally higher than that in tomato, the Meeting estimated a maximum residue level, STMR and HR of 0.3, 0.10 and 0.17 mg/kg respectively for cherry tomato and tomato. The Meeting noted that the GAP in USA is for US crop group 8-10, and agreed to extrapolate to subgroup of eggplants.

Short term dietary exposure assessment showed that residues in tomato dried exceed the acute reference dose (ARfD) of 0.01 mg/kg bw, at 310% of ARfD for general population (Australia). No alternative GAP with sufficient data for tomato was available.

The Meeting withdrew the previous recommendation for fruiting vegetables other than cucurbits of 0.2 mg/kg.

Beans with pods

The GAP in the USA on bean is two applications at rate of 117 g ai/ha with a PHI of 1 days. In trials conducted in USA matching the US GAP, residues of fenpyroximate in bean with pod were: (n=8) < 0.05(2), 0.09(3), 0.15, 0.18, 0.19 mg/kg. The residues of fenpyroximate and M-1 were: < 0.1(2), 0.14(3), 0.20, 0.24 and 0.24 mg/kg.

The GAP in the Spain on bean is one application at rate of 102 g ai/ha with a PHI of 7 days. In trials conducted in Greece, France and Italy approximating the Spain GAP, residues in bean with pod were: (n=16) 0.02, 0.03(4), 0.06(3), 0.07, 0.08(2), 0.1, 0.13, 0.14, 0.23 and 0.41 mg/kg. The residues of fenpyroximate and M-1 were: 0.03, 0.04(4), 0.07(3), 0.08, 0.09(2), 0.11, 0.14, 0.15, 0.24 and 0.42 mg/kg. The Meeting noted that the residues from trial approximating the Spain GAP were higher than residues from trials in US, and agree to estimate the maximum residue level, STMR and HR of 0.5, 0.075 and 0.42 mg/kg for bean with pod based on trials in Europe, and extrapolated the estimates to subgroup of beans with pods. Furthermore, the Meeting withdraws its previous recommendation of 0.4 mg/kg for common bean (pods and/or immature seeds).

Potato

GAP in US is two applications at rate of 117 g ai/ha with a PHI of 7 days. In trials conducted in USA matching the GAP, residues of fenpyroximate in potato tubers were: (n=16) < 0.05(16) mg/kg. The Meeting noted that residues of fenpyroximate or M-1 from trials at 5 times rate were < 0.05 mg/kg, and estimated the maximum residue level, STMR and HR of 0.05*, 0 and 0 mg/kg respectively for potatoes.

Maize

The GAP in the USA on maize is two applications at rate of 117 g ai/ha with a PHI of 14 days. In trials conducted in USA matching the US GAP, residues of fenpyroximate or M-1 in maize grain were: (n=10) < 0.01(10). The Meeting estimated the maximum residue level, STMR of 0.01*, 0.01 mg/kg respectively for maize.

Tree nuts

GAP in US on tree nut (US crop group 14) is two applications at rate of 117 g ai/ha with a PHI of 14 days. In trials on almond (5), pecan (5) and walnut (3) conducted in USA with exaggerated rate (one application of 450 g ai/ha 14 days PHI), residues of fenpyroximate or M-1 in tree nut meats were: (n=13) < 0.05(13) mg/kg. The Meeting estimated the maximum residue level, STMR and HR of 0.05*, 0 and 0 mg/kg respectively for tree nuts.

Coffee

GAP in Brazil on coffee is two applications at rate of 50–100 g ai/ha with a PHI of 15 days. In trials on coffee conducted in Brazil matching the GAP, residues in coffee beans were: (n=8) < 0.01(3), < 0.025(3), 0.03 and 0.04 mg/kg. The Meeting estimated the maximum residue level, STMR and HR of 0.07, 0.025 and 0.04 mg/kg respectively for coffee bean.

Hops

GAP in Europe (Austria) on hop is one application at rate of 76.8–268.8 g ai/ha with a PHI of 21 days. In trials conducted in Germany and Japan approximating the Austrian GAP, residues of fenpyroximate in dried hops were: (n=6) 1.2, 3.7, 4.3, 5.0, 7.4 and 8.2 mg/kg. The residues of fenpyroximate and M-1 were: 2.2, 4.7, 5.0, 5.3, 7.4 and 8.2 mg/kg. The Meeting estimated the maximum residue level, STMR of 15, 5.15 mg/kg respectively for hops, dry, and replaced the previous recommendation of 10 mg/kg.

Tea

GAP in India on tea is one application at rate of 25 g ai/ha with a PHI of 7 days. In trials conducted in India approximating Indian GAP, residues of fenpyroximate in tea leaves were (n=10): 0.3, 0.68, 0.93, 0.95, 1.2, 1.4, 1.8, 3.01, 3,7 and 3.9 mg/kg. The residues of fenpyroximate and M-1 were: 0.33, 0.71, 0.98(2), 1.3, 1.5, 1.8, 3.0, 3.8 and 4.1 mg/kg. The Meeting estimated the maximum residue level and STMR of 8 and 1.4 mg/kg, respectively, for tea, green, black, dry.

Animal feed items

Bean forage

The GAP in the USA on bean is two applications at rate of 117 g ai/ha with a PHI of 1 days. In trials conducted in USA matching the US GAP, residues of fenpyroximate and M-1 in forage of bean were: (n=8) < 0.1(2), 1.2, 2.6, 3.0, 3.1, 4.5 and 6.8 mg/kg. The Meeting estimated the median residues and high residues of 2.8 and 7.5 (highest individual) mg/kg for bean forage.

Maize forage and stover

The GAP in the USA on maize is two applications at rate of 117 g ai/ha with a PHI of 14 days.

In trials conducted in USA matching the US GAP, residues of fenpyroximate and M-1 in maize forage were: (n=9) 0.31, 0.32, 0.33, 0.37, 0.38, 0.47, 0.58, 0.81 and1.12 mg/kg. The residues of fenpyroximate in maize stover were: (n=10) 0.17, 1.0, 1.0, 1.1, 1.1, 1.5, 1.6, 1.6, 1.8 and 2.2 mg/kg. The residues of fenpyroximate and M-1 in maize stover were: (n=10) 0.30, 1.4, 1.4, 1.6, 1.9, 2.2, 2.5, 3.0, 3.3 and 3.9 mg/kg. The Meeting estimated the median residues and highest residue of 0.38 and 1.3 (highest individual) mg/kg for maize forage, maximum residue level of 5 mg/kg, median residue of 2.05 and highest residue of 4.1 mg/kg (highest individual) for maize fodder.

Maize silage

GAP in Europe on maize is one applications at rate of 50 g ai/ha with a PHI of 28 days. In trials conducted in Hungary matching the GAP, residues in maize silage grains were: (n=2) 0.24 and 0.90 mg/kg. Two trials are not sufficient for recommendation.

Almond hulls

GAP in US on tree nut (US crop group 14) is two applications at rate of 117 g ai/ha with a PHI of 14 days. In trials on almond conducted in USA with exaggerated rate (one application of 450 g ai/ha 14 days PHI), residues in almond hull were: (n=5) 0.76, 0.92 1.2, 1.3, 1.4 mg/kg. No trials conducted matching GAP.

Fate of residues during processing

The Meeting received information on the fate of incurred residues of fenpyroximate during the processing of beans, apples, tomatoes, grapes strawberries and orange.

Hydrolysis

The Meeting received information on the nature of the residues of fenpyroximate under simulated processing conditions (pasteurization: 20 minutes at 90 °C and pH 4, baking/brewing/boiling: 60 minutes at 100 °C and pH 5, sterilization: 20 minutes at 120 °C and pH 6), fenpyroximate was not hydrolytically stable under processing conditions representative of sterilisation, pasteurisation, and brewing, baking and boiling. M-3 (41–89%) was detected as predominant product of hydrolysis in all conditions, with M-6 (< 3%) as minor hydrolysis products. M-1(< 2%) was only detected in under pasteurisation condition.

The fate of fenpyroximate residues has been investigated in a number of studies simulating household or commercial processing of beans, apples, tomatoes, grapes, strawberries, potatoes,

orange, maize and tea. Residues of M-3 were < 0.01 mg/kg in all processed commodities except canned beans (up to 0.07 mg/kg) and strawberry jam (0.02 mg/kg).

Processing studies were conducted with exaggerated residues of fenpyroximate (relative to field trial residues) in RAC commodities of apple, bean, grape, strawberry, maize, and tea. Residues of M-3 were < 0.01 mg/kg in all processed commodities except canned beans (up to 0.07 mg/kg) and strawberry jam (0.02 mg/kg). These levels of M-3 were less than 15% of the residues of parent compound. Therefore, the Meeting decided that M-3 were not included in calculation for processing factor.

The residues of fenpyroximate increase in some dried or concentrated commodities (apple pomace, dry apple, grape raisin, aspirated grain fraction of maize, orange oil and dry orange)

Processing factors calculated from sum of fenpyroximate and M-1, and estimated STMR-P and HR-P

	Processed Fraction	Processing Factor	Best estimate PF	RAC STMR or STMR-P*	RAC HR or HR-P*
Beans	Fresh beans (RAC)			0.08	0.42
	Washed beans	1.2, 0.89	1.045	0.084	0.44
	Cooked beans	0.73, 0.47	0.6	0.048	0.25
	Canned beans	0.42, 0.46	0.44	0.035	0.18
Apples	Fresh apple (RAC)			0.075	0.15
	Washed fruits	0.71, 1.1	0.905	0.679	0.136
	Wet Pomace	2.2, 5.5	3.85	0.289	
	Dry Pomace	5.1, 12.0	8.55	0.641	
	Pasteurised juice	0.18, 0.14	0.16	0.012	
	Pasteurised sauce	0.18, 0.18	0.18	0.0135	
	Dried apples	3.8, 5.0	4.4	0.33	0.66
Tomatoes	Whole tomato (RAC)			0.1	0.17
	Washed tomatoes	0.92, 0.83	0.875	0.088	0.15
	Tomato juice	0.85, 0.42	0.635	0.064	
	Canned tomatoes	0.54, 0.25	0.395	0.04	0.067
	Puree	0.76, 0.67	0.715	0.072	
Grapes	Whole fruit (RAC)			0.035	0.06
	Wet pomace	4.7, 5.1	4.9	0.19	
	Wine	0.27, 0.04	0.155	0.005	
	Pasteurised juice	0.27, 0.04	0.155	0.005	
	Washed grapes	0.82, 0.60	0.71	0.025	0.043
	Raisins	2.9, 1.1	2	0.07	0.12
Strawberries	Whole fruit (RAC)			0.06	0.2
	Washed fruit	0.53, 0.58	0.555	0.0333	0.111
	Canned fruit	0.05, 0.19	0.12	0.0072	
	Jam	0.19, 0.38	0.285	0.0171	
Orange	Fresh fruits (RAC)			0.15	0.28
	Juice	< 0.019, < 0.044	0.032	0.0048	
	Molasses	0.093, 0.046	0.07	0.011	
	Oil	72.2, 13.3	43	6.5	
	Dried fruit	5.3, 5.0	5.2	0.78	1.5
Tea	Dry tea leave			1.5	4.1
	infusion	0.0069, 0.0068 0.013, 0.019 0.0069, 0.0070 0.0088	0.0098	0.015	0.04
Maize	Grains (RAC)			0.01	
	Grits	0.016	0.016	0.00016	
	Meal	0.15	0.15	0.0015	
	Flour	0.37	0.37	0.0037	
	Refined oil (dry milling)	0.99	0.99	0.0099	
	Refined oil (wet milling)	0.31	0.31	0.0031	
	Aspirated grain fraction	86	86	0.86	

*: STMR-P or HR-P was calculated by processing factor of fenpyroximate and M-1.

Processing factors calculated from fenpyroximate for estimation of MRL

	Processed Fraction	Processing Factor	Best estimate PF	MRL*
Apples	RAW Dried apples	 3.8, 4,9	 4.4	0.2 1
Grapes	RAW Raisins	 2.9, 1.04	 2.0	0.1 0.2
Orange	Fresh fruits (RAC) *Oil*	 74, 4,7	 39	0.6 25

*MRL for processed commodities was calculated by processing factor of fenpyroximate.

As residues in dried apple are higher than residues in apple fruit, the Meeting estimated a maximum residue level of 1 (0.2×4.4) mg/kg for dried apples.

As residues in orange oil are higher than residues in orange fruit, the Meeting estimated a maximum residue level of 25 (0.6×39) mg/kg for citrus oil.

As residues in grape raisin are higher than residues in grape berry, the Meeting estimated a maximum residue level of 0.2 (0.1×2) mg/kg for grape, dry, and replaced the previous recommendation of 0.3 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 fenpyroximate at the equivalent of 1.0, 3.0 and 10 ppm in the feed for 28 consecutive days.

Residues of fenpyroximate/Fen-OH and M-3 in milk from high dose group reached plateau of 0.017 mg/kg at 3 days after the first dose and were 0.010 mg/kg at day 28. Residues of fenpyroximate/Fen-OH and M-3 in milk from high dose group were 0.006–0.022 mg/kg, from low and median dose group were < 0.008 mg/kg for all samples. Other minor metabolites were < 0.005mg/kg.

Residues of fenpyroximate/Fen-OH and M-3 in liver from high dose group were 0.71–0.911 mg/kg, 0.28–0.42 mg/kg from median, 0.16–0.22 from low dose group. Other metabolites were less than 0.01 mg/kg from high dose group or undetected.

Residues of fenpyroximate/Fen-OH and M-3 in kidney from high dose group were 0.359–0.459 mg/kg, 0.24–0.36 mg/kg from median dose group, and 0.18–0.23 mg/kg from low dose group. Other metabolites were either less than 0.01 mg/kg or undetected.

Residues of fenpyroximate/Fen-OH and M-3 in muscle from high, median and low dose group were 0.025–0.059, 0.012–0.017 and < 0.01 mg/kg, with other metabolites (including M-3) less than 0.01mg/kg or undetected.

Residues of fenpyroximate/Fen-OH and M-3 in fat from high, median and low dose group were 0.046–0.169, 0.035–0.083 and 0.01–0.018 mg/kg, with no other metabolites above 0.01mg/kg.

A laying hens feeding study was not available.

Estimation of livestock dietary burdens

Potential cattle feed items include: bean forage, corn, corn forage and stover, apple pomace and dry citrus pulp. 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 2017 edition of the FAO Manual.

Summary of livestock dietary burden (ppm fenpyroximate equivalents of dry matter diet) (to be finished)

	US-Canada		EU		Australia(		Japan	
	Max	Mean	Max	mean	max	Mean	max	Mean
Beef cattle	0.885	0.524	3.49	1.625	14.8 (3.503)	5.812 (1.595)	0.009	0.009
Dairy cattle	1.956	0.905	6.89	2.81	16.5 (3.503)[AB]	6.36 (1.595)[CD]	1.63	0.48
Broilers	0.00852	0.00852	0.01	0.01			0.01	0.01
Layers	0.00852	0.00852	0.5(0.502)[E]	0.26(0.255)[F]			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

The Meeting noted that the calculated maximum animal burden (16.5 ppm) was from Australia (mainly from bean forage). As fenpyroximate is not registered for use in bean in Australia and Australia doesn't import any forage, the meeting decided to refine the animal burden calculation (to exclude bean forage). The refined calculation of Dietary burden for beef cattle and dairy cattle for Australia is shown in (parentheses).

Animal commodity maximum residue levels

The calculation used to estimate highest total residues for use in estimating maximum residue levels, STMR and HR values for cattle matrices is shown below.

	Feed level ppm) for milk residues	Residues (mg/kg) in milk	Feed level (ppm) for tissue residues	Residues (mg/kg)			
				Muscle	liver	Kidney	Fat
MRL (mg/kg), beef or dairy cattle							
Feeding study	3	0.005	3	0.017	0.42	0.36	0.083
	10	0.013	10	0.059	0.91	0.459	0.169
Dietary burden and high residue estimation	3.503	0.0056	3.503	0.020	0.455	0.367	0.089
STMR (mg/kg), beef or dairy cattle							
Feeding study	1	0	1	< 0.01	0.19	0.20	0.015
	3	0.005		3	0.015	0.30	0.066
Dietary burden and median residue estimated	1.595	0.0015	1.595	0.011	0.24	0.23	0.03

The Meeting estimated a maximum residue level of 0.01* mg/kg for fenpyroximate for milk, of 0.1 mg/kg for mammalian meat (fat), of 0.5 mg/kg for edible offal (mammalian) and 0.1 mg/kg for mammalian fats. The Meeting estimated an STMR of 0.0015 mg/kg for milk, of 0.011 mg/ kg for mammalian meat, 0.24 mg/kg for edible offal (mammalian) and 0.03 mg/kg for mammalian fat.

Since neither feeding study nor metabolism study on laying hens was available, the Meeting was unable to estimate maximum residue level for poultry commodities.

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: *fenpyroximate.*

Definition of the residue (for dietary risk assessment) for plant commodities: *sum of parent fenpyroximate and itert-butyl (Z)-α-(1,3-dimethyl-5-phenoxypyrazol-4-ylmethyleneamino-oxy)-p-toluate (its Z-isomer M-1), expressed as fenpyroximate.*

Definition of the residue (for compliance with the MRL) for animal commodities: *sum of fenpyroximate, 2-hydroxymethyl-2-propyl (E)-4-[(1,3-dimethyl-5-phenoxypyrazol-4-yl)-methylenaminooxymethyl]benzoate(Fen-OH), and (E)-4-[(1,3-dimethyl-5-phenoxypyrazol-4-yl)methyleneaminooxymethyl]benzoic acid(M-3), expressed as fenpyroximate*.

Definition of the residue (for dietary risk assessment) for animal commodities: *sum of fenpyroximate, 2-hydroxymethyl-2-propyl (E)-4-[(1,3-dimethyl-5-phenoxypyrazol-4-yl)-methylenaminooxymethyl]benzoate(Fen-OH), (E)-4-[(1,3-dimethyl-5-phenoxypyrazol-4-yl)methyleneaminooxymethyl]benzoic acid(M-3), and (E)-4-{[(1,3-dimethyl-5-(4-hydroxyphenoxy)pyrazol-4-yl]methyleneaminooxymethyl}benzoic acid (M-5, free and its conjugates), expressed as fenpyroximate.*

The residue is fat soluble.

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P (mg/kg)	HR, HR-P, highest residue (mg/kg)
CCN	Name	New	Previous		
FP 0226	Apple	0.2		0.075	0.15
FI 0326	Avocado	0.2	0.2	0.05	0.1
FP 0230	Pear	0.2		0.078	0.14
FS 0013	Subgroup of cherries	2	2	0.585	0.99
FS 0247	Peach	0.8		0.155	0.33
FS 0240	Apricot	0.4		0.17	0.25
FS 0014	Subgroup of plums (including fresh prunes	0.8		0.155	0.33
FC 0001	Group of citrus fruit	0.6	0.5	0.020	0.0364
FB 0269	Grapes	0.1	0.1	0.035	0.06
FB 0275	Strawberries	0.3	0.8	0.06	0.2
FB 0272	Raspberry	0.2		0.07	0.11
VC 0424	Cucumber	0.3	0.3	0.13	0.24
VC 0431	Squash, summer	0.06		0.025	0.04
VC 0046	Melons, except watermelon,	0.2	0.05	0.05	0.09
VC 0432	Watermelon	0.05		0.1	0.1
VO 0051	Subgroup of peppers, except martynia, okra and roselle	0.2		0.05	0.13
VO 2046	Subgroup of eggplants	0.3		0.1	0.17
VO 0448	Tomatoes	0.3		0.1	0.17
VO 2700	Cherry tomato,	0.3		0.1	0.17
	Subgroup of bean with pod (*Phaseolus spp.*)	0.5		0.075	0.42
VR 0589	Potato,	0.05*	0.05	0	0
GC 0645	Maize	0.01*		0.01	0.01
TN 0085	Tree nuts	0.05*	0.05*	0	0
SB 0716	Coffee beans	0.07		0.025	0.04
DH 1100	Hops, dry	15	10	5.15	
DT 1114	Tea, green, black, dried	8		1.4	
	Milk	0.01*	0.01*	0.0015	
	Mammalian meat, other than marine mamalian	0.1(fat)	0.2(fat)	0.011(muscle)	0.02
MO 0105	Edible offal, mammalian	0.5	0.02	0.24	0.455
MF 0100	Mammalian fat, except milk fats	0.1	0.01	0.03	0.089
DF 0226	Apple, dried	1		0.33	0.66
DF 5263	Grapes dried	0.2	0.3	0.07	0.12
	Citrus oil	25		6.5	
OC 0645	Maize fodder	5		2.05	4.1

Table of withdraw of previous MRL recommendations.

Commodity	Recommended MRL (mg/kg)	STMR or STMR-P	HR, HR-P, (mg/kg)

CCN	Name	New	Previous	(mg/kg)	
VO 0050	Fruiting vegetable other than cucurbits	w	0.2		
FP 0009	Pome fruits	w	0.3		
DF 0014	Prunes dry	w	0.7		
FS 0012	Stone fruits	w	0.4		
VP 0526	Common beans (pod and/or immature seeds	w	0.4		
HS 0444	Pepper Chilli, dry	w	1.0		

Table of additional STMR and HR values for use in dietary intake estimation.

Commodity		STMR or STMR-P (mg/kg)	HR, HR-P (mg/kg)
CCN	Name		
JF 0226	Apple juice	0.012	
	Apple sauce	0.0135	
JF 0269	Grape juice	0.005	
	Grape wine	0.005	
JF 0448	Tomato juice	0.064	
	Tomato canned	0.04	0.67
MW 0448	Tomato purée	0.072	
DM 0001	Citrus molasses	0.011	
JF 0001	Citrus juice	0.0048	
CF 0645	Maize meal	0.0015	
CF 1255	Maize flour	0.0037	
	Maize grits	0.00016	
OR 0645	Maize oil	0.0099	
	Tea infusion	0.015	

Table of median and highest residue values for use in livestock dietary burden estimation.

Commodity		Median residue (mg/kg)	Highest residue (mg/kg)
CCN	Name		
AB 0226	Apple pomace dry	0.641	
	Apple pomace wet	0.289	
	Aspirated grain fraction	0.86	
AB 0001	Citrus pulp, dry	0.78	1.5
AL 1030	Bean forage	2.8	7.5
AF 0645	Maize forage	0.38	1.3
AS 0645	Maize fodder	2.1	4.1

Desirable information

Information on feeding study and metabolism on laying hen is desired for estimation of maximum residue level for poultry commodities.

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The IEDI of fenpyroximate based on the STMRs estimated by this Meetings for the 17 GEMS/Food regional diets were 3–10% of the maximum ADI of 0.01 mg/kg bw. The Meeting concluded that the long-term dietary intake of residues of fenpyroximate is unlikely to present a public health concern.

Short-term dietary exposure

An ARfD for fenpyroximate is 0.01 mg/kg bw. The Meeting estimated the International Estimated Short-Term Intake (IESTI) of fenpyroximate for commodities for which STMR, HR and maximum residue levels were estimated by the current Meeting. The IESTI were less than 100% of a maximum ARfD for the commodities estimated, except for cherry (110% for children from Netherland and Denmark), peach (130% for children from Japan and Canada), watermelon (190% for children from

Canada), dried tomato (310% the for general population from Australia), and dried plums (270% for children from Australia). The Meeting concluded that the short-term intake of fenpyroximate residues from uses considered by the current Meeting may present a public health concern for these commodities.

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R-4060 R-4288	Iwamoto, T.	1994	Residue determination of fenpyroximate in or on grapes treated with fenpyroximate in or on grapes treated with fenpyroximate 5% SC at 50 ppm (ai)–Fukui -, Japan Plant Protection Association and The Institute of Toxicology, Japan, Not GLP, Unpublished
R-4067	Tillkes, M.	1993	Determination of the residues of fenpyroximate (Hoe 094552) and M-1 (Hoe 112573) in/on Grapes, Dr. Specht & Partner Chemische Laboratorien GMBH, GLP, Unpublished
R-4069	Raquet, H. *et al*	1992	Hoe 094552–water miscible suspension–50 g/kg (Code: Hoe 094552 00 SC05 A104): Investigation of residues in processed grapes following two applications of Hoe 094552, Hoechst Aktiengesellschaft, GLP, Unpublished
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R-4142	Sochor, K. & Burstell, H.	1993	Report on Plant Protection Residue Trial, Hoechst AG, Germany, Not GLP, Unpublished
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R-4194	Barney, W.P.	2007	Volume 2 Fenpyroximate: Magnitude of the Residue on Pepper (Bell & Non Bell, Field and Greenhouse), IR-4 Project Headquarters Rutgers, NJ, USA
R-4336	Ueyama, I.	2009	Residue of fenpyroximate: residue of fenpyroximate E-isomer on Bell Pepper harvested in Miyagi and Nagano Prefectures, The Institute of Environmental Toxicology, Ibaraki, Japan, Not GLP, Unpublished
R-4337	Ueyama, I.	2009	Residue of fenpyroximate: residue of fenpyroximate Z-isomer on Bell Pepper harvested in Miyagi and Nagano Prefectures, The Institute of Environmental Toxicology, Ibaraki, Japan, Not GLP, Unpublished
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R-4178	Oxspring, S.	2004	Final report on project AF/6094/NN To determine the magnitude of fenpyroximate residues at intervals and harvest in the raw agricultural commodity outdoor tomato resulting from one directed application of fenpyroximate 5 SC, in Spain and Greece, in 2001, Agrisearch UK, GLP, Unpublished
R-4179	Oxspring, S.	2005	Final report on project AF/6095 To determine the magnitude of fenpyroximate residues at intervals and harvest in the raw agricultural commodity protected tomatoe resulting from one directed application of fenpyroximate 5 SC, in the UK, Southern France and Italy, in 2001, Agrisearch UK Ltd, GLP, Unpublished
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R-4160	Gillis, N.	2003	Final Report on Project AF/6778/NN: To determine the magnitude of fenpyroximate residue at intervals and at harvest in the raw agricultural commodity bean with pods (protected) resulting from a single directed application of fenpyroximate 5SC, in Spain during 2002, Agrisearch UK, GLP, Unpublished
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