Fluazinam (303)

First draft prepared by Dr Julian Cudmore and Mrs Sonia Tessier, Chemicals Regulation Division of the Health and Safety Executive, UK

EXPLANATION

Fluazinam acts as a fungicide with activity against fungus from the class of *Oomycetes*, especially against *Phytophthora infestans*. It works protectively and needs to be applied before the disease attacks. At the Forty-eighth Session of the CCPR (2016), fluazinam was scheduled for evaluation as a new compound by the 2018 JMPR.

The Meeting received information on the identity, physical chemical properties, metabolism (plants, rotational crops and animals), environmental data, methods of analysis, freezer storage data, GAP information, supervised residue trials, fate of residues on processing and animal transfer studies.

IDENTITY

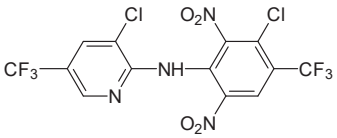
ISO Common Name	Fluazinam	
Synonyms	IFK-1216	
Chemical name	IUPAC:	3-chloro-*N*-(3-chloro-5-trifluoromethyl-2-pyridyl)-α,α,α-trifluoro-2,6-dinitro-*p*-toluidine
	CAS:	3-chloro-*N*-[3-chloro-2,6-dinitro-4-(trifluoromethyl)phenyl]-5-(trifluoromethyl)-2-pyridinamine
CIPAC No.	521	
CAS No	79622-59-6	
EEC No.	-	
Structural formula		
Molecular formula	$C_{13}H_4Cl_2F_6N_4O_4$	
Molecular mass	465.1	

Table 1 Summary of identification and characterisation of residues in grape berries and grape leaves dosed with ^{14}C-pyraclostrobin

	Grape berries		Grape leaves	
	Tolyl label	Chlorophenyl label	Tolyl label	Chlorophenyl label

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

Property	Results	Reference
Appearance	Yellow, odourless crystalline solid at 20 °C (Munsell colour 2.5GY 9/8)	Kimura, T. 1991a, Report No. 91 0508KT; Kimura, T. 1991b, Report No. 91 0509KT; Kimura, T. 1991c, Report No. 91 0510KT;
Vapour pressure	2.3×10^{-5} Pa at 25 °C, 1.3×10^{-4} Pa at 35 °C, 6.7×10^{-4} Pa at 45 °C	Yoder, S.J. 1992, Report No. 4039-91-0385-AS-001
Volatility	Henry's law constant at 20 °C 6.626×10^{-6} Atm/m^3/mole = 0.671 Pa/m^3/mole	McFadden, J.J. 2000, Report No. F-150-A
Boling point & melting point	Melting point = 117 °C (390 K)	van Helvoirt, J.A.M.W. 1993a, Report No. 089033

Property	Results	Reference
	The molten test substance is not stable above about 150 °C (423 K).	van Helvoirt, J.A.M.W. 1993b, Report No. 089044
Octanol/Water Partition Coefficient	Determined at 20 °C In water: Log P_{ow} = 4.53 At pH 4: Log P_{ow} = 4.99 At pH 7: Log P_{ow} = 4.82 At pH 9: Log P_{ow} = 4.05	Weissenfeld, M. 2008, Report No. B85397
Solubility in water	Determined at 20 °C at pH 5: 1.06 × 10^{-4} g/L at pH 7: 1.35 × 10^{-4} g/L at pH 9: 2.72 × 10^{-3} g/L (column elution method)	Brekelmans, M.J.C. 2002, Report No. 341189
Solubility in organic solvents	Determined at 20 °C n-Hexane: 6.7 g/L Methanol: 162 g/L Ethyl ether: 168 g/L Dichlorethane: 485 g/L Toluene: 512 g/L Ethyl acetate: 624 g/L Acetone: >645 g/L	Haga, T. 1991, Report No. 9112OHS-001
Specific gravity/density	Relative Density D^{20}_4: 1.81	van Rijsbergen, L.M. 2002a, Report No. 341123
Hydrolysis	Label I: U-^{14}C-phenyl Fluazinam Radiochemical purity: 100% Label II: 2,6-^{14}C-pyridyl Fluazinam Radiochemical purity: 97.7% ^{14}C-Fluazinam was found to be stable to hydrolysis in buffer solution at pH 4 after 5 d at 50 °C. At pH 7 and 9, ^{14}C-Fluazinam was hydrolytically unstable. DT_{50} = 4.5 d (label I) 2.7 d (label II) at pH 7 and 25 °C. DT_{50} = 3.5 d (label I) 3.9 d (label II) at pH 9 and 25 °C Fluazinam was hydrolysed to CAPA, which was then steadily degraded to DCPA (stable to hydrolysis). At pH 7, CAPA (M1) represented more than 95% of the applied radioactivity (Labels I and II) at the end of the incubation at 25 °C (day 29). At 50 °C, it reached a maximum of about 99% (Label I) and 98% (Label II) after 1 or 5 days of incubation at 50 °C, respectively, then was readily hydrolysed to DCPA (M2). At the end of incubation, DCPA (M2) accounted for 70.9% (day 56) and 38.0% (day 29) of the applied radioactivity for Labels I and II, respectively. The third radioactive fraction (M3) did not exceed 4.5 and 5.4% (label I and label II) of the applied radioactivity. At pH 9 CAPA (M1) represented more than 94% of the applied radioactivity (Labels I and II) at the end of the incubation at 25 °C (day 29). At the higher temperature, it reached a maximum of about 95% (Label I) and 94% (Label II) after 1 day of incubation at 50 °C. Thereafter, it was hydrolysed to DCPA (M2). At the end of incubation (day 29), DCPA (M2) accounted for between 95% and 96% of the applied	van der Gaauw, A. 2003, Report No. 846211

Property	Results	Reference
	radioactivity for both labels	
Photolysis	Label I: ^{14}C-phenyl Fluazinam Radiochemical purity: >99% Label II: ^{14}C-pyridyl Fluazinam Radiochemical purity: >99% The half-life in sterile pH 5 buffer was 2.5 days for both labels. One major photolyte was detected for both labels accounting for 17.1% (label I) and 14.0% (Label II). It was identified as G-504. The other photolytic product was CO_2 (17.7% and 16.0% of label I and II, respectively after 30 days).	Lentz, N.R. and Korsch, B.H. 1995, Report No. 5312-94-0019-EF-002
	Quantum Yield ($\Phi = K_h/I_a$): 5.1×10^{-5} (pH 9 buffer) 1.7×10^{-5} (pH 6 distilled water) 2.1×10^{-6} (pH 5 buffer)	Wadley, A.M. 1992, Report No. RIC1726
Dissociation constant (pKa)	Determined at 20 °C: The average pKa from three trials was 7.34 in the pH range of 2-12 using the UV spectrophotometric method (OECD 112).	Gallacher, A.C. 1992, Report No. 4039-91-0387-AS-001

Technical material

Property	Results	Reference
Appearance	Purity 97.7%: Yellow, solid (Munsell colour 5Y 9/4 or 5Y/5), with a weak aromatic hydrocarbon-like odour. Purity 96.8%: Mustard yellow, solid granular powder at 24 °C (Munsell colour 5Y 8/10), with a strong musty odour.	Asai, N. 1991a, Report No.1216-90-06303-1; Oguri, M. 1991, Report No. 1216-90-06302-1; Asai, N. 1991b, Report No.1216-90-06304-1; Wojcieck, B.C. 1993, Report No. 4039-92-0500-AS-001;
Melting range	Purity 96.8%: Sample melted at 119 °C (metal block/capillary)	Wojcieck, B.C. 1993, Report No. 4039-92-0500-AS-001;
Bulk Density	Purity 96.8%: 1.02 g/cm^3 at 25 °C	Wojcieck, B.C. 1993, Report No. 4039-92-0500-AS-001;
Solubility in organic solvents at 25ºC	Purity 96.8%: Hexane: 8 g/L Methanol: 192 g/L Ethyl ether:231 g/L Dichloromethane: 675 g/L Toluene: 451 g/L Ethyl acetate: 722 g/L Acetone: 853 g/L Octanol: 41 g/L	Sanders, J.M. 1993, Report No. 4039-91-0384-AS-001
Octanol/Water Partition Coefficient at 25 °C	Purity 96.8%: Mean coefficient = 1.08×10^4 Mean log K_{ow} = 4.03	Sanders, J.M. 1992 Report No. 4039-91-0386-AS-001
Thermal stability (Flammability)	Purity 96.7%: Preliminary test: The test substance could not be ignited by a flame, although it melted and turned brown. Fluazinam technical material is not “highly flammable” according to the test method.	van Rijsbergen, L.M. 2002b, Report No. 341191
Thermal stability (Auto flammability)	Purity 96.7%: No self-ignition up to 400 °C	van Rijsbergen, L.M. 2002c, Report No. 341202

Property	Results	Reference
Thermal stability (Explosive properties)	Purity 97.8%: Fluazinam technical material is not thermally sensitive (effect of a flame) and is not sensitive to shock and friction. Fluazinam is not considered as explosive on the basis of the test results.	Angly H. 2005, Report No. 2005.2004.EXP

Formulation

Formulations of fluazinam are available as suspension concentrates and wettable powders.

Formulation type	Active substance/s and content	Application type
SC (Soluble Concentrate)	Fluazinam 500 g/L	Foliar applications
WP (Wettable Powder	Fluazinam 500 g/Kg	Foliar applications

METABOLISM AND ENVIRONMENTAL FATE

Radiolabel Position

Radiolabelled studies were undertaken using ^{14}C -fluazinam labelled either in the phenyl or pyridyl ring as shown in Figure 1.

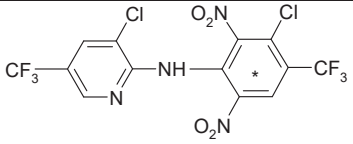
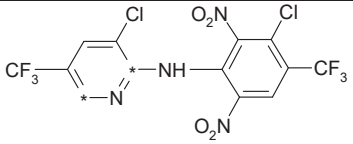
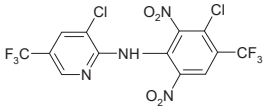
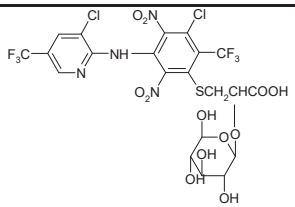
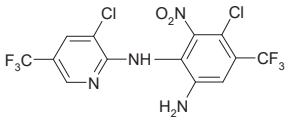
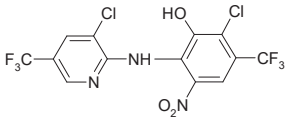
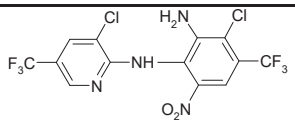
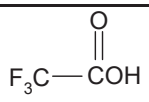
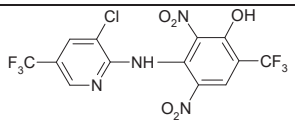
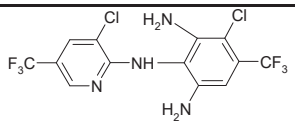
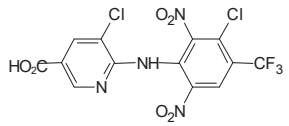
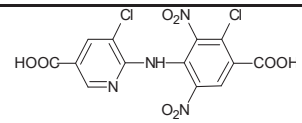
^{14}C-(Ph)-Fluazinam [^{14}C-Phenyl] Fluazinam Phenyl label	 * position of ^{14}C radiolabel (phenyl ring)
^{14}C-(Py)-Fluazinam [^{14}C-Pyridine] Fluazinam Pyridyl label	 * position of ^{14}C radiolabel (pyridine ring)

Figure 1 [^{14}C]-labelled test materials used in animal metabolism, plant metabolism and environmental fate studies

The chemical structures of the major degradation compounds from the metabolism of fluazinam are provided below in Table 1.

Table 1 Structure of compounds appearing in metabolism and environmental fate studies

Chemical name (IUPAC)	Compound Name/Code	Structure	Occurrence in metabolism studies
3-Chloro-*N*-(3-chloro-5-trifluoromethyl-2-pyridyl)-*α,α,α*-trifluoro-2,6-dinitro-*p*-toluidine	Fluazinam, IKF-1216		Potatoes, peanut (foliage), grapes, apples, laying hen (liver, kidney, muscle, fat, egg yolk), RAT
3-[[4-amino-3-[[3-chloro-5-(trifluoromethyl)-2-pyridyl]amino]- *α,α,α*-trifluoro-6-nitro-*o*-tolyl]thio]-2-(β-D-glucopyranosyloxy) propionic acid	AMGT		Potatoes grapes, wine, apples

Chemical name (IUPAC)	Compound Name/Code	Structure	Occurrence in metabolism studies
2-(6-amino-3-chloro-*α,α,α*-trifluoro-2-nitro-*p*-toluidino)-3-chloro-5-(trifluoromethyl) pyridine	AMPA		Potatoes, peanut (foliage), wine goat (liver, kidney, muscle, fat, milk), laying hen (liver, kidney, muscle, fat, egg yolk and white), RAT
2-chloro-6-[(3-chloro-5-(trifluoromethyl)-2-pyridyl)amino]- *α,α,α*-trifluoro-5-nitro-*m*-cresol	SDS-67230		Grapes, apples
2-(2-amino-3-chloro-*α,α,α*-trifluoro-6-nitro-*p*-toluidino)-3-chloro-5-(trifluoromethyl) pyridine	MAPA		Laying hen (liver, kidney, muscle, fat egg yolk and white)
Trifluoroacetic acid	TFAA		Potatoes, peanut (foliage), apples rotational crops: lettuce (DAT 30) carrots (DAT 30) barley grain: DAT 120 DAT 365
5-[(3-chloro-5-(trifluoromethyl)-2-pyridyl)amino]- *α,α,α*-trifluoro-4,6-dinitro-*o*-cresol	HYPA		Laying hen (liver, kidney, muscle, fat egg yolk and white); SOIL (major)
3-chloro-2-(2,6-diamino-3-chloro-*α,α,α*-trifluoromethyl-*p*-toluidino)-3-chloro-5-(trifluoromethyl) pyridine	DAPA		goat (liver, kidney, muscle, fat, bile, urine, milk) laying hen (liver, kidney, muscle, fat egg yolk and white), RAT
5-Chloro-6-(3-chloro-2,6-dinitro-4-trifluoromethylanilino) nicotinic acid	CAPA		Potato Hydrolysis
6-(4-Carboxy-3-chloro-2,6-dinitroanilino)-5-chloronicotinic acid	DCPA		Hydrolysis
4,9-dichloro-6-nitro-8-(trifluoromethyl)-pyrido-[1,2-α]benzimidazole-2-carboxylic acid	G-504		Hydrolysis

Plant metabolism

The meeting received information on metabolism of fluazinam after foliar application in apple, grape, potato, and peanut. Fluazinam was either labelled in the phenyl or pyridine ring.

Potato

Two studies investigating the metabolism of fluazinam in potatoes were provided to the meeting.

Study 1 (Galica, H. 1991)

Seed potatoes (variety Urgenta) were planted outdoors in a clay loam soil. Within the study potatoes were treated with phenyl (radiochemical purity 98.8%, specific activity 116.2 mCi/g) and pyridyl labelled (radiochemical purity 99.3%, specific activity 129 mCi/g) fluazinam formulated as a SC.

Two application regimes were investigated; in the first regime (low dose) potatoes received 4 applications at 0.6 kg ai/ha and in the second regime (high dose) potatoes received 4 applications at 1.8 kg ai/ha. Applications were undertaken 55, 76, 99 and 105 days after sowing.

Potato tubers were sampled 7 and 22 days after the last application with the latter time period representing crop maturity.

Potato tubers were washed and peeled. Potato tubers, peel and pulp were homogenised and the radioactivity was determined by combustion. The samples were stored frozen at ≤-18 °C and analysed within 4 months.

The TRR in potato tubers and peel and washings are shown in Tables 2 and 3.

Table 2 Radioactive residues in potato tuber after application of ^{14}C-phenyl or ^{14}C-pyridyl-fluazinam

Harvest	Label	Dose	Amount found by		
			Combustion [mg eq/kg] (% TRR)	Washings [mg eq/kg] (% TRR)	Total [mg eq/kg] (% TRR)
7 DALA (green harvest)	Pyridyl	4 × 0.6 kg ai/ha	0.055 (94.8%)	0.003 (5.2%)	0.058 (100%)
	Phenyl	4 × 0.6 kg ai/ha	0.065 (97.0%)	0.002 (3.0%)	0.067 (100%)
	Pyridyl	4 × 1.8 kg ai/ha	0.105 (97.2%)	0.003 (2.8%)	0.108 (100%)
	Phenyl	4 × 1.8 kg ai/ha	0.109 (98.2%)	0.002 (1.8%)	0.111 (100%)
22 DALA (maturity)	Pyridyl	4 × 0.6 kg ai/ha	0.072 (88.9%)	0.009 (11.1%)	0.081 (100%)
	Phenyl	4 × 0.6 kg ai/ha	0.069 (94.5%)	0.004 (5.5%)	0.073 (100%)
	Pyridyl	4 × 1.8 kg ai/ha	0.100 (95.2%)	0.005 (4.8%)	0.105 (100%)
	Phenyl	4 × 1.8 kg ai/ha	0.114 (95.8%)	0.005 (4.2%)	0.119 (100%)

Table 3 Radioactive residues in potato peel and pulp after application of ^{14}C-phenyl or ^{14}C-pyridyl-fluazinam

Harvest	Label	Dose	Total radioactive residues	
			Peel [mg eq/kg]	Pulp [mg eq/kg]
7 DALA (green harvest)	Pyridyl	4 × 0.6 kg ai/ha	0.105	0.050
	Phenyl	4 × 0.6 kg ai/ha	0.083	0.064
	Pyridyl	4 × 1.8 kg ai/ha	0.243	0.092
	Phenyl	4 × 1.8 kg ai/ha	0.119	0.108
22 DALA (maturity)	Pyridyl	4 × 0.6 kg ai/ha	0.107	0.067
	Phenyl	4 × 0.6 kg ai/ha	0.106[a]	0.064
	Pyridyl	4 × 1.8 kg ai/ha	0.189	0.090
	Phenyl	4 × 1.8 kg ai/ha	0.139	0.111

[a] This result from peel combustion was not comparable to the values determined by extraction/ combustion using the two extraction procedures (0.079 and 0.076 mg/kg)

Homogenised potato tubers, pulp and peel were extracted using two different procedures.

In the first extraction procedure, samples were extracted four times with acetonitrile: water (80: 20, v/v) followed by extraction with acetonitrile, methanol: water (80:20, v/v) and water. The acetonitrile: water extracts were partitioned to characterise the organo-soluble radioactivity.

Following the initial solvent extraction, the remaining soilds were further treated with cellulase followed by acid and base hydrolysis. The solutions obtained from enzyme, acid and base treatments were partitioned with an organic solvent. Selected extracts were pooled and concentrated by rotary evaporation. Concentrated extracts from pulp were partitioned with dichloromethane and/or ethyl acetate at neutral pH, pH 1-2 and pH 12.

The TRR following the first extraction procedure are shown in Table 4

Table 4 Distribution of radioactivity in potato pulp and peel fractions following application of ^{14}C-phenyl or ^{14}C-pyridyl-fluazinam expressed as residues in whole potato–extraction procedure 1

Fraction	Pyridyl label, low dose			Phenyl label, low dose		
	Peel [mg eq/kg] (% TRR)	Pulp [mg eq/kg] (% TRR)	Whole potato [mg eq/kg] (% TRR)	Peel [mg eq/kg] (% TRR)	Pulp [mg eq/kg] (% TRR)	Whole potato [mg eq/kg] (% TRR)
ACN 80%	0.049 (53.8%)	0.024 (45.3%)	0.028 (47.5%)	0.030 (38.0%)	0.018 (34.6%)	0.019 (34.5%)
Organic phase	0.034 (69.4%)	0.007 29.2%)	0.011 (39.3%)	0.019 (63.3%)	0.004 (22.2%)	0.006 (30.0%)
Aqueous phase	0.015 (30.6%)	0.017 (70.8%)	0.017 (60.7%)	0.011 (36.7%)	0.014 (77.8%)	0.014 (70.0%)
ACN	0.000 (0.0%)	<0.001 (<1.9%)	<0.001 (<1.7%)	0.001 (1.3%)	0.000 (0.0%)	<0.001 (<1.8%)
Water	0.002 (2.2%)	0.002 (3.8%)	0.002 (3.4%)	0.002 (2.5%)	0.003 (5.8%)	0.003 (5.5%)
MeOH 80%	0.001 (1.1%)	<0.001 (<1.9%)	<0.001 (<1.7%)	0.001 (1.3%)	<0.001 (<1.9%)	<0.001 (<1.8%)
PES						
Hydrolysis (cellulase)	0.002 (2.2%)	0.002 (3.8%)	0.002 (3.4%)	0.001 (1.3%)	0.002 (3.8%)	0.002 (3.6%)
Hydrolysis (HCl)	0.013 (14.3%)	0.018 (34.0%)	0.017 (28.8%)	0.009 (11.4%)	0.025 (48.1%)	0.023 (41.8%)
Hydrolysis (HCl, reflux)	0.010 (11.0%)	0.005 (9.4%)	0.006 (10.2%)	0.009 (11.4%)	0.003 (5.8%)	0.004 (7.3%)
Hydrolysis (1M KOH)	0.005 (5.5%)	0.001 (1.9%)	0.002 (3.4%)	0.003 (3.8%)	0.001 (1.9%)	0.001 (1.8%)
Hydrolysis (6M KOH)	0.007 (7.7%)	-	0.001 (1.7%)	0.018 (22.8%)	-	0.002 (3.6%)
Remaining soilds	0.002 (2.2%)	0.001 (1.9%)	0.001 (1.7%)	0.005 (6.3%)	<0.001 (<1.9%)	0.001 (1.8%)
PES total	0.039 (42.9%)	0.027 (51%)	0.029 (49.2%)	0.045 (57%)	0.032 (61.5%)	0.033 (59.9)
Total ^{14}C-residues [a]	0.091 (100%)	0.053 (100%)	0.059 (100%)	0.079 (100%)	0.052 (100%)	0.055 (100%)

[a] Total by summation and hence differ slightly from the levels in Table 3

Most of the radioactivity was found in the acetonitrile: water extractions. Thereafter, radioactivity was present at low levels in all extracts and only HCl hydrolysis of the PES yielded sufficient radioactivity to allow further analysis. The residue remaining in the solids after hydroysis was very low with 0.001 mg eq/kg for whole potato.

In the second extraction procedure, potato tubers, pulp and peel samples were extracted four times with acetonitrile: water (80:20, v/v) followed by Soxhlet extraction with acetonitrile overnight. Extracts were pooled and concentrated by rotary evaporation, partitioned twice with dichloromethane and once with ethyl acetate at neutral pH and at pH 1. These phases were

pooled and concentrated. The remaining water phase was hydrolysed with 1M HCl (pH 1) at 70 °C under nitrogen for 18 hours. Partitioning was performed with dichloromethane (tubers) or hexane (peel) and one to two times with ethyl acetate.

Extracts from the peel were partitioned at neutral pH and pH 1. The organic phases were pooled, further concentrated and analysed by TLC using reference standards. The water phase from pulp was lyophilised and hydrolysed with 2M HCl under reflux and partitioned with ethyl acetate. Unextracted radioactivity was determined by combustion.

The PES of the peel or potato tuber were subjected to acid hydrolysis (1 M HCl) at 90 °C. The resulting mixture was centrifuged, filtered and rinsed with 1M HCl and the remaining soilds analysed by combustion.

The results for extraction procedure 2 are outlined in Table 5.

Table 5 Distribution of radioactivity in potato pulp and peel fractions-extraction procedure 2

Extraction	Pyridyl label, low dose			Phenyl label, low dose		
	Peel [mg eq/kg potato] (% TRR)	Pulp [mg eq/kg potato] (% TRR)	Whole potato [mg eq/kg potato] (% TRR)	Peel [mg eq/kg potato] (% TRR)	Pulp [mg eq/kg potato] (% TRR)	Whole potato [mg eq/kg potato] (% TRR)
ACN: water (80:20, v/v)	0.007 (9.6%)	0.021 (28.8%)	0.028 (38.4%)	0.003 (4.5%)	0.017 (25.3%)	0.020 (29.8%)
Organic phase	0.004 (5.5%)	0.004 (5.5%)	0.008 (11.0%)	0.001 (1.5%)	0.004 (6.0%)	0.005 (7.4%)
Aqueous phase	0.003 4.1%)	0.018 (24.7%)	0.021 (28.8%)	0.002 (3.0%)	0.013 (19.3%)	0.015 (22.3%)
Hydrolysis of aqueous phase						
Organic phase	0.001 (1.4%)	0.001 (1.4%)	0.002 (2.7%)	<0.001 (<1.5%)	<0.001 (<1.5%)	<0.001 (<1.5%)
Aqueous phase	0.002 (2.7%)	0.017 (23.3%)	0.019 (26.0%)	0.002 (3.0%)	0.013 (19.3%)	0.015 (22.3%)
Soxhlet acetonitrile	0.001 (1.4%)	0.001 (1.4%)	0.002 (2.7%)	0.001 (1.5%)	<0.001 (<1.5%)	<0.002 (1.5%)
PES						
Organic phase	<0.001 (<1.4%)	0.000 (0.0%)	<0.001 (<1.4%)	<0.001 (<1.5%)	<0.001 (<1.5%)	<0.001 (<1.5%)
Aqueous phase	0.002 (2.7%)	0.023 (31.5%)	0.025 (34.3%)	0.001 (1.5%)	0.030 (44.6%)	0.031 (46.1%)
Remaining solids	0.005 (6.9%)	0.014 (19.2%)	0.019 (26.0%)	0.003 (4.5%)	0.012 (17.9%)	0.015 (22.3%)
Total PES	0.008 (11%)	0.037 (50.7%)	0.045 (61.7%)	0.005 (7.5%)	0.043 (64%)	0.047 (69.9%)
Total ^{14}C-residues	0.015 (20.6%)	0.059 (80.8%)	0.074 (101.4%)	0.074 (11.9%)	0.059 (87.8%)	0.067 (99.7%)
Total recovery based on whole potato	0.074 (101.4%)			0.067 (99.7%)		

In terms of whole potato, the acetonitrile: water and Soxhlet extracts accounted for 41.1% (0.03 mg eq/kg) and 31.3% (0.022 mg eq/kg) for potatoes treated with pyridyl- and phenyl-labelled fluazinam, respectively.

Expressed in terms of whole potato, the acid hydrolysis of the PES released 34.3% (0.025 mg eq/kg) for the pyridyl-label and 46.1% (0.031 mg eq/kg potato) for the phenyl-label.

TLC analysis was undertaken on various samples from the organic phases from the acetonitrile extracts, hydrolysis of the aqueous phases and some of the organic phases obtained from hydrolysis of pulp and peel PES. Owing to the oily consistency, the aqueous phases could not be further analysed. The results from TLC analyses of potato peel are shown in Table 6.

Table 6 Distribution of fluazinam and metabolites in organo-soluble fraction from acetonitrile extracts from potato peel analysed by TLC

Radioactive fraction	Rf-value SS 2 / SS 8	Identity	Potato peels (22 DALA)		
			[%] found in organic phase	[mg eq/kg peel]	[mg eq/kg whole potato]
Pyridyl-label: low dose					
M1	93 / 69	Fluazinam	13.2	0.004	0.001
M2	66 / 59	Unknown	3.4	0.001	<0.001
M3	97 / 44	Unknown	32.4	0.011	0.002
M4	89 / 35	MAPA	3.7	0.001	<0.001
M5	89 / 01	Unknown	21.4	0.007	0.001
M6	78 / 01	CAPA	14.8	0.005	0.001
M7	59 / 01	HYPA	7.6	0.002	<0.001
M8	44 / 01	Unknown	1.7	0.001	<0.001
Total			100.0	0.032	0.005
Phenyl-label: low dose					
M1	94 / 78	Fluazinam	38.9	0.003	<0.001
M4	94 / 39	MAPA	8.5	0.001	<0.001
M5	94 / 01	Unknown	29.9	0.002	<0.001
M6	73 / 01	CAPA	5.4	<0.001	<0.001
M7	59 / 01	HYPA	4.5	<0.001	<0.001
M9	44 / 57	Unknown	6.0	<0.001	<0.001
M10	37 / 16	Unknown	6.8	0.001	<0.001
Total			100.0	0.007	0.001

In potato peel from the pyridyl-label, fluazinam was detected amounting to 0.004 mg eq/kg (0.001 mg/kg whole as potato). The largest fraction was unknown M3 with 0.011 mg eq/kg (0.002 mg eq/kg whole potato).

All other fractions did not represent more than 0.007 mg eq/kg (0.001 mg eq/kg whole potato).

In potato peel from the phenyl-label, fluazinam was the most abundant fraction with 0.003 mg/kg (<0.001 mg/kg as whole potato). No other radioactive fraction accounted for more than 0.002 mg eq/kg (<0.001 mg eq/kg as whole potato).

The proposed metabolic pathway for fluazinam in potatoes is outlined in Figure 2.

Figure 2 Proposed metabolic pathway of fluazinam in potato

Study 2 (Jentoft, N.H. 1997)

Seed potatoes (variety Kennebec) were planted in a sand loam soil. Phenyl (radiochemical purity 98%, specific activity 57.3 mCi/mmole)) or pyridyl (radiochemical purity 98%, specific activity 66.2 mCi/mmole) labelled fluazinam was applied as a SC formulation. Three different application regimes were investigated as outlined in table 7.

Table 7 The application details for the metabolism studies conducted on potato

Plant designated as:	Phenyl 1	Phenyl 2	Pyridyl
Application rate per treatment (kg a.i./ha)	0.505	0.505	0.430
Number of applications	4	4	4
Application rate (total) (kg a.i./ha)	2.02	2.02	1.72
Interval between applications (days)	14, 11, 9	14, 11, 9	14, 11, 9
Harvest	6 days after last application	7 days after last application	7 days after last application

Potatoes were harvested either 6 or 7 days after the last application. Tubers were harvested, air dried, and brushed lightly to remove soil.

Potatoes were homogenised for analysis with TRR determined by combustion. In addition, one potato from each group was peeled and the peel and pulp homogenised. The samples were stored frozen at ≤ -18 °C with all analysis being completed within 3 months.

Potato tuber, peel and pulp samples were each extracted two times with three volumes of acetonitrile. Undissolved solids were removed by centrifugation after each extraction. The extracts from each plant were combined and concentrated by rotary evaporation. The remaining solids were then re-extracted with three volumes of acetonitrile: water (1:1, v/v) using the same procedure and the acetonitrile: water extracts from each plant combined.

Further analysis of the PES was undertaken to demonstrate the conversion of fluazinam into natural products (starch). The PES was subjected to acid hydrolysis (1M H_2SO_4) for 6 hours. After neutralization, the resulting glucose solution was reduced to sorbitol with $NaBH_4$. Sorbitol was acetylated with acetic anhydride and then water was added to stop the reaction.

The TRR in potatoes are shown in Table 8.

Table 8 Distribution of radioactivity in potato tuber fractions following application of ^{14}C-phenyl or ^{14}C-pyridyl-fluazinam

Fraction	Phenyl 1		Phenyl 2		Phenyl average		Pyridyl	
	[mg eq/kg]	% TRR	[mg eq/kg]	% TRR	[mg eq/kg]	% TRR	[mg eq/kg]	% TRR
Homogenate	0.0102	100.0	0.0120	100.0	0.0111	100.0	0.0250	100.0
ACN extract	0.0017	16.9	0.0024	19.9	0.0020	18.4	0.0079	31.5
ACN: Water (1:1) extract	0.0014	13.9	0.0025	20.7	0.0019	17.3	0.0038	15.2
Total extracted residue	0.0031	30.8	0.0049	40.6	0.0040	35.7	0.0117	46.7
PES	0.0048	47.5	0.0065	54.7	0.0057	51.1	0.0119	47.8
Total recovery [%]	78.3		95.3		86.8		94.5	

None of the extracts accounted for more than 0.01 mg eq/kg and as a result only limited identification work was undertaken. HPLC analysis of all of the extracts was undertaken. The resulting data was used to characterise the residue as the total polar fraction and the total non-polar fraction. Therefore overall the TRR found in potato tubers was characterised as 1) PES, 2) total extractable polar fraction and 3) total extractable non polar fraction.

The PES contained about half of the TRR, while the polar extractable and the non-polar extractable fractions contained 27-31% and 9-16% of the TRR, respectively (see Table 10).

To examine the nature of the residue in the PES, the glucose units of starch were converted to sorbitol hexa-acetate through a series of chemical reactions. For both the phenyl- and pyridyl-labelled ^{14}C-fluazinam samples, the amount of radioactivity recovered in twice-re-crystallised sorbitol hexa-acetate was sufficient to account for almost all of the radioactive residue in the PES fraction.

The total polar extractable fraction represents 0.003 and 0.008 mg eq/kg in the phenyl- and pyridyl-label treated potatoes, respectively. Since the levels were low attempts to identify components present in this fraction were unsuccessful, although TFAA was identified. However, the fact that the residue was eluted from an HPLC reverse phase column significantly earlier than fluazinam implies that the components do not retain the original fluazinam structure. Any fluazinam derivative retaining most of the carbon skeleton of the parent compound would have to be substituted with a highly polar group in order to elute that early. This is indicative that the components in this fraction are made up of small polar compounds.

The total nonpolar fraction represents less than 0.001 and 0.004 mg eq/kg for the phenyl- and pyridyl-label treated potatoes respectively. As with the polar fraction, the low residue levels found resulted in limited identification work. The resulting HPLC data demonstrated that there was no single component above 0.001 mg eq/kg. A comparison of the retention times allowed fluazinam to be identified and its level estimated. Small amounts of radioactivity were eluted at the retention times corresponding to AMGT and AMPA. Additional confirmation of fluazinam was achieved by TLC. Confirmation of identity of the other analytes was not achieved.

Table 9 Assignment of whole potato TRR to fluazinam and metabolites

Fraction	Phenyl		Pyridyl	
	[mg eq/kg]	% TRR	[mg eq/kg]	% TRR
Homogenate	0.011	100	0.025	100
PES	0.006	51.1	0.012	47.8
Starch	0.005	43.9	0.012	47.3
Extracts	0.004	35.7	0.012	46.7
Total polar	0.003	27.2	0.008	30.9
TFAA	<0.001	0.9	<0.001	0.6
Total non-polar	0.001	8.5	0.004	15.8
AMGT	<0.001	2.2	<0.001	2.7
AMPA	<0.001	1.4	<0.001	3.1
fluazinam	<0.001	2.3	0.001	5.9
other	<0.001	2.4	0.001	4.1

Additional studies on the distribution of residue in potato were carried out by analysing peel and pulp separately. The results demonstrate differences in the distribution of the residues in peel compared to pulp (see Table 10). For peel, almost all the extractable residues appear in the acetonitrile extract, while the extractable residues from pulp are more or less evenly split between acetonitrile and acetonitrile: water (1:1, v/v) extracts.

Table 10 Distribution of radioactivity in potato peel and pulp fractions

Fraction	Pulp		Peel	
	[%TRR]	[mg eq/kg]	[%TRR]	[mg eq/kg]
Initial	100%	0.0159	100%	0.0109
Acetonitrile extract	29%	0.0046	42%	0.0046
Acetonitrile: water (1:1) extract	21%	0.0033	6%	0.0006
Total extractable	50%	0.0080	48%	0.0052
PES	46%	0.0073	42%	0.0046
Total recovery	96%		90%	

After application of phenyl-label and pyridyl-label ^{14}C-fluazinam on potato plants, overall levels of ^{14}C residues in potato tubers were low. The highest residue levels were found in the pyridyl-labelled treated potatoes with 0.025 mg eq/kg.

Non-polar residues were very low. The total amount of non-polar residue was less than 0.004 mg/kg (15.8% TRR) and consisted of multiple components. The amount of fluazinam found in all samples was a maximum of 0.001 mg eq/kg (5.9% TRR).

The polar fraction showed maximum residues of 0.008 mg/kg (30.9% TRR). TFAA was identified in this fraction.

The PES accounted for a maximum residue of 0.012 mg eq/kg (47.3% TRR). The PES consisted mosttly of radioactivity incorporated into natural products such as starch.

The presence of radioactivive residues in starch from both labels indicates that both rings are broken down into fragments that can enter the carbon pool.

Grape (Neal, 1996)

Field-grown grapevines (variety Pinot Noir) were treated twice with ^{14}C-labelled fluazinam. Plants were treated with either the phenyl (radiochemical purity 99.4%, specific activity 122.6 mCi/g) or pyridyl-label (radiochemical purity 98%, specific activity 101.9 mCi/g) at a rate of 750 g ai/ha. The test material was formulated to be representative of a 500 SC formulation. The first application was made at 80% of petal fall and the second at bunch closure (35 days after the first application).

Grapes were harvested 71 days after the last application. The samples were stored at ≤ -18 °C with the analysis being completed within 6 months.

There were two parts to the study.

In the first part, the nature and magnitude of residues in grape berries were determined. Berries treated with both labels were extracted with acetonitrile: water (9:1, v/v). The mixture was separated by centrifugation and the liquid phase was removed. The process was repeated with acetonitrile: water (9:1, v/v) and then with acetonitrile: water (1:1, v/v). The combined extracts were concentrated and subjected to liquid scintillation counting (LSC).

The acetonitrile/ water combined extract was partitioned with three portions of hexane and then with four portions of ethyl acetate. The aqueous phase remaining after extraction was separated into several fractions by a combination of reversed-phase, cation and anion exchange chromatography. The individual extracts were concentrated and subjected to LSC.

For investigation of the nature of the residue, the hexane and ethyl acetate phases were concentrated and analysed by HPLC; radioactivity was quantitated by counting fractions collected from the HPLC run. The concentrated extracts were also analysed by GC-MS (hexane extracts) or by LC-MS (ethyl acetate extracts, following purification be semi-preparative HPLC) in order to identify the major component. To assure that the products derived from the different labels were the same, portions from the different labels were mixed and the mixture was analysed by HPLC.

The radioactivity in the remaining post-extraction solids (PES) were quantified by combustion analysis.

The PES were subjected to a series of treatments in which the solids were degraded in a stepwise fashion into six fractions representing cell wall components. The levels of radioactivity were determined by LSC. The resulting aqueous phase extract was extracted with three portions of hexane followed by four portions of ethyl acetate. The aqueous phase remaining after extraction was separated into several fractions by a combination of reversed-phase, cation and anion exchange chromatography in order to record radioactivity containing sugars. All of the fractions were concentrated, and the radioactivity analysed by LSC.

Samples of grapes were also analysed further to determine the level of fluazinam and AMGT. Analysis of fluazinam was undertaken using a GC –ECD and AMGT was determined by HPLC-UV.

The TRR determined in grape berries was 1.69 mg eq/kg from grapes treated with phenyl-label and 1.66 mg eq/kg in grapes treated with pyridyl-label fluazinam (Table 11).

Table 11 Distribution of radiolabel in grapes following application of ^{14}C-phenyl or ^{14}C-pyridyl-fluazinam

Fraction	Phenyl-label[a]		Pyridyl-label	
	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]
Grape, TRR	1.69	100	1.66	100
Initial extraction (ACN: water extractions)	0.96	56.8	0.81	48.8
Hexane phase	0.48	28.4	0.32	11.4
Fluazinam	0.36	21.3	0.19	11.4
SDS-67230	0.0067	0.4	0.015	0.9
Others	0.12	7.1	0.13	7.8
Ethyl acetate phase	0.24	14.2	0.21	12.7
AMGT	0.06	3.6	0.065	3.9
Others [b]	0.18	10.7	0.15	8.7
Aqueous phase	0.22	13.0	0.15	9.0
Sugars	0.026	1.5	0.045	2.7
Others [b]	0.194	11.5	0.11	6.3
PES	0.73	43.2	0.85	51.2
Starch[c]	≤0.024	<5	≤0.024	<5
Protein[c]	≤0.024	<5	≤0.024	<5

Fraction	Phenyl-label[a]		Pyridyl-label	
	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]
Pectin[c]	≤0.024	<5	≤0.024	<5
Lignin	0.11	11.1	0.17	10.2
Ethyl acetate extract	0.015	0.89	0.036	2.2
Hemicellulase	0.14	8.3	0.16	9.7
Ethyl acetate extract	0.018	1.0	0.072	4.3
Cellulose	0.18	17.9	0.17	10.2
Ethyl acetate	0.018	1.06	0.038	2.3
Total	1.69	100	1.66	100

[a] Data from one of two batches analysed used for calculation. Total residue in second batch was 1.16 mg/kg.

[b] Broadly distributed over chromatographic run with significant number of peaks

[c] Study only reports values as being less than 5% of the TRR (≤0.024 mg eq/kg) for these fractions

The hexane phase contained fluazinam as the major radioactive compound. Fluazinam was found at levels of 0.36 mg eq/kg (21.3% of the TRR) and 0.19 mg eq/kg (11.4% of the TRR) for the phenyl- and pyridyl-labelled grapes, respectively. A minor metabolite was identified as SDS-67230 (< 1% of the TRR).

The ethyl acetate phase contained a glucose conjugate of AMPA, designated as AMGT at levels of 0.06 mg eq/kg (3.6% of the TRR) and 0.065 mg eq/kg (3.9% of the TRR) for the phenyl- and pyridyl-labels respectively.

The remainder of the radioactive residues in each of the organic extracts was accounted for as material broadly distributed over the chromatographic trace with no significant discrete radioactive peaks.

In the aqueous phase remaining after extraction, the most polar fraction was found to contain radioactivity that had been re-incorporated into sugars. This indicates that extensive degradation of fluazinam followed by re-incorporation of ^{14}C into natural products.

The PES was subjected to a series of treatments in which the solids were degraded in stepwise fashion into fractions representing cell wall components (water-soluble polysaccharides and proteins, starch, protein, pectin, lignin, hemicellulose and cellulose). This involved the PES being incubated sequentially with phosphate buffer, incubation with phosphate buffer and α-amalyse, incubation with tris buffer and protease, incubation with sodium acetate/EDTA, incubation with acetic acid and sodium chlorite, incubation with potassium hydroxide solution and incubation with sulphuric acid.

Less than 5% of the radioactivity was released in each of the starch, protein and pectin fractions. The lignin, hemicellulose and cellulose fractions contained 0.1-0.2 mg eq/kg of radioactivity. Each of these three fractions was extracted with ethyl acetate; the extracts were found to contain from 0.015 mg eq/kg up to 0.072 mg eq/kg of radioactivity. The extracts were further analysed by HPLC; none of the resulting HPLC-fractions amounted to more than 0.01 mg eq/kg. There was one discrete peak found in the hemicellulose and cellulose fractions from the pyridyl-labelled sample which did not match any of the standards; however, it was present at approximately 0.01 mg eq/kg in each of the two fractions.

In the second part of the study, bunches of grapes from the treated and untreated vines from both labels were used to produce two kinds of wine; vin de goutte and vin de presse. Fermentations were started with bunches (grapes plus stems) which had not been frozen. Bunch of grapes were transferred into glass metabolism bottles equipped with sterile filters and $^{14}CO_2$ trapping systems. The bottles were ventilated with nitrogen gas to avoid aerobic conditions and the fermentation process was started by adding yeast-suspension to each bottle. The fermentations were stopped when the specific gravity reached values below 1 and no more CO_2 was generated. At the end of the fermentations, each of the samples was filtered through a nylon bag (normally used for wine production) resulting in the vin de goutte samples. Each of the marcs from the filtrations were pressed to get the vin de presse.

The wines were extracted four times with hexane followed by four times with ethyl acetate. The aqueous phase remaining after organic extraction was analysed for radioactivity by LSC. Each extract obtained was concentrated and analysed by HPLC. Radioactivity was quantitated by counting the fractions from the HPLC run.

The combined hexane extract was concentrated and purified by HPLC on a silica gel column, the resulting fractions were analysed for radioactivity by LSC. Vin de presse (phenyl label) was extracted in a separate experiment with three portions of hexane; the combined extract was concentrated and purified by HPLC. After further purification by HPLC the resulting products were analysed by GC-MS. Vin de presse (pyridyl label) was extracted in a third run with four portions of hexane; the combined extract was concentrated and analysed by GC-MS.

The combined ethyl acetate extract was concentrated and purified by semi-preparative HPLC in several steps. After final purification the product was analysed by LC-MS. An aliquot of the ethyl acetate extract was also concentrated and heated after

addition of HCl. The reaction mixture was cooled and extracted three times with ethyl acetate. The combined extract was concentrated and analysed by HPLC.

In order to determine whether the ethanol produced from the fermentation contained radioactive residues, samples of wine were distilled. The volume of two collected fractions and the residual liquid were measured and the radioactivity in each sample was determined by LSC.

The TRRs determined in the samples of *vin de presse* produced from grapes treated with either phenyl- or pyridyl-labelled fluazinam were found to be 0.73 mg eq/kg.

Vin de goutte produced from grapes treated with phenyl-labelled fluazinam contained 0.41 mg eq/kg of radioactive residues, while the *vin de goutte* from the pyridyl-labelled treatment contained 0.54 mg eq/kg of radioactive residues (Table 12).

Table 12 Distribution of radioactivity in wine and various fractions after application of ^{14}C-phenyl or ^{14}C-pyridyl-fluazinam

Fraction	Phenyl-label		Pyridyl-label	
	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]
Vin de presse				
Wine	0.73	100	0.73	100
Unaccounted for radioactivity†	0.22	30.7	0.14	19.3
Hexane extract	0.046	6.3	0.049	6.7
AMPA	0.027	3.7	0.024	3.3
others [a b]	0.019	2.6	0.025	3.4
EtOAc Extract	0.13	17.8	0.21	28.8
AMGT	0.053	7.3	0.076	10.4
AMPA	0.010	1.4	0.014	1.9
others [a b]	0.067	9.2	0.12	16.4
Total AMPA [c]	0.037	5.1	0.038	5.2
Aqueous phase	0.33	45.2	0.33	45.2
Ethanol	Not determined	Not determined	0.043	5.9
Vin de goutte				
Wine	0.41	100	0.54	100
Unaccounted for radioactivity†	0.033	8.1	0.09	17.1
Hexane extract	0.017	4.1	0.018	3.3
AMPA	0.010	2.4	0.0065	1.2
others [a b]	0.007	1.7	0.012	2.1
Ethyl acetate Extract	0.13	31.7	0.19	35.2
AMGT	0.051	12.4	0.065	12.0
AMPA	0.0041	1.0	0.0077	1.4
others [a b]	0.075	18.3	0.1173	21.7
Total AMPA [c]	0.014	3.4	0.014	2.6
Aqueous phase	0.23	56.1	0.24	44.4
Ethanol	0.022	5.4	0.032	5.9

† Information on the radioacitivty unaccounted for is not given in the study

[a] Calculated by subtracting mg eq/kg values of metabolites from total in extract

[b] Remaining radioactivity distributed over several fractions, trace levels of fluazinam and MAPA (isomer of AMPA in which the nitro and amino groups are reversed) were reported

[c] Sum of amount in hexane and ethyl acetate extracts

The hexane extract was found to contain AMPA (maximum 0.027 mg eq/kg, 3.7% of the TRR) as the only significant component. Very low levels of fluazinam and MAPA were detected. However, the quantities were not sufficient for GC-MS analysis. The assignment was based upon HPLC retention times only.

The ethyl acetate extracts contained AMGT (0.051–0.076 mg eq/kg, 7.3–12.4% of the TRR) as the only significant radioactive product; minor amounts of AMPA were also present. In each of the organic extracts, no other discrete metabolites that amounted to more than 1% of the TRR were observed. The remainder of the radioactive residues in each of the organic extracts was accounted for as material broadly distributed over the chromatographic trace with no significant discrete radioactive peaks.

The ethanol produced in the fermentation of the grapes was found to contain radioactivity (0.022–0.032 mg/kg, 5.4–5.9% of the TRR).

The proposed metabolic pathway for fluazinam in grapes is shown in Figure 3.

Figure 3 Proposed metabolic pathway of fluazinam in grapes

Apple (McClanahan, 1996)

Apple trees (variety Golden delicious) grown outdoor were treated with either phenyl (radiochemical purity 98%, specific activity 57.3 mCi/mmol) or pyridyl-labelled fluazinam (radiochemical purity 98%, specific activity 66.2 mCi/mmol).

The test material was applied as a flowable concentrate formulation in a total of six foliar applications of approximately 0.93 kg ai/ha per application. The first application was applied at the tight cluster growth stage, 161 days before harvest. The following five applications were made at intervals of 9, 22, 34, 34, and 30 days.

Samples of immature fruit and foliage were removed throughout the course of the study. All remaining apples were harvested 32 days after the last application. The samples were initially stored at 4 °C for 1 week and then stored frozen prior to analysis at ≤ -18 °C with the analysis being conducted within 6 months. Storage stability was investigated in the study be the re-extraction of several fractions 9 months after storage. The metabolic profiles determined initially and after 9 months were comparable.

Fruits were washed with acetonitrile to remove surface residues and then ground with dry ice and centrifuged to yield pomace and juice. Apple foliage samples were either cut into small pieces or powdered with dry ice. The total radioactivity in pomace and foliage samples was determined by combustion.

Pomace samples were extracted with acetonitrile and partitioned with ethyl acetate to generate aqueous and organic fractions. Juice was partitioned with ethyl acetate to generate the same fractions. The surface washes, ethyl acetate and aqueous fractions were each analysed by high performance liquid chromatography with radioactivity detection (HPLC-RAD).

Metabolites were identified by one or more of the following techniques: co-elution with authentic standards, mass spectrometric identification, and derivatisation followed by mass spectrometry, NMR or degradation techniques.

The nature of the un-extracted residue remaining in the PES from the pomace were characterised by degradation experiments. Un-extracted radioactive residues were fractionalised sequentially into starch, protein, pectin, lignin, hemicellulose, cellulose and insoluble radiocarbon. The process involved incubation of the PES with sodium acetate and EDTA at pH 4, incubation with acetic acid and anhydrous sodium chlorite, incubation with acetic acid at pH 4 and incubation with potassium hydroxide solution.

The total radioactive residue levels in apples treated with either ^{14}C-phenyl- or ^{14}C-pyridyl-fluazinam ranged from 1.9–2.8 mg eq/kg. Levels in the pomace extract and juice were very similar between the two labels, while levels in the surface wash and pomace PES were slightly different between the two labels.

The residue levels and percentage of the total radioactive residue of each fraction are shown in Table 13.

Table 13 Radioactivity levels in apples following application of ^{14}C-phenyl or ^{14}C-pyridyl-fluazinam

Fraction	Phenyl-label		Pyridyl-label	
	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]
Whole Apple	1.877	100	2.802	100
Surface Wash	0.684	36.4	1.282	45.8
Juice	0.158	8.4	0.207	7.4
Pomace Extract	0.209	11.1	0.309	11.0
Pomace PES	0.827	44.1	1.003	35.8

The distribution of the radioactivity residues in the surface wash, pomace and juice are outlined in Table 14.

Table 14 Distribution of radioactive residues in the surface wash, apple pomace and juice

	Fraction	Phenyl-label		Pyridyl-label	
		mg eq/kg	% TRR	mg eq/kg	% TRR
Surface wash	ACN wash	0.684	36.40	1.282	45.77
	Fluazinam	0.648	34.5	1.178	42.04
	SDS-67230	0.036	1.90	0.07	2.48
	Unidentified	-	-	0.034	1.25
Pomace	ACN extraction	0.209	11.32	0.309	11.031
	EtoAc phase	0.085	4.54	0.15	5.22
	Aqueous phase	0.105	5.57	0.13	4.62
	Fluazinam	0.04	2.11	0.076	2.73
	SDS-67230	0.007	0.36	0.01	0.36
	AMGT	0.004	0.19	0.011	0.38
	Sugars	0.064	3.43	0.07	2.49
	TFAA	0.003	0.16	-	-
	Unidentified	0.091	5.07	0.142	5.07

	Fraction	Phenyl-label		Pyridyl-label	
		mg eq/kg	% TRR	mg eq/kg	% TRR
	PES	0.827	44.1	1.003	35.8
	Pectin	0.035	1.86	0.054	1.92
	Lignin	0.112	5.95	0.207	7.38
	Hemicellulose	0.225	12.02	0.336	12.0
	Cellulose	0.116	6.20	0.242	8.63
	Unidentified	0.063	3.37	0.033	1.18
	Remaining solids	0.276	14.70	0.131	4.69
Juice	ACN extraction	0.158	8.4	0.207	7.4
	EtoAc phase	0.023	1.24	0.059	2.13
	Aqueous phase	0.13	6.67	0.134	4.79
	Fluazinam	0.001	0.06	-	-
	AMGT	0.01	0.52	0.014	0.52
	Sugars	0.097	5.16	0.1	3.55
	TFAA	0.02	1.07	-	-
	Unidentified	0.03	1.59	0.093	3.33

The main residue identified was parent fluazinam. Metabolites retaining the fluazinam moiety isolated from apples included AMGT and SDS-67230, although at levels <3% of the TRR.

After the initial extractions for apple pomace the PES accounted for 44.1% of the TRR (0.827 mg eq/kg) for the phenyl label and 35.8% of the TRR (1.003 mg eq/kg) for the pyridyl label. Degradation procedures showed that a portion of the radioactivity in the PES was associated with structural polymers of apple tissue such as pectin, lignin, hemicellulose and cellulose.

Additional evidence of complete degradation of fluazinam was the identification of trifluoroacetic acid (TFAA).

Specific quantification and identification work was not undertaken of the foliage samples. However, the chromatograms were qualitatively similar to those observed for the apple pomace extracts, indicating that the same metabolites were present in the foliage as those in the apple.

In summary, the main residue in apples treated with fluazinam was the unchanged parent, ranging from 0.689-1.254 mg eq/kg (37 to 45% of the TRR).

The two metabolites of fluazinam that retained the basic structural form of the parent molecule, SDS-67230 and AMGT, were present at levels below 3% of the TRR (≤ 0.08 mg eq/kg).

Radiolabelled sugars formed by incorporation of radioactivity accounted for 0.16–0.17 mg eq/kg (6% -9% of the TRR).

Structural polymeric compounds such as hemicellulose, pectin and cellulose accounted for 0.49–0.839 mg eq/kg (26%-30% of the TRR).

Trifluoroacetic acid comprised about 1% of the TRR (0.023 mg eq/kg).

The proposed metabolic pathway of fluazinam in apples is shown in Figure 4.

Figure 4 Proposed metabolic pathway of fluazinam in apples

Peanut (Hartman, D.A. 1995)

Peanut plants (variety florunner) were grown from seed in a sandy loam soil and later transplanted in water troughs filled with the same soil. Separate studies investigating the metabolism were undertaken in 1992, 1993 and 1994.

The plants were initially grown outdoors but weather conditions necessitated moving the growing plants into either a greenhouse or covering the plants with a portable enclosure.

The peanut plants were treated with four applications of 0.56 kg ^{14}C-fluazinam/ha each (total of 2.24 kg a.i/ha). Plants were treated with either phenyl (radiochemical purity 98%, specific activity 57 mCi/mmole) or pyridyl-labelled fluazinam (radiochemical purity 99.5%, specific activity 66 mCi/mmole).

The test material was formulated as a 40% flowable suspension and was applied by mixing with water and then spraying the peanut foliage. The details of the study regimes for the 1992, 1993 and 1994 metabolism investigations are shown in Table 15.

Table 15 The application details for the metabolism studies conducted on peanuts

Crop of year	1992	1993	1994
Number of troughs	3	4	4
Application rate per treatment (kg ai/ha)	0.56	0.56	0.56
Number of applications	4	4	4
Spraying intervals (days)	25, 30, 25	19, 17, 18	21, 22, 23
Sample collection Days after the last application	90	90	90
Samples	Foliage, shells, nutmeats	Foliage, shells, nutmeats	Foliage, shells, nutmeats

The samples taken from the 1992 investigation were used to develop methodology for the collection and analysis of samples from the investigations undertaken in 1993 and 1994. As such the 1992 data have not been considered further.

Samples of foliage and peanut were collected with the peanuts being shelled for separate analysis of the shells and nutmeats.

The foliage was rinsed with water and methanol before homogenisation and assayed by HPLC. The water/methanol rinse was also analysed.

Peanut shells were rinsed with water to remove any soil remaining on the shells. The water rinse was not analysed.

All samples were stored frozen at ≤ -18 °C with the analysis being completed within 6 months.

Homogenised tissues were combusted in order to determine the total radioactive residue in the three crop fractions. The individual crop fractions were then subject to different extraction procedures as follows:

The foliage and shells were extracted three times with acetonitrile: water (80:20, v/v). The extracts were combined and concentrated at reduced pressure using a rotary evaporator. The concentrated extracts were partitioned with dichloromethane.

The nutmeats were extracted three times with hexane, followed in some instances by 1-2 extractions with acetonitrile and then 1-2 times with water. The hexane extracts were combined and the solvent removed at reduced pressure. The acetonitrile and water extracts were treated similarly.

The PES remaing after the solvent extractions from each crop fraction were treated at elevated temperatures (80 °C) with acid (6 M HCl) or base (24% KOH). In addition, treatments with various enzymes, were undertaken to degrade the PES in a stepwise fashion for further characterisation of the radioactive residue. The degradation experiments involved the PES being incubated sequentially with phosphate buffer, incubation with phosphate buffer and α-amalyse, incubation with tris buffer and protease, incubation with sodium acetate/EDTA, incubation with acetic acid and sodium chlorite, incubation with potassium hydroxide solution and incubation with sulphuric acid.

Metabolites were identified using several techniques, including HPLC co-elution with standards, direct identification by mass spectrometry and comparison with standards, NMR and degradation experiments.

The distribution of residues in the peanut foliage, shells and nutmeats are outlined in Table 16.

Table 16 Radioactive residues in peanut crop fractions following the application of ^{14}C-phenyl-fluazinam or ^{14}C-pyridyl fluazinam

Foliage

Fraction	Phenyl-label		Pyridyl-label	
	mg eq/kg	% TRR	mg eq/kg	% TRR
80% ACN	9.4	36.7	14.34	46.7
Aqueous phase	4.72	18.4	7.94	31.2
Organic phase	4.68	18.3	6.45	25.5
Fluazinam	1.9	7.4	2.3	7.5
AMPA	0.40	1.6	0.24	0.8
TFAA	0.87	3.4	-	-

Fraction	Phenyl-label		Pyridyl-label	
	mg eq/kg	% TRR	mg eq/kg	% TRR
Unidentified	6.23	24.3	11.8	38.4
PES	15.9	61.7	17.8	57.7
Phosphate buffer	1.2	4.7	2.0	6.5
Carbohydrates	3.3	12.8	3.2	10.4
Protein	2.0	7.8	2.5	8.1
Pectin	2.3	8.9	2.1	6.8
Lignin	2.9	11.2	3.7	11.9
hemicellulose	1.5	5.7	2.0	6.5
Cellulose	1.6	6.3	1.5	4.9
Remaining solids	1.1	4.3	0.8	2.6
Total	25.3	98.4	32.2	104

Shells

Fraction	Phenyl-label		Pyridyl-label	
	mg eq/kg	% TRR	mg eq/kg	% TRR
80% ACN	0.42	54.6	1.89	43.9
Aqueous phase	0.34	44.1	0.89	20.7
Organic phase	0.081	10.5	1.0	23.2
Fluazinam	-	-	0.4	9.3
unidentified	0.42	54.6	1.49	34.6
PES	0.45	56.3	2.78	62.1
Phosphate buffer	0.06	6.9	0.20	4.4
Carbohydrates	0.03	3.5	0.08	1.8
Protein	0.02	2.5	0.05	1.2
Pectin	0.02	2.1	0.09	2.2
Lignin	0.06	7.5	0.52	12.2
hemicellulose	0.07	9.1	0.40	9.3
Cellulose	0.12	15.6	1.08	25.0
Remaining solids	0.07	9.1	0.36	6.0
Total	0.87	111	4.67	106

Nutmeats

Fraction	Phenyl-label		Pyridyl-label	
	mg eq/kg	% TRR	mg eq/kg	% TRR
Hexane	0.37	51.3	0.64	54.3
ACN	0.016	2.13	0.014	1.2
Water	0.19	25.7	-	-
Sucrose	0.07	9.6	0.05	4.2
TFAA	0.28	38.4	-	-
Fatty acids	0.23	31.5	0.58	48.7
Glycerol	0.02	2.7	2.5	2.5
Unidentified	0.02	2.8	0.24	20.1
PES	0.27	38.4	0.52	44.5
Phosphate buffer	0.06	9.3	0.25	21.9
Carbohydrates	0.04	5.8	0.09	7.9
Protein	0.10	13.7	0.07	5.9
Pectin	<0.01	<1	0.01	1.0
Lignin	<0.01	<1	0.01	0.9

Fraction	Phenyl-label		Pyridyl-label	
	mg eq/kg	% TRR	mg eq/kg	% TRR
hemicellulose	<0.01	<1	0.02	1.4
Cellulose	0.06	8.2	0.12	10.2
Remaining solids	0.01	1.4	<0.01	<1
Total	0.85	118	1.17	100

In the nutmeats, fluazinam and structurally related compounds were not found. Treatment with acid, base and enzymes did not release any of these compounds. The peanut nutmeats were shown to have radioactivity reincorporated in sucrose (0.07 mg eq/kg for the phenyl-label and 0.05 mg eq/kg for the pyridyl-label) and fatty acids (0.23 mg eq/kg for the phenyl-label and 0.58 mg eq/kg for the pyridyl-label). These levels represent approximately 5 to 10% (sucrose) and 30 to 50% (fatty acids) of the TRR. It was also established that approximately 10% of the radioactivity in nutmeats (both labels) was present in a molecular weight fraction greater than 10,000. This suggests that ^{14}C from fluazinam was incorporated into natural macromolecules such as proteins. Taken together, these results establish that ^{14}C from both labels was broken down into CO_2 or other small molecules that could enter the carbon pool and be reincorporated into natural products.

Further evidence of the complete fragmentation of the phenyl ring was seen in the observation of a trifluoroacetate signal in a 19F NMR of peanut oil (phenyl-label).

The peanut foliage from the phenyl treatment was found to contain fluazinam at 1.9 mg eq/kg (7.4% of the TRR) and AMPA at 0.4 mg eq/kg (1.6% of the TRR). For the pyridyl treatment fluazinam was at 2.3 mg eq/kg (7.5% of the TRR) and AMPA at 0.24 mg eq/kg (0.8% of the TRR).

The aqueous fraction from the phenyl label foliage contained TFAA at 0.87 mg eq/kg (3.4% of the TRR). The extractable fractions from peanut foliage were examined under a variety of HPLC conditions. These analyses demonstrated the complex, multi-component nature of the extractable fractions.

In the case of the peanut shells, fluazinam was identified at a level of 0.4 mg eq/kg (9.3% of the TRR) following treatment with the pyridyl label. Fluazinam was not identified in the shells following treatment with the phenyl label.

The metabolism of fluazinam in peanuts showed extensive degradation and re-incorporation of the radioactivity into natural products.

In nutmeats, neither fluazinam nor any structurally related metabolites containing the phenyl-pyridyl ring were present in detectable amounts. The radioactivity was found to have been incorporated into sucrose, fatty acids and proteins.

In foliage, fluazinam was detected at levels from 1.8 to 2.3 mg eq/kg. The reduction metabolite AMPA was also detectable at levels from 0.24 to 0.4 mg eq/kg (0.8–1.6% of the TRR). The remaining radioactivity consisted of multiple components, including TFAA, indicating that extensive degradation of the fluazinam molecule had occurred.

In peanut shells only fluazinam was found at detectable levels (0.04 mg eq/kg, 9.3% of the TRR). No other residues were identified.

The proposed metabolic pathway for fluazinam in peanuts is outlined in Figure 5.

Figure 5 Proposed metabolic pathway of fluazinam in peanuts

In Figure 6 an overall proposal for the metabolic pathway of fluazinam in plants is outlined.

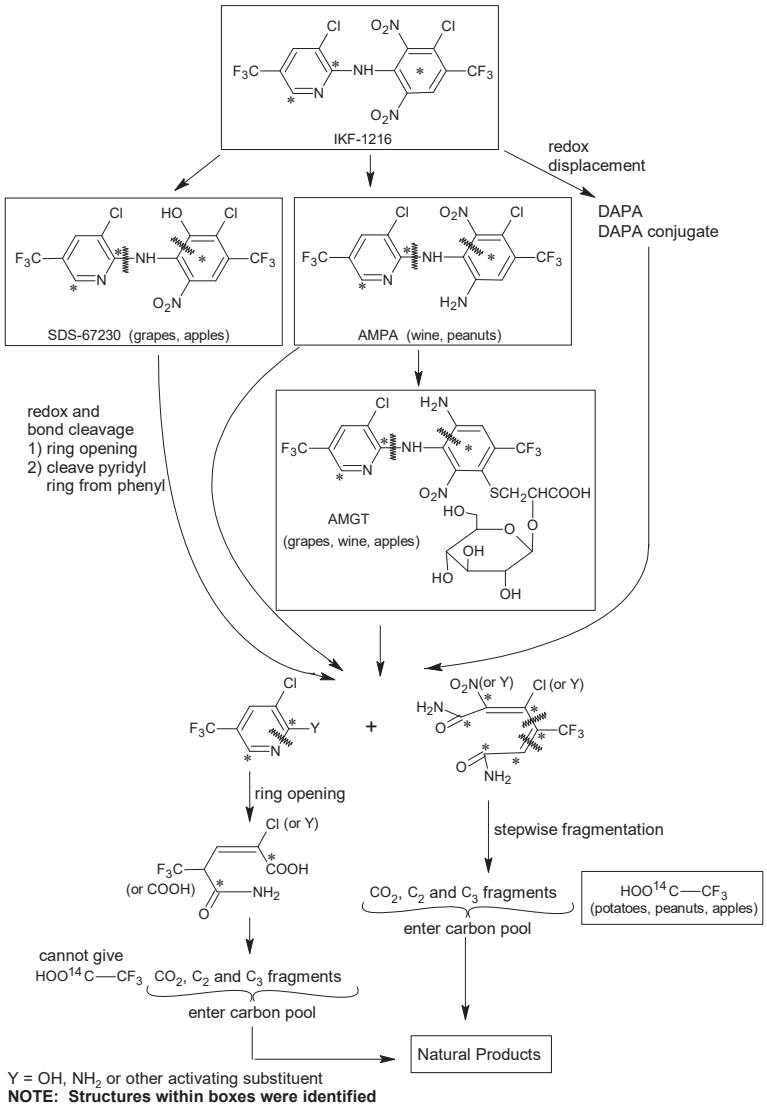


Figure 6 Proposed metabolic pathway of fluazinam in plants

Animal metabolism

The meeting received information on metabolism of fluazinam in ruminants (lactating goat) and poultry (laying hens). Fluazinam was either labelled in the phenyl or pyridine ring.

Lactating goat (Cheng, T. 1993)

Lactating goats (Alpine, Toggenberg or Nubian) were orally dosed with either phenyl or pyridyl-labelled fluazinam once a day for four consecutive days at a nominal rate of 20 mg/animal/day (actual dose rates were 19.9 mg/animal/day equivalent to 13.4 ppm feed (as received) for the phenyl label and 19.5 mg/animal/day equivalent to 9.14 ppm feed (as received) for the pyridyl label).

Milk, urine and faeces were collected daily. The animals were sacrificed approximately 23 hours after administration of the last dose and samples of liver, kidney, fat, muscle, blood, bile, gastrointestinal (GI) tract, and GI contents were taken.

Samples were homogenised, analysed by LSC or LSC following combustion to determine the total radioactivity content.

Samples of the kidney, liver and muscle were extracted with acetonitrile: water (1:1, v/v) and centrifuged; the resulting supernatant was partitioned with saturated aqueous sodium chloride solution and acetonitrile. The organic phase was concentrated

using nitrogen. The aqueous phase was lyophilised to dryness and the residues extracted (3×) with methanol containing 1% trichloroacetic acid. The combined extracts were concentrated under a nitrogen stream.

Fat samples were homogenised with acetonitrile: water (1:1, v/v) and hexane. The acetonitrile: water extracts were then partitioned with saturated sodium chloride solution. Further extractions were then undertaken with ethanol/hexane/water with heating. After cooling to room temperature, the samples were filtered. Nitrogen streams were used to evaporate the upper hexane phase. The suspensions that formed were extracted three times with ethanol and the combined extracts were analysed.

Milk was extracted with acetonitrile and the pellet further extracted with acetonitrile: water (1:1, v/v). The combined extracts were separated into aqueous and organic phases with saturated aqueous sodium chloride. The organic phase was concentrated before analysis. The aqueous phase was lyophilised and extracted (3×) with methanol containing 1% trichloroacetic acid. The combined methanol extracts were concentrated by nitrogen stream and analysed.

Metabolites were isolated from urine by extracting with ethyl acetate and concentrating under vacuum. The organic phase was reduced in volume under vacuum until two phases formed. The aqueous phase was discarded. The organic phase was mixed with isopropanol and concentrated under vacuum until the organic solvent was removed. Metabolites were eluted by HPLC on a reverse-phase column using acetonitrile. Urine was also enzyme-treated with either protease or β-glucuronidase/sulfatase and extracted with methanol containing 1% diethyl amine.

Extracts were analysed by LSC to determine the level of radioactivity present. Fractions containing the largest amount of radioactivity were analysed by 2D-TLC and HPLC to determine the metabolic profile. Metabolites were identified by comparing reference standards to the examined extracts. Mass spectrometry and NMR were also used to obtain spectral data on the isolated excreta metabolites.

All samples were stored at -20 ° C with the analysis all being conducted within 6 months. The stability of the residues in selected samples of liver and milk were examined by the comparison of metabolic profiles; the metabolic profile observed in the initial analysis and after freezer storage for 4-7 months was similar.

The TRR in in mg eq/kg and as a percentage of the total dose applied is shown in Table 17.

Table 17 Radioactive residues in ruminant tissues from lactating goats administered ^{14}C-phenyl or ^{14}C-pyridyl-fluazinam

Sample	Phenyl Label		Pyridyl Label	
	TRR (mg eq/kg)	% of total dose applied	TRR (mg eq/kg)	% of total dose applied
Liver	0.470	0.62	0.852	1.24
Kidney	0.034	<0.01	0.060	0.01
Muscle	0.035	0.05	0.025	0.04
Fat	0.160	0.23	0.262	0.36
Total in Tissues	0.699	0.90	1.199	1.65
Milk	0.018–0.071	0.31	0.018–0.078	0.59
Blood	0.015	<0.01	0.049	<0.01
Bile	4.660	0.08	2.901	0.16
GI tract	0.152	0.82	0.125	0.59
GI tract contents	n/a	9.04	n/a	10.51
Urine	n/a	8.91	n/a	11.55
Faeces	n/a	66.18	n/a	62.37
Total	-	86.24	-	87.42

The total recovery of radioactivity in the samples collected was 86.2% (phenyl-label) and 87.4% (pyridyl-label) of the total radioactivity administered. Radioactivity recovered in faeces accounted for 62–66% of the total radioactivity administered. The urine (cage wash and cage wipe included) contained 8.9% (phenyl-label) and 11.6% (pyridyl-label) of the total radioactivity administered. Of the edible tissues, the highest percentage of radioactivity was found in the liver (0.62 and 1.24% for the phenyl and pyridyl labels respectively).

The entire milk production for the phenyl-label and pyridyl-label contained 0.31% and 0.59% of the total radioactivity administered. The levels found in the milk at each milking interval are shown in Table 18 and Figure 7.

Table 18 Daily radioactivity concentrations in milk from lactating goats administered ^{14}C-phenyl or ^{14}C-pyridyl-fluazinam

Collection Day	Phenyl Label		Pyridyl Label	
	TRR (mg/kg)	% of total dose applied	TRR (mg/kg)	% of total dose applied
Day 1 p.m.	0.046	0.04	0.060	0.08
Day 2 a.m.	0.018	0.02	0.018	0.04

Collection Day	Phenyl Label		Pyridyl Label	
	TRR (mg/kg)	% of total dose applied	TRR (mg/kg)	% of total dose applied
Day 2 p.m.	0.048	0.04	0.070	0.11
Day 3 a.m.	0.021	0.03	0.021	0.04
Day 3 p.m.	0.062	0.06	0.071	0.11
Day 4 a.m.	0.020	0.02	0.022	0.05
Day 4 p.m.	0.071	0.06	0.078	0.10
Sacrifice (Day 4)	0.032	0.04	0.028	0.06
Total	-	0.31	-	0.59

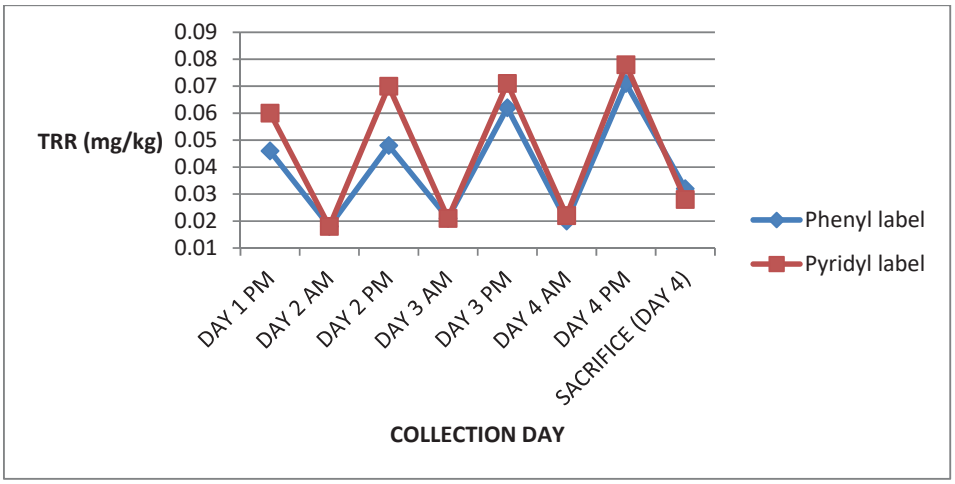


Figure 7 Daily radioactivity concentrations in milk

The distribution of the radioactivity in tissues is shown in Table 19.

Table 19 Distribution of radioactivity in extractable and unextractable fractions in samples from lactating goats dosed with ^{14}C-phenyl or^{14}C-pyridyl-fluazinam

Sample	Aqueous Extracted		Organic Extracted		PES		Total (% TRR)
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	
Phenyl label							
Liver	0.048	10.3	0.082	17.5	0.302	64.2	92.0
Kidney	<0.01	20.1	0.013	39.5	0.012	35.4	95.0
Muscle	<0.01	4.4	0.013	37.6	0.014	39.7	81.7
Fat	<0.01	4.2	0.127	79.9	0.012	7.5	91.6
Milk	<0.01	3.8	0.058	83.8	<0.01	8.8	96.4
Bile	4.660	100	na	na	na	na	100
Urine	0.704	95.8	na	na	na	na	95.8
Pyridyl label							
Liver	0.083	9.8	0.141	16.5	0.497	58.3	84.6
Kidney	0.012	20.0	0.025	41.0	0.021	34.7	95.7
Muscle	<0.01	5.7	0.011	43.1	0.012	47.0	95.8
Fat	<0.01	2.6	0.195	74.3	0.012	4.5	81.4
Milk	<0.01	3.4	0.068	93.0	<0.01	3.0	99.4
Bile	2.901	100	na	na	na	na	100
Urine	0.909	93.3	na	na	na	na	93.3

na–not applicable

For liver 26–28% of the residue was extracted. For kidney 60–61% of the residue was extracted. For muscle 42–49% of the residue was extacted. For fat 77–84% of the residue was extracted, while for milk 88–96% of the residue was extracted.

The identification and distribution of metabolites is shown in Table 20.

Table 20 Identification of fluazinam and its metabolites in combined aqueous and organic extracts of samples from lactating goats dosed with ^{14}C-phenyl- or ^{14}C-pyridyl- fluazinam

Matrix	Aq-1 (DAPA conjugate)	I-A	DAPA Sulfamate (I)	DAPA Sulfamate (II)	AMPA Sulfamate (III)	DAPA (IV)	AMPA (V)	Total
	% TRR [mg eq/kg]							
Phenyl label								
Liver	3.6 [0.017]	nd	2.7 [0.013]	2.7 [0.013]	6.3 [0.030]	12.5 [0.059]	nd	27.8
Kidney	20.1 [<0.01]	nd	3.8 [<0.01]	6.5 [<0.01]	10.1 [<0.01]	15.3 [<0.01]	3.7 [<0.01]	59.6
Muscle	4.4 [<0.01]	nd	nd	nd	nd	17.5 [<0.01]	20.1 [<0.01]	42.0
Fat	nd	nd	nd	nd	nd	49.2 [0.078]	34.9 [0.055]	84.1
Milk	3.8 [<0.01]	nd	nd	4.2 [<0.01]	11.5 [<0.01]	30.3 [0.021]	37.9 [0.026]	87.6
Bile	nd	nd	84.6 [3.942]	nd	7.3 [0.340]	8.1 [0.378]	nd	100
Urine	nd	7.6 [0.056]	66.5 [0.489]	19.5 [0.143]	nd	2.1 [0.016]	nd	95.7
Pyridyl label								
Liver	nd	nd	1.5 [0.013]	3.1 [0.026]	5.5 [0.047]	8.7 [0.074]	7.5 [0.064]	26.3
Kidney	15.9 [0.010]	nd	2.2 [<0.01]	8.4 [<0.01]	19.0 [0.011]	8.8 [<0.01]	6.8 [<0.01]	61.0
Muscle	5.7 [<0.01]	nd	nd	nd	nd	16.8 [<0.01]	26.3 [<0.01]	48.8
Fat	nd	nd	nd	nd	nd	28.3 [0.074]	48.6 [0.126]	76.9
Milk	3.4 [<0.01]	nd	nd	2.1 [<0.01]	13.7 [0.01]	26.4 [0.019]	50.9 [0.037]	96.4
Bile	nd	nd	72.4 [2.100]	nd	12.6 [0.366]	15.0 [0.435]	nd	100
Urine	nd	7.9 [0.077]	63.3 [0.617]	19.6 [0.190]	nd	2.6 [0.025]	nd	93.3

nd = not detected

Parent fluazinam was not detected in any of the samples.

The residues in meat, milk and edible tissues mainly comprised of the metabolites AMPA (maximum levels 0.126 mg eq/kg in fat, 48.6% of the TRR) and DAPA (0.078 mg eq/kg in fat, 49.2% of the TRR) and their sulfamate conjugates.

The polar metabolite (Aq-1) that was present in liver, kidney, muscle and milk could be hydrolysed to DAPA using hydrochloric acid, and was therefore characterised as a DAPA conjugate. One metabolite designated I-A, was found only in the urine and appeared to also hydrolyse to DAPA upon acid hydrolysis. However, it was not further characterised as it was not present in tissues or milk.

As liver and kidney contained significant amounts of unextracted solids following the initial extractions, enzymatic hydrolysis was performed using protease, sulfatase and β-glucuronidase treatments.

In another experiment, the PES liver samples were subjected to strong acid hydrolysis (6 M HCl for 4 hours). The post-enzyme extraction and post acid hydrolysis radioactivity distributions are summarized in Tables 21 and 22 respectively.

Table 21 Distribution of radioactivity released from the PES of liver and kidney by enzymatic hydrolysis

Matrix	% TRR released by enzymatic treatment [mg eq/kg]	% TRR] remaining in solids [mg eq/kg]
Liver-PES	N/A	100 [0.302] [a]
Protease	79.7 [0.241]	23.9 [0.072]
Sulfatase	32.8 [0.099]	62.6 [0.189]

Matrix	% TRR released by enzymatic treatment [mg eq/kg]	% TRR] remaining in solids [mg eq/kg]
β -glucuronidase	31.1 [0.094]	72 [0.217]
Kidney -PES	N/A	100 [0.021] [b]
Protease	93.8 [0.020]	16.9 [<0.01]
Sulfatase	62.7 [0.013]	36.5 [<0.01]
β -glucuronidase	59.3 [0.012]	42.6 [<0.01]

[a] Sample from phenyl label

[b] Sample from pyridyl label

Table 22 Distribution of radioactivity released from the PES for liver by acid hydrolysis

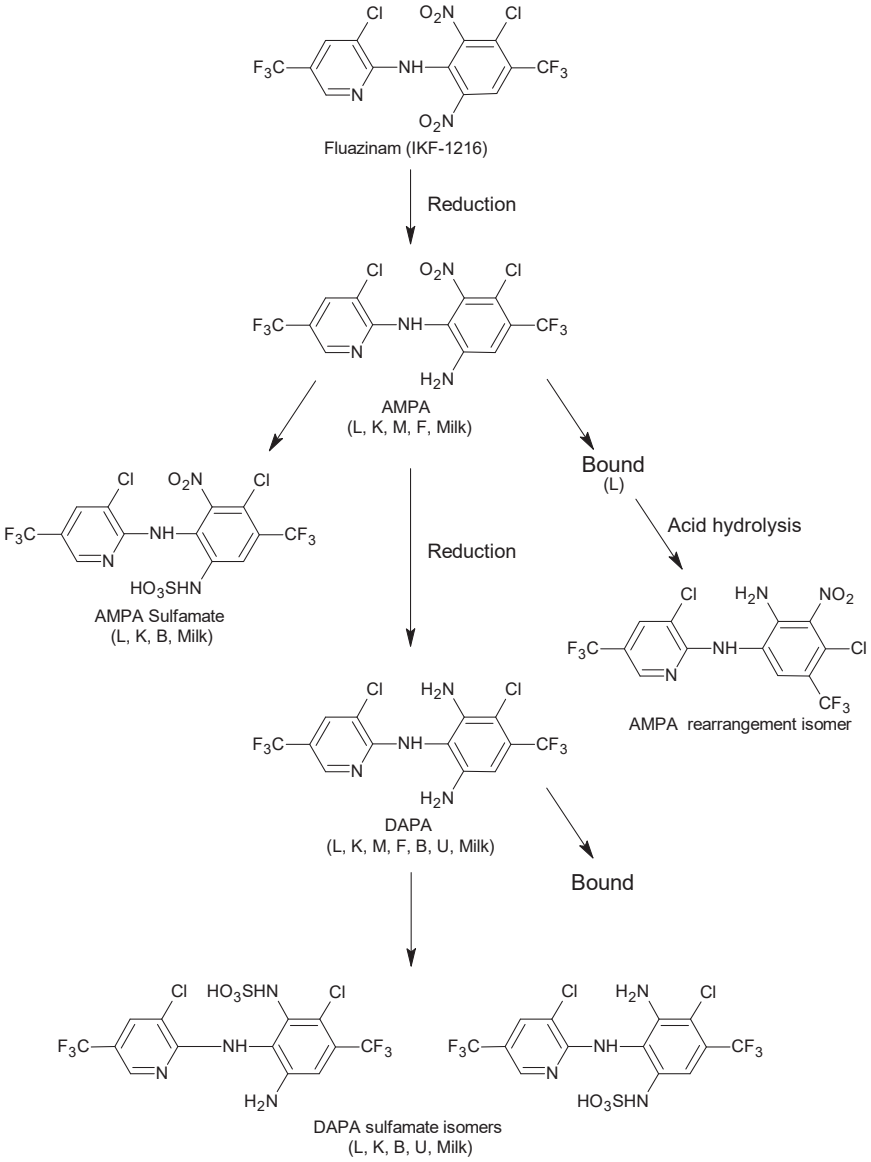
Matrix	% TRR [mg eq/kg]			
	PES	Aqueous	Organic	Remaining soilds
Liver (pheyl label)	100 [0.302]	14.5 [0.049]	86.5 [0.294]	3.2 [0.011]
Liver (pyridyl label)	100 [0.497]	17.5 [0.108]	39.9 [0.251]	3.6 [0.023]

For liver, 31 to 80% of bound radioactivity was released using the various enzyme treatments. In the case of kidney, 43 to 94% of bound radioactivity was released. For both liver and kidney the highest release of the bound residue occurred after treatment with protease indicating the radioactivity was associated with protein. No specific metabolites could be identified. However, the data indicated the absence of glucuronide and sulfate conjugates in the liver and kidney.

Following acid hydrolysis of the liver PES, the majority of the radioactivity was released into the organic extractable phase (up to 86.5% of the unextractable radioactivity). DAPA was a minor component, while the majority of the released radioactivity was a less polar metabolite which did not co-elute with any metabolite standard but was subsequently identified as a rearrangement product of AMPA. It was shown that AMPA could re-arrange to this isomer under acidic conditions. The levels of DAPA and the rearrangement product of AMPA released from the PES were not quantified.

The metabolism of fluazinam in lactating ruminants proceeds by reduction to give the metabolite AMPA, with further reduction to give the metabolite DAPA and conjugation of both AMPA to DAPA to sulfamate conjugates.

The proposed metabolic pathway is given in Figure 8.



(Letters in parentheses indicate matrix in which metabolite was identified:
L=Liver, K=Kidney, M=Muscle, F=Fat, B=Bile, U=Urine)

Figure 8 Proposed metabolic pathway of fluazinam in lactating ruminants

Poultry (Cheng, T. 1995)

Laying hens (white leghorn) were orally dosed by capsule with either the phenyl or pyridyl-labelled fluazinam once a day for four consecutive days at a nominal rate of 10 mg/kg of feed, as received (actual dose rates were 10.08 ppm equivalent to 0.764 mg/kg bw per day for the phenyl label and 10.62 ppm equivalent to 0.759 mg/kg bw per day for the pyridyl label).

The radiochemical purity and specific activity of the phenyl label were 98.2% and 91 419 dpm/µg and the radiochemical purity and specific activity for the pyridyl label were 97.6% and 93 991 dpm/µg.

Eight hens were dosed with the phenyl-labelled test material and seven with the pyridyl-labelled test material. Ten control animals received capsules that contained dextrose only.

Eggs and excreta were weighed and collected daily. Eggs collected from each day were separated in to yolks and white and pooled. The animals were sacrificed approximately 6 hours after administration of the last dose and samples of kidney, liver, fat, skin, thigh and breast muscle, gastrointestinal tract and contents, blood and any shelled eggs in the oviduct were taken.

Samples were homogenised, combusted and analysed by LSC. All tissue and egg samples were extracted sequentially with acetone (3×), methanol and methanol: water (1:1, v/v).

Following the initial solvent extractions, the PES from the liver were treated with protease, ß-glucuronidase, or sulfatase. After centrifugation, the pellet was extracted with methanol. Additional samples of the liver PES were treated with acid (HCl) and then extracted with methanol, acetone or ethyl acetate. Selected extracts were also subjected to acid hydrolysis in an attempt to characterise unknown polar components.

Metabolites present in the excreta were investigated to aid in the identification or characterization of metabolites found in tissues. Polar metabolite fractions were subjected to separation and clean-up using strong anion exchange (SAX) and C18 columns, followed by analysis using strong cation exchange HPLC-MS analysis.

Extracts were analysed by LSC. Fractions containing the largest amount of radioactivity were analysed by 2D-TLC and HPLC to determine the metabolic profile. Metabolites were identified by comparing reference standards to the examined extracts. Mass spectroscopy was also used to obtain spectral data on the isolated excreta metabolites.

Samples were all stored frozen (≤-18 °C) and analysed within 6 months. The metabolic profiles of selected samples of liver and hen were examined initially and after 4 months of storage. Changes in the metabolic profiles were observed which were most prominent in eggs and for the unidentified metabolites 10, 11 and 13.

The TRR in mg eq/kg and as a percentage of the total radioactivity applied are shown in Table 23 and 24.

Table 23 Radioactive residues in poultry tissues from laying hens administered ^{14}C-phenyl or ^{14}C-pyridyl-fluazinam

Sample	Phenyl Label		Pyridyl Label	
	TRR (mg eq/kg)	% of total dose applied	TRR (mg eq/kg)	% of total dose applied
Blood	0.392	0.14	0.215	0.08
Fat (abdominal)	0.936	0.57	0.959	0.49
Kidneys	0.438	0.11	0.349	0.09
Liver	1.047	0.92	0.920	0.88
Muscle (breast)	0.026	0.08	0.021	0.06
Muscle (thigh)	0.059	0.11	0.047	0.08
Skin with fat	0.493	0.45	0.581	0.58
Egg White	0.040	0.04	0.039	0.03
Egg Yolk	1.169	0.52	1.022	0.35
Total in Tissues	-	2.94	-	2.64
Excreta	na	101	na	99.1
Pan paper wash	na	0.70	na	0.49
GI tract and contents	na	11.1	na	11.9
Total	-	116	-	114

nd = not detected

Table 24 Daily radioactivity concentrations in eggs from laying hens administered ^{14}C-phenyl or ^{14}C-pyridyl-fluazinam

Collection Day	Phenyl Label				Pyridyl Label			
	Egg White		Egg Yolk		Egg White		Egg Yolk	
	TRR (mg eq/kg)	% of total dose applied	TRR (mg eq/kg)	% of total dose applied	TRR (mg eq/kg)	% of total dose applied	TRR (mg eq/kg)	% of total dose applied
Day 1	nd	nd	nd	nd	nd	nd	nd	nd
Day 2	0.003	<0.01	nd	nd	nd	nd	nd	nd
Day 3	0.016	<0.01	0.154	0.03	0.016	0.01	0.162	0.06
Day 4	0.027	0.02	0.598	0.19	0.029	0.01	0.680	0.13
Sacrifice (Day 4)	0.040	0.02	1.169	0.30	0.039	0.01	1.022	0.16
Total	0.086	0.04	1.921	0.52	0.084	0.03	1.864	0.35

nd = not detected

The majority of administered radioactivity was recovered in the excreta including the GI tract (113% for the phenyl label and 111% for the pyridyl label). Total egg production contained 0.38-0.56% of the applied dose.

Total residues in egg white and egg yolk were 0.040 mg eq/kg and 1.169 mg eq/kg respectively for the phenyl label, and 0.039 mg eq/kg and 1.022 mg eq/kg respectively for the pyridyl label.

Residues in tissues were highest in the liver, accounting for 1.047 and 0.920 mg eq/kg (0.92 and 0.88% of applied dose) for the phenyl and pyridyl labels respectively, and abdominal fat, accounting for 0.936 and 0.959 mg eq/kg, (0.57 and 0.4 9% of applied dose), respectively. There was no significant difference in the elimination and distribution of radioactivity between samples from hens administered the phenyl-labelled or pyridyl-labelled ^{14}C-fluazinam, indicating that cleavage did not occur.

The distribution of the radioactivity in tissues is shown in Table 25.

Table 25 Distribution of radioactivity in extractable and unextractable fractions in samples from laying hens dosed with ^{14}C-phenyl or ^{14}C-pyridyl-fluazinam

Sample	Acetone Extracted		Methanol Extracted		Methanol/water Extracted		PES		Total (% TRR)
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	
Phenyl label									
Liver	48.1	0.50	3.7	0.04	1.8	0.02	44.0	0.46	97.6
Kidney	62.0	0.27	5.4	0.02	2.1	<0.01	28.5	0.13	98.0
Muscle	59.6	0.04	6.9	<0.01	1.3	<0.01	31.4	0.02	99.2
Fat	111.5	1.04	1.1	0.01	0.1	<0.01	0.5	<0.01	113.2
Egg Yolk	73.7	0.86	2.4	0.03	0.8	<0.01	21.1	0.25	98.0
Egg White	92.7	0.04	5.1	<0.01	3.8	<0.01	7.6	<0.01	109.2
Excreta	37.3	-	7.8	-	9.5	-	-	-	-
Pyridyl label									
Liver	48.1	0.44	3.8	0.04	1.7	0.02	52.8	0.49	106.4
Kidney	63.4	0.22	5.5	0.02	1.9	<0.01	32.6	0.11	103.0
Muscle	57.4	0.03	8.4	<0.01	2.2	<0.01	32.1	0.02	100.1
Fat	100.9	0.97	1.1	0.01	0.1	<0.01	0.4	<0.01	102.5
Egg Yolk	74.7	0.76	3.2	0.03	0.8	<0.01	23.7	0.24	102.4
Egg White	93.1	0.04	4.4	<0.01	2.2	<0.01	3.4	<0.01	103.1
Excreta2	40.0	-	7.1	-	8.8	-	-	-	-

The total residue extracted was high for fat (100%), egg yolk (77–79%) and egg whites (100%). For liver 54% of the residue was extracted, for kidney 70–71% of the residue was extracted and for muscle 68% of the residue was extracted.

The identification and distribution of metabolites is shown in Tables 26 and 27.

Table 26 Distribution of fluazinam and its metabolites in samples from laying hens dosed with ^{14}C-phenyl-fluazinam

Sample	Liver % TRR [mg/kg]	Kidney % TRR [mg/kg]	Muscle % TRR [mg/kg]	Fat % TRR [mg/kg]	Egg White % TRR [mg/kg]	Egg Yolk % TRR [mg/kg]
Fluazinam	2.74 [0.027]	1.00 [<0.01]	1.13 [<0.01]	2.21 [0.02]	<1.00 [<0.01]	1.53 [0.018]
MAPA	2.50 [0.024]	1.61 [<0.01]	2.50 [<0.01]	8.84 [0.079]	3.43 [<0.01]	1.46 [0.018]
DAPA	3.17 [0.031]	1.98 [<0.01]	6.02 [<0.01]	5.90 [0.055]	4.54 [<0.01]	1.25 [0.015]
AMPA	13.1 [0.127]	18.0 [0.07]	32.4 [0.019]	81.9 [0.767]	48.5 [0.019]	6.06 [0.071]
Unknown 5	2.16 [0.021]	3.29 [0.013]	2.09 [<0.01]	2.95 [0.027]	6.21 [<0.01]	1.25 [0.015]
Unknown 6	1.39 [0.014]	3.35 [0.013]	nd	nd	17.4 [<0.01]	1.67 [0.020]
HYPA	4.86 [0.048]	3.16 [0.012]	5.60 [<0.01]	2.63 [0.024]	2.50 [<0.01]	5.30 [0.062]
Unknown 8	1.97 [0.015]	2.48 [0.010]	1.55 [<0.01]	nd	1.95 [<0.01]	2.96 [0.036]
Unknown 9	1.54 [0.015]	2.54 [0.010]	1.67 [<0.01]	<1.0 [<0.01]	1.67 [<0.01]	4.53 [0.053]

Sample	Liver	Kidney	Muscle	Fat	Egg White	Egg Yolk
	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]
Unknown 10	5.87 [0.057]	10.1 [0.039]	1.43 [<0.01]	<1.0 [<0.01]	4.45 [<0.01]	6.27 [0.074]
Unknown 11	2.74 [0.027]	4.28 [0.017]	3.22 [<0.01]	<1.0 [<0.01]	<1.0 [<0.01]	11.2 [0.131]
Unknown 12	4.79 [0.040]	8.87 [0.035]	0.66 [<0.01]	<1.0 [<0.01]	<1.0 [<0.01]	2.79 [0.033]
Unknown 13	<1.0 [<0.01]	<1.0 [<0.01]	1.31 [<0.01]	<1.0 [<0.01]	nd	20.0 [0.234]
Unknown 14	1.64 [0.016]	1.55 [<0.01]	nd	<1.0 [<0.01]	<1.0 [<0.01]	1.95 [0.023]
Total % TRR	48.4	61.2	60.6	104	90.7	68.2

nd = not detected

Table 27 Distribution of fluazinam and its metabolites in samples from laying hens dosed with ^{14}C-pyridyl-fluazinam

Sample	Liver	Kidney	Muscle	Fat	Egg White	Egg Yolk
	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]	% TRR [mg/kg]
Fluazinam	2.65 [0.022]	1.64 [<0.01]	<1.0 [<0.01]	2.10 [0.020]	<1.0 [<0.01]	<1.0 [<0.01]
MAPA	2.16 [0.018]	2.77 [<0.01]	2.35 [<0.01]	7.54 [0.072]	3.07 [<0.01]	1.55 [0.016]
DAPA	2.50 [0.021]	3.15 [<0.01]	6.20 [<0.01]	6.21 [0.059]	7.54 [<0.01]	2.83 [0.031]
AMPA	13.8 [0.115]	19.0 [0.055]	30.1 [0.014]	67.9 [0.651]	43.4 [0.017]	12.4 [0.127]
Unknown 5	2.74 [0.023]	3.28 [<0.01]	1.21 [<0.01]	1.81 [0.017]	9.12 [<0.01]	<1.0 [<0.01]
Unknown 6	2.41 [0.02]	3.4 [0.01]	nd	nd	19.1 [<0.01]	1.27 [0.013]
HYPA	4.95 [0.041]	3.84 [0.01]	5.91 [<0.01]	4.30 [0.041]	2.79 [<0.01]	3.81 [0.039]
Unknown 8	2.12 [0.017]	2.08 [<0.01]	1.84 [<0.01]	1.53 [0.014]	1.58 [<0.01]	4.09 [0.042]
Unknown 9	0.91 [<0.01]	1.70 [<0.01]	2.70 [<0.01]	2.29 [0.022]	1.68 [<0.01]	3.95 [0.040]
Unknown 10	5.44 [0.045]	10.5 [0.030]	3.56 [<0.01]	1.53 [0.014]	2.98 [<0.01]	8.39 [0.088]
Unknown 11	2.98 [0.025]	3.28 [<0.01]	<1.0 [<0.01]	<1.0 [<0.01]	<1.0 [<0.01]	9.02 [0.092]
Unknown 12	3.27 [0.027]	6.24 [0.018]	1.44 [<0.01]	<1.0 [<0.01]	nd	1.41 [0.014]
Unknown 13	<1.0 [<0.01]	<1.0 [<0.01]	<1.0 [<0.01]	nd	nd	17.6 [0.180]
Unknown 14	1.73 [0.014]	1.64 [<0.01]	<1.0 [<0.01]	nd	<1.0 [<0.01]	2.40 [0.025]
Total % TRR	47.7	59.5	55.3	95.2	91.3	68.7

nd = not detected

Parent fluazinam was only detected in small amounts, accounting for less than 3.0% of the TRR.

The main metabolite in muscle, fat, liver, egg white and egg yolk was identified as AMPA; from 6% of the TRR (0.071 mg eq/kg, egg yolk, phenyl label) up to 82% of the TRR (0.767 mg eq/kg, fat, phenyl label).

The metabolites MAPA and DAPA were also detected in all samples with levels ranging from 1.46% of the TRR in egg yolk (0.018 mg eq/kg) to 8.84% of the TRR in fat (0.079 mg eq/kg) for MAPA and from 1.25% of the TRR in egg yolk (0.015 mg eq/kg) to 7.54% of the TRR in egg white (<0.01 mg eq/kg) for DAPA.

HYPA was also identified and was generally present at lower levels, with a maximum of 5.91% of the TRR in muscle (<0.01 mg eq/kg).

The unknown polar metabolites, 10–13, found in egg and tissues were found to be unstable on storage for 120 days at -30 to -10 °C. The relative amounts of the polar metabolites changed over the storage interval and were identified as N-acetyl cysteine AMPA and the N-acetyl cysteine conjugate of fluazinam. Based on this it was tentatively proposed that the metabolites 10-13 are the initial/intermediate glutathione conjugates of AMPA and fluazinam which then undergo degradation on storage to produce N-acetyl cysteine AMPA and the N-acetyl cysteine conjugate of fluazinam.

The PES of the liver were subject to enzymatic and acid hydrolysis.

Samples of the PES were treated with protease, sulfatase and β-glucuronidase. The data indicates that approximately half the PES from liver was protein bound residues. Subsequent treatment of the sample with acid released AMPA and the acid re-arrangement product of AMPA. The exact levels of these two metabolites were not reported. The distribution of radioactivity released from the PES of liver following enzymatic hydrolysis is outlined in Table 28.

Table 28 Distribution of radioactivity released from the PES of liver by enzymatic hydrolysis

Matrix	% TRR released by enzymatic treatment [mg eq/kg]	% TRR] remaining in solids [mg eq/kg]
Liver–PES (phenyl label)	N/A	100 [0.46]
Protease	50 [0.23]	50 [0.23]
Sulfatase	0	100 [0.46]
β -glucuronidase	0	100 [0.46]
Liver–PES (pyridyl label)	N/A	100 [0.49]
Protease	50 [0.25]	50 [0.25]
Sulfatase	0	100 [0.46]
β -glucuronidase	0	100 [0.46]

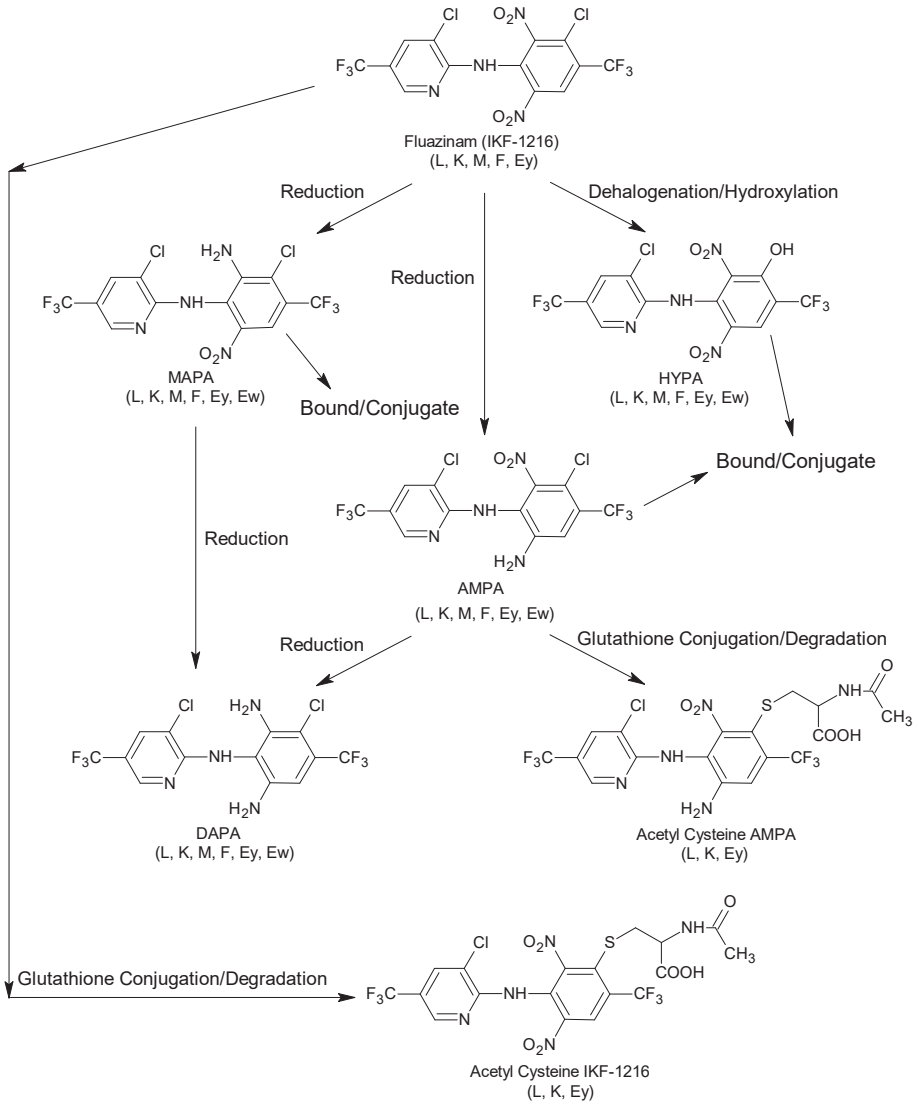
The liver PES were treated with HCL with increasing acid strength (successive treatment with 1 M, 6 M and 12 M HCl). The distribution of radioactivity in the PES following acid hydrolysis is outlined in table 29.

Table 29 Distribution of radioactivity released from the PES of liver by acid hydrolysis

Matrix	% TRR released by enzymatic treatment [mg eq/kg]	% TRR remaining in solids after sequential treatments [mg eq/kg]
Liver–PES (phenyl label)	N/A	100 [0.46]
1 M HCl	26.4 [0.12]	73.6 [0.34]
6 M HCl	34.7 [0.16]	38.9 [0.18]
12 M HCL	39.5 [0.18]	0
Liver–PES (pyridyl label)	N/A	100 [0.49]
1M HCl	22.2 [0.11]	77.8 [0.38]
2M HCl	28.6 [0.14]	49.2 [0.24]
12 M HCl	39.5 [0.19]	9.7 [0.05]

Acid hydrolysis of the liver PES released the majority of the bound radioactivity through use of increasing acid strength. The major components of the released radioactivity were identified as AMPA and the re-arrangement isomer of AMPA. At least two unknown polar components were also found. Based on a similar chromatographic behaviour to the polar components found in methanol extracts of excreta samples, these components were tentatively assigned as glutathione conjugates of fluazinam and AMPA. The exact levels were not reported.

The metabolism of fluazinam in laying poultry proceeds by reduction to give the metabolites AMPA and MAPA or by dehalogenisation/hydroxylation to give HYPA. AMPA and MAPA are further reduced to give the metabolite DAPA. The proposed metabolic pathway is given in Figure 9.



(Letters in parentheses indicate matrix in which metabolite was identified:
L=Liver, K=Kidney, M=Muscle, F=Fat, Ey=Egg yolk, Ew=Egg white)

Figure 9 Proposed metabolic pathway of fluazinam in laying poultry

Environmental fate in soil

The FAO Manual on the Submission and Evaluation of Pesticide Residues Data for the Estimation of Maximum Residue Levels in Food and Feed (2016) explains the data requirements for studies of environmental fate. The focus should be on those aspects that are most relevant to MRL setting.

Confined rotational crop studies

The nature of the residue in rotational crops has been investigated in two studies.

In the first study (Roninson, R.A. and Hoffman, B.A, 1994) either phenyl (radiochemical purity 99.4%) or pyridyl-labelled fluazinam (radiochemical purity 98.8%) was applied to the bare soil. The application rate was approximately 2 × 1.12 kg ai/ha with an application interval of 28 days.

Rotational crops of barley, carrots and lettuce were planted 30, 120 and 365 days after the last application. Due to crop failure, the barley planted at 30 DAT in the plot treated with ^{14}C-phenyl -fluazinam was replanted at 68 DAT.

Samples of soil, at a depth of approximately 30.5 cm, were taken prior to application, immediately following each application, at each plant back interval and at harvest. Soil samples were subjected to homogenization and combustion analysis. Crops from the control and treated plots were harvested at the immature and mature stage. Crop samples were homogenised with dry ice and the TRR in the crop fractions determined by combustion.

Subsamples of each crop homogenate were extracted three times with methanol: acetone (1:1, v/v). The mixture was filtered after each extraction, and the extraction solvent was combined and concentrated under vacuum until only an aqueous fraction remained. The resulting sample was partitioned three times with dichloromethane. All liquid samples were analysed directly by LSC. The post-extraction solids (PES) were allowed to dry and were then subjected to combustion analysis.

Crops that were very dry (i.e. barley straw and grain) were hydrated by adding ~3 volumes per sample weight (v/w) of water and storing overnight at ~2 °C. The initial extraction with methanol: acetone (1:1, v/v) was increased from 2% v/w to 6% v/w to allow for further hydration and sample swelling. The remainder of the extraction proceeded as described above.

In the second study (Robinson, R.A and Hoffman, B.A, 1995) the nature of the radioactive residue was examined in more detail. Identification and characterisation work was undertaken in the second study included the identification of metabolites by GC-MS, co-chromatography by HPLC and LC-MS, and further analysis of the PES. Extensive sample clean up using preparative HPLC (using C18 and amino columns) and SPE columns were employed as part of this analysis.

The residues in soil are shown in Table 30.

Table 30 Residue levels of ^{14}C-fluazinam in soil cores after application of 2 ×1.12 kg a.i./ha to the bare soil

Plant back interval (days)	Stage of crop/days after planting	Radioactive residue [mg eq/kg] PYRIDYL LABEL		Radioactive residue [mg eq/kg] PHENYL LABEL	
		Soil depth 0-15 cm	Soil depth 15-35.5 cm	Soil depth 0-15 cm	Soil depth 15-35.5 cm
Pre application	---	n.d. [a]	n.d.	n.d.	n.d.
Application 1 (April)	---	0.260	<LOQ [b]	0.117	0.008
Application 2 (May)	---	0.590	<LOQ	0.608	0.009
30†	Planting	0.403	<LOQ	0.401	<LOQ
	lettuce immature / 68	0.643	<LOQ	0.372	<LOQ
	lettuce mature / 89	0.230	0.013	0.478	<LOQ
	carrot immature / 99	0.384	0.012	0.370	0.015
	carrot mature / 155	0.370	0.051	0.410	0.037
	barley forage / 68;99	0.321	<LOQ	0.378	0.018
	barley mature / 138;174 [c]	0.577	0.016	0.542	0.076
120	Planting	0.583	<LOQ	0.782	0.026
		0.309	0.052	0.305	0.024
		0.286	<LOQ	0.350	0.016
	At various crop stages sampled and shipped, but no analytical data provided.	---	---	---	---
365	At planting and at various crop stages sampled and shipped, but no analytical data provided.	No data	No data	No data	No data

[a] not detected

[b] LOQ: The limit of quantitation for soil sample oxidation analysis ranged from 0.005 to 0.008 mg eq/kg.

[c] For the phenyl label the plant back interval investigated was 68 days for barley as the crop had to be replanted due to crop failure. 138 DAT represents the harvest of the crop from the pyridyl label and 174 DAT represents the harvest of the crop for the phenyl label.

The TRR determined in the upper soil layer 174 days after application were comparable to the TRR determined immediately after the second application. The data also demonstrates that only small amounts of residue were transferred to the lower soil layer.

The TRR values for the immature and mature crop samples of lettuce, carrots and barley at plant-back intervals of 30, 120 and 365 DAT are summarised in Table 31.

Table 31 Total radioactive residue (TRR) in rotated crops after application of ^{14}C-phenyl and ^{14}C-pyridyl-fluazinam to the bare soil

Crop	^{14}C-phenyl-fluazinam [mg eq/kg]			^{14}C-pyridyl-fluazinam [mg eq/kg]		
	30 day PBI	120 day PBI	360 day PBI	30 day PBI	120 day PBI	360 day PBI
Immature Lettuce	0.318	0.470	0.104	0.119	0.036	0.049
Mature Lettuce	0.282	0.174	0.040	0.065	0.034	0.039
Immature Carrot Roots	0.101	0.066	0.015	0.087	0.036	0.010
Immature Carrot Tops	0.429	0.164	0.056	0.333	0.045	0.059
Mature Carrot Roots	0.070	0.066	0.012	0.045	0.024	<0.010
Mature Carrot Tops	0.349	0.223	0.040	0.222	0.034	0.057
Barley Forage	0.135	0.934	0.529	0.327	0.075	0.138
Barley Grain	0.054	0.155	0.296	0.234	0.065	0.228
Barley Straw	0.093	0.256	0.273	1.249	0.105	0.266

The TRR generally decreased with time for lettuce and carrots at successive plant-back intervals. The TRR found in lettuce, from ^{14}C-pyridine-fluazinam treated soil, was consistently low with similar residues found at each of the plant-back intervals. The residue concentrations in barley grain appeared to increase with increasing plant-back time in the ^{14}C-phenyl-fluazinam treated plots. Similar TRR levels were observed in the barley grain from the 30 DAT and 365 DAT plant-back intervals in the ^{14}C-pyridine-fluazinam treated plots, but the levels at 120 DAT were markedly lower.

All the mature plant samples with a TRR above 0.01 mg/kg were subject to extraction. The results are shown in Table 32.

Table 32 Partitioning of fluazinam in Rotational Crop Fractions

Fraction	Plant-back Interval					
	30 DAT		120 DAT		365 DAT	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
^{14}C-Phenyl label						
Immature lettuce						
Organic	2.06	0.007	1.34	0.006	4.42	0.005
Aqueous	88.07	0.280	96.01	0.452	74.25	0.077
PES	9.87	0.031	2.65	0.012	21.33	0.022
Mature lettuce						
Organic	<0.01	<0.001	1.69	0.003	5.58	0.002
Aqueous	94.81	0.267	93.50	0.163	62.41	0.025
PES	5.19	0.015	4.81	0.008	32.01	0.013
Immature Carrot roots						
Organic						
Aqueous	12.36	0.013	6.72	0.004	8.79	0.001
PES	69.60	0.07	85.76	0.057	63.53	0.01
	18.04	0.018	7.52	0.005	27.68	0.004
Immature carrot tops						
Organic						
Aqueous	5.01	0.021	4.99	0.008	7.42	0.004
PES	81.48	0.35	75.95	0.125	43.34	0.024
	13.51	0.058	19.06	0.031	49.24	0.028
Mature Carrot roots						
Organic						
Aqueous	9.55	0.007	8.93	0.006	28.03	0.003
PES	82.37	0.057	78.29	0.052	48.21	0.006
	8.08	0.006	12.78	0.008	28.76	0.003
Mature carrot tops						
Organic						
Aqueous	2.63	0.009	2.41	0.005	10.58	0.004
PES	85.83	0.300	89.16	0.199	40.63	0.016
	11.54	0.040	8.43	0.019	48.79	0.020
Barley grain						
Organic	8.14	0.004	2.41	0.004	3.73	0.011
Aqueous	40.98	0.023	75.40	0.117	58.96	0.175
PES	50.88	0.027	22.19	0.034	37.31	0.110
Barley forage						

Fraction	Plant-back Interval					
	30 DAT		120 DAT		365 DAT	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Organic	25.78	0.035	2.23	0.021	6.96	0.037
Aqueous	53.74	0.072	94.26	0.880	77.30	0.409
PES	20.48	0.028	3.51	0.033	15.74	0.083
Barley straw						
Organic	8.06	0.007	2.65	0.007	6.00	0.016
Aqueous	57.84	0.054	82.58	0.211	59.54	0.163
PES	34.10	0.032	14.77	0.038	34.46	0.094
^{14}C-Pyridyl label						
Immature lettuce						
Organic	12.77	0.015	19.63	0.007	15.08	0.007
Aqueous	54.03	0.064	42.76	0.016	50.19	0.025
PES	33.20	0.040	28.97	0.010	34.73	0.017
Mature lettuce						
Organic	8.74	0.006	9.74	0.003	8.41	0.003
Aqueous	48.71	0.031	42.74	0.014	42.66	0.017
PES	42.55	0.028	37.83	0.013	48.93	0.019
Immature carrot roots						
Organic						
Aqueous	31.22	0.027	12.36	0.004	8.40	0.001
PES	40.64	0.036	62.61	0.023	62.94	0.006
	28.14	0.024	16.57	0.006	28.66	0.003
Immature carrot tops						
Organic						
Aqueous	16.40	0.055	22.54	0.01	10.58	0.006
PES	36.77	0.122	52.80	0.024	42.60	0.025
	46.83	0.156	24.66	0.011	46.82	0.028
Mature Carrot roots						
Organic						
Aqueous	14.11	0.006	14.06	0.003	Not extracted (TRR <0.010 mg/kg)	
PES	54.86	0.025	60.31	0.015		
	31.03	0.014	25.63	0.006		
Mature carrot tops						
Organic						
Aqueous	10.94	0.024	10.37	0.004	14.41	0.008
PES	34.72	0.077	47.00	0.016	34.33	0.020
	54.34	0.121	42.63	0.014	51.26	0.029
Barley grain						
Organic	3.70	0.009	4.43	0.003	5.00	0.011
Aqueous	5.10	0.012	27.02	0.017	19.83	0.046
PES	91.20	0.213	68.55	0.045	75.17	0.171
Barley forage						
Organic	11.48	0.037	15.32	0.011	6.39	0.009
Aqueous	62.32	0.204	61.95	0.046	61.55	0.085
PES	26.20	0.086	19.43	0.015	29.85	0.041
Barley straw						
Organic	6.50	0.081	6.67	0.007	6.00	0.016
Aqueous	50.31	0.629	34.69	0.037	51.34	0.137
PES	43.19	0.539	45.22	0.047	42.66	0.113

The organosoluble residues in the edible portions of crops used for human consumption were very low and generally below 0.01 mg/kg in mature crops, except 365 DAT barley grain where the residue was 0.011 mg/kg. Organosoluble residues from crop fractions used as animal feed items were more variable. The highest residues detected were in ^{14}C-pyridyl-labelled barley straw (0.081 mg/kg). However, the ^{14}C-phenyl-labelled barley straw from the same sampling time had organo-soluble residues below 0.01 mg/kg, indicating that the residues present in the ^{14}C-pyridyl-labelled fraction were extensively degraded and no longer contained the fluazinam structural backbone. HPLC profiles of the organo-soluble fractions showed radioactive regions containing a multitude of peaks. The low levels of radioactivity in most crop fractions precluded extensive investigation. Parent fluazinam was not detected in any extract from any crop sample.

In general, aqueous extracts in crops grown in ^{14}C-phenyl-labelled soils differed significantly from those in ^{14}C-pyridyl-labelled soils. HPLC profiles of the ^{14}C-phenyl-label extracts showed one main peak accounting for 60 to 100% of the fraction

(metabolite region A). The ^{14}C-pyridine-label did not contain this peak, but rather had two areas of radioactivity (metabolite region B and C). This difference in the profile between the two labels provided further evidence that the fluazinam phenyl-pyridyl ring structure has been cleaved and been extensively metabolised.

The metabolite distributions in aqueous extracts are shown in Tables 33– 38.

Table 33 Metabolite distribution for the aqueous lettuce extracts from ^{14}C]phenyl-fluazinam treated soil

Sample / Planting Interval	TFAA		Metabolite Region A[a]		Total[b]	
	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]
Immature Lettuce						
30 DAT	85.14	0.271	2.93	0.009	88.07	0.280
120 DAT	96.01	0.452	nd	nd	96.01	0.452
365 DAT	67.62	0.070	6.63	0.007	74.25	0.077
Mature Lettuce						
30 DAT	94.81	0.267	nd	nd	94.81	0.267
120 DAT	93.50	0.163	nd	nd	93.50	0.163
365 DAT	52.80	0.021	9.61	0.004	62.41	0.025

[a] HPLC analysis of the metabolite regions indicate the presence of more than one metabolite

[b] The total values may vary from the initial values due to rounding

nd: not detected

Table 34 Metabolite distribution for the aqueous lettuce extracts from ^{14}C-pyridyl-fluazinam treated soil

Sample / Planting Interval	Metabolite Region B[a]		Metabolite Region C[a]		Metabolite Region D[a]		Total[b]	
	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]
Immature Lettuce								
30 DAT	19.66	0.023	34.37	0.041	nd	nd	54.03	0.064
120 DAT	19.40	0.007	23.36	0.009	nd	nd	42.76	0.016
365 DAT	15.94	0.008	34.70	0.017	nd	nd	50.19	0.025
Mature Lettuce								
30 DAT	10.34	0.007	38.37	0.024	nd	nd	48.71	0.031
120 DAT	11.54	0.004	22.59	0.007	8.61	0.003	42.74	0.014
365 DAT	15.34	0.006	27.32	0.011	nd	nd	42.66	0.017

[a] HPLC analysis of the metabolite regions indicate the presence of multiple components

[b] The total values may vary from the initial values due to rounding

nd: not detected

Table 35 Metabolite distribution for the aqueous carrot extracts from ^{14}C-phenyl-fluazinam treated soil

Sample / Planting Interval	TFAA		Metabolite Region A[a]		Metabolite Region E[a]		Total[b]	
	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]
Immature Tops								
30 DAT	79.44	0.341	2.04	0.009	nd	nd	81.48	0.350
120 DAT	74.22	0.122	1.73	0.003	nd	nd	75.95	0.125
365 DAT	29.27	0.016	14.07	0.008	nd	nd	43.34	0.024
Immature Roots								
30 DAT	69.90	0.070	nd	nd	nd	nd	69.90	0.070
120 DAT	84.58	0.056	nd	nd	1.18	<0.001	85.76	0.056
365 DAT	nd	nd	nd	nd	nd	nd	nd	nd
Mature Tops								
30 DAT	85.83	0.300	nd	nd	nd	nd	85.83	0.300
120 DAT	87.39	0.195	1.77	0.004	nd	nd	89.16	0.199
365 DAT	25.14	0.010	11.83	0.005	3.65	0.001	40.62	0.016
Mature Roots								
30 DAT	82.37	0.057	nd	nd	nd	nd	82.37	0.057
120 DAT	73.39	0.049	4.90	0.003	nd	nd	78.92	0.052
365 DAT	35.48	0.004	4.73	<0.001	7.99	<0.001	48.20	0.004

[a] HPLC analysis of the metabolite regions indicate the presence of multiple components

[b] The total values may vary from the initial values due to rounding

nd: not detected

Table 36 Metabolite distribution for the aqueous carrot extracts from ^{14}C-pyridyl-fluazinam treated soil

Sample / Planting Interval	Metabolite Region B[a]		Metabolite Region C[a]		Metabolite Region D[a]		Total[b]	
	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]
Immature Tops								
30 DAT	20.11	0.067	16.66	0.055	nd	nd	36.77	0.122
120 DAT	16.11	0.007	36.69	0.017	nd	nd	52.80	0.024
365 DAT	12.46	0.007	30.14	0.018	nd	nd	42.60	0.025
Immature Roots								
30 DAT	40.64	0.036	nd	nd	nd	nd	40.46	0.036
120 DAT	15.41	0.006	47.20	0.017	nd	nd	62.61	0.023
365 DAT	20.03	0.002	42.91	0.004	nd	nd	62.94	0.006
Mature Tops								
30 DAT	17.36	0.039	17.36	0.039	nd	nd	34.72	0.078
120 DAT	13.98	0.005	33.02	0.011	nd	nd	47.00	0.016
365 DAT	7.84	0.005	19.77	0.012	6.71	0.004	34.32	0.021
Mature Roots								
30 DAT	17.96	0.008	36.90	0.017	nd	nd	54.86	0.025
120 DAT	13.44	0.003	46.87	0.012	nd	nd	60.31	0.015

[a] HPLC analysis of the metabolite regions indicate the presence of multiple components

[b] The total values may vary from the initial values due to rounding

nd: not detected

Table 37 Metabolite distribution for the aqueous barley extracts from ^{14}C-phenyl-fluazinam treated soil

Sample / Planting Interval	TFAA		Metabolite Region A[a]		Total[b]	
	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]
Grain						
68 DAT	37.48	0.021	3.50	0.002	40.98	0.023
120 DAT	74.21	0.115	1.19	0.002	75.40	0.117
365 DAT	58.96	0.175	nd	nd	58.96	0.175
Straw						
68 DAT	47.57	0.044	10.27	0.010	57.84	0.054
120 DAT	79.71	0.204	2.87	0.007	82.58	0.211
365 DAT	43.50	0.119	16.04	0.044	59.84	0.163
Forage						
30 DAT	46.77	0.063	6.97	0.009	53.74	0.072
120 DAT	94.26	0.880	nd	nd	94.26	0.880
365 DAT	70.21	0.371	7.09	0.038	77.30	0.409

[a] HPLC analysis of the metabolite regions indicate the presence of multiple components

[b] The total values may vary from the initial values due to rounding

nd: not detected

Table 38 Metabolite distribution for the aqueous barley extracts from ^{14}C-pyridyl-fluazinam treated soil

Sample / Planting Interval	Metabolite Region B[a]		Metabolite Region C[a]		Total[b]	
	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]
Grain						
30 DAT	3.45	0.008	1.65	0.004	5.10	0.012
120 DAT	13.98	0.009	13.13	0.008	27.02	0.017
365 DAT	10.49	0.024	9.34	0.022	19.83	0.046
Straw						
30 DAT	28.13	0.352	22.18	0.277	50.31	0.629
120 DAT	14.20	0.015	20.49	0.022	34.69	0.037

Sample / Planting Interval	Metabolite Region B[a]		Metabolite Region C[a]		Total[b]	
	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]	[% TRR]	[mg eq/kg]
365 DAT	25.27	0.067	26.07	0.070	51.34	0.137
Forage						
30 DAT	23.00	0.075	39.32	0.129	62.32	0.204
120 DAT	18.17	0.013	43.78	0.033	61.95	0.046
365 DAT	24.38	0.034	37.17	0.051	61.55	0.085

[a] HPLC analysis of the metabolite regions indicate the presence of multiple components

[b] The total values may vary from the initial values due to rounding

nd: not detected

Carrots

Residues were generally lowest in mature carrot roots. For the 365- DAT plantings, the TRR in carrot roots was <0.01 mg eq/kg for the ^{14}C -pyridyl-label, and the total extractable residues were <0.01 mg eq/kg for each fraction from the ^{14}C-phenyl-label. At the two earlier plant-back intervals, trifluoroacetic acid (TFAA) was the main residue from the phenyl label.

For the pyridyl-labelled treatment residues in the organo-soluble extract were <0.01 mg eq/kg for the 30 and 120 DAT plantings. In the aqueous extract the TRR was 0.025 mg eq/kg (60.3% TRR) and 0.015 mg eq/kg (54.9%) for the 30 and 120 DAT plantings respectively.

The HPLC analyses of pyridyl-label aqueous extracts gave radioactive regions similar in retention times to those from barley. Characterisation of a main region of the pyridyl-label aqueous extract from barley indicated that it contained components produced by extensive metabolism of the pyridine ring, including ring opening and fragmentation.

Lettuce

The residues in mature lettuce generally decreased for successive planting dates except for the ^{14}C-pyridyl-label where the TRR for the 120-DAT and 360-DAT samples had comparable levels.

For both labels at all plant-back times, the organo-extractable residues were <0.01 mg/kg.

The main residue in the aqueous extracts from the ^{14}C-phenyl-label was TFAA, ranging from 0.267 mg/kg (94.8% TRR) for the 30-DAT planting to 0.021 mg/kg (52.8% TRR) for the 365-DAT planting.

The aqueous-extractable residues for the pyridyl-label ranged from 0.014 mg/kg (41.2% TRR) for the 120-DAT planting to 0.031 mg/kg (47.7% TRR) for the 30-DAT planting. These extracts had similar HPLC profiles to those for barley straw, where extensive degradation and fragmentation of the pyridine ring had been found.

Barley Forage and Straw

The extracts of barley forage and straw from the early plantings were chosen for metabolite isolation and identification, since the highest levels of aqueous extractable residues were found in these samples. For the ^{14}C-phenyl-label, the main aqueous extractable residue was isolated and identified as TFAA.

With the [^{14}C]-pyridine-label, a main aqueous extractable region from HPLC was characterised by spectroscopic analyses and derivatisation. The LC-MS nd NMR analysis demonstrated that there were at least two components resulting from ring opening and fragmentation.

Organo-extractable residues were low in barley forage and straw. No fluazinam-related compounds $\geq$0.01 mg/kg were detected.

Barley Grain

The TRR for barley grain ranged from 0.054 to 0.296 mg/kg for the ^{14}C-phenyl-label and from 0.065 to 0.234 mg/kg for the ^{14}C-pyridine-label.

Organo-extractable residues were generally low for both labels, $\leq$0.011 mg/kg. The main residue present in the aqueous extracts from the phenyl label was TFAA, ranging from 0.021 mg/kg (37.5% TRR) for the 30-DAT planting to 0.175 mg/kg (59.0% TRR) for the 365-DAT planting.

The aqueous-extractable fractions from the pyridyl-label slowly increased from 0.012 mg/kg (5.1% TRR) of the 30-DAT planting to 0.046 mg/kg (19.8% TRR) of the 365-DAT planting. The HPLC analyses of these fractions indicated that these extracts had profiles similar to those for the barley straw, where extensive degradation and fragmentation of the pyridine ring had been found.

Analysis of the PES

The PES from the mature crops and barley forage of the 365 DAT plantings were subjected to enzyme hydrolysis with cellulase, acid hydrolysis (with HCL) and base hydrolysis (NaOH). The results of the enzymatic, acid and base hydrolysis experiments are outlined in Tables 39–45.

Table 39 Extraction distribution summary for release of bound residues from lettuce (365 DAT)

Fraction	Phenyl label	Pyridyl label
	% TRR [mg eq/kg]	% TRR [mg eq/kg]
TRR in PES following solvent extraction	32.01 [0.013]	48.93 [0.019]
Cellulase hydrolysis		
Aqueous fraction	11.47 [0.005]	13.67 [0.005]
Remaining solids 1	20.54 [0.008]	35.26 [0.014]
Acid hydrolysis		
HCl-1	9.24 [0.004]	14.11 [0.006]
HCl-3	1.67 [0.001]	6.89 [0.003]
Ether	2.14 [0.001]	4 [0.002]
Distillate	5.32 [0.002]	3.17 [0.001]
Precipitate	0.12 [<0.001]	0.05 [<0.001]
Remaining solids 2	11.30 [0.004]	21.15 [0.008]
HCl phase	4.66 [0.002]	-
Remaing solids	6.64 [0.002]	-
Base hydrolysis		
NaOH phase	-	14.06 [0.005]
Remaining solids	-	7.09 [0.003]

Table 40 Extraction distribution summary for release of bound residues from carrot roots (365 DAT)

Fraction	Phenyl label	Pyridyl label
	% TRR [mg eq/kg]	% TRR [mg eq/kg]
TRR in PES following solvent extraction	28.76 [0.003]	-
Cellulase hydrolysis		
Aqueous fraction	6.55 [0.001]	-
Remaining solids 1	22.21 [0.002]	-
Acid hydrolysis		
HCl-1	16.81 [0.002]	-
HCl-3	6.62 [0.001]	-
Ether	1.14 [<0.001]	-
Distillate	9.05 [0.001]	-
Precipitate	-	-
Remaining solids 2	5.40 [<0.001]	-
Acid phase	-	-
Remaining solids	-	-
Base hydrolysis		
NaOH phase	-	-
Remaining solids	-	-

- not analysed

Table 41 Extraction distribution summary for release of bound residues from carrot tops (365 DAT)

Fraction	Phenyl label	Pyridyl label
	% TRR [mg eq/kg]	% TRR [mg eq/kg]
TRR in PES following solvent extraction	48.79 [0.02]	51.26 [0.029]
Cellulase hydrolysis		
Aqueous fraction	17.09 [0.007]	17.41 [0.01]
Remaining solids	31.70 [0.013]	33.85 [0.019]
Acid hydrolysis		
HCl-1	17.79 [0.007]	14.11 [0.008]
HCl-3	6.46 [0.003]	8.05 [0.005]

Fraction	Phenyl label	Pyridyl label
	% TRR [mg eq/kg]	% TRR [mg eq/kg]
Ether	2.53 [0.001]	6.06 [0.003]
Distillate	3.47 [0.001]	-
Precipitate	5.32 [0.002]	-
Remaining solids 2	13.91 [0.006]	19.74 [0.011]
Acid phase	3.51 [0.002]	-
Remaining solids 3	10.4 [0.004]	-
Base hydrolysis		
NaOH phase	8.81 [0.003]	17.01 [0.009]
Remaining solids	1.59 [0.001]	2.73 [0.002]

- not analysed

Table 42 Extraction distribution summary for release of bound residues from barley grain (365 DAT)

Fraction	Phenyl label	Pyridyl label
	% TRR [mg eq/kg]	% TRR [mg eq/kg]
TRR in PES following solvent extraction	37.31 [0.110]	75.17 [0.171]
Cellulase hydrolysis		
Aqueous fraction	18.26 [0.054]	35.08 [0.08]
Remaining solids 1	19.05 [0.056]	40.09 [0.091]
Acid hydrolysis		
HCl-1	10.45 [0.031]	24.82 [0.056]
HCl-3	6.32 [0.019]	16.05 [0.036]
Ether	2.43 [0.007]	6.14 [0.014]
Distillate	1.61 [0.005]	2.23 [0.005]
Precipitate	0.09 [<0.001]	0.40 [0.001]
Remaining solids 2	8.60 [0.025]	15.27 [0.035]
Base hydrolysis		
NaOH phase	7.70[0.022]	12.79 [0.029]
Remaining solids	0.90 [0.003]	2.48 [0.006]

Table 43 Extraction distribution summary for release of bound residues from barley forage (365 DAT)

Fraction	Phenyl label	Pyridyl label
	% TRR [mg eq/kg]	% TRR [mg eq/kg]
TRR in PES following solvent extraction	15.74 [0.083]	29.85 [0.041]
Cellulase hydrolysis		
Aqueous fraction	7.87 [0.042]	9.88 [0.014]
Remaining soilds 1	7.87 [0.042]	19.97 [0.027]
Acid hydrolysis		
HCl-1	3.56 [0.019]	6.72 [0.009]
HCl-3	2.24 [0.012]	-
Ether	0.27 [0.001]	-
Distillate	1.04 [0.006]	-
Precipitate	-	-
Remaining soilds 2	4.31 [0.022]	13.25 [0.018]
Base hydrolysis		
NaOH phase	3.53 [0.018]	10.50 [0.014]
Remaining solids	0.78 [0.004]	2.75 [0.004]

- not analysed

Table 44 Extraction distribution summary for release of bound residues from barley straw (365 DAT)

Fraction	Phenyl label	Pyridyl label
	% TRR [mg eq/kg]	% TRR [mg eq/kg]
TRR in PES following solvent extraction	34.46 [0.094]	42.66 [0.113]
Cellulase hydrolysis		
Aqueous fraction	11.28 [0.031]	11.38 [0.03]
Remaining solids	23.18 [0.063]	31.28 [0.083]
Acid hydrolysis		
HCl-1	8.11 [0.063]	16.16 [0.043]
HCl-3	5.64 [0.015]	5.02 [0.013]
Ether	1.47 [0.004]	8.03 [0.021]
Distillate	1 [0.003]	1.90 [0.005]
Precipitate	-	1.21 [0.003]
Remaining solids 2	15.07 [0.041]	15.12 [0.04]
Base hydrolysis		
NaOH phase	11.21 [0.03]	13.62 [0.036]
Remaining solids	3.86 [0.011]	1.5 [0.004]

- not analysed

Table 45 Distribution of metabolites in the aqueous phase from the cellulase hydrolysis

Phenyl label

Sample	Metabolite region F‡			Metabolite region G‡			Totals		
	% by HPLC	% of TRR	mg eq/kg	% by HPLC	% of TRR	mg eq/kg	% by HPLC	% of TRR	mg eq/kg
Mature lettuce	100	11.47	0.005	ND	ND	ND	100	11.47	0.005
Mature carrot tops	100	6.55	0.001	ND	ND	ND	100	6.55	0.001
Mature carrot roots	54.5	9.31	0.004	45.5	7.78	0.003	100	17.09	0.007
Barley grain	88.89	16.23	0.048	11.11	2.03	0.006	100	18.26	0.054
Barley straw	38.52	4.35	0.012	61.48	6.93	0.019	100	11.28	0.031
Barley forage	65.61	5.16	0.028	34.39	2.71	0.014	100	7.87	0.042

Pyridyl label

Sample	Metabolite region H [a]			Metabolite region I [a]			Totals		
	% by HPLC	% of TRR	mg eq/kg	% by HPLC	% of TRR	mg eq/kg	% by HPLC	% of TRR	mg eq/kg
Mature lettuce	100	13.67	0.005	ND	ND	ND	100	13.67	0.005
Mature carrot tops	56.01	9.75	0.006	43.99	7.66	0.004	100	17.41	0.01
Barley grain	69.27	24.3	0.055	30.73	10.78	0.025	100	35.08	0.08
Barley straw	53.16	6.05	0.016	46.84	5.33	0.014	100	11.38	0.03
Barley forage	66.43	6.56	0.009	33.57	3.32	0.005	100	9.88	0.014

[a] HPLC analysis indicates more than one metabolite in the region

ND–not detected

Cellulase hydrolysis succeeded in releasing up to 51% of the PES. Analyses of the aqueous fractions from enzyme hydrolysis indicated two regions of radioactivity by HPLC analysis. Subsequent mild acid and strong base reactions succeeded in

releasing most of the unextractable residues. After base hydrolysis the resulting PES-fractions were all <10% of the TRR and <0.01 mg/kg, with the exception of phenyl-label barley straw where a TRR of 0.011 mg/kg was obtained.

In another experiment, the pyridyl-label barley grain PES fraction obtained from extraction with methanol/acetone (1:1, v/v) was treated with hot water to gelatinise any starch present, cooled and then incubated with α-amylase. HPLC analyses demonstrated that the α-amylase treatment released approximately 43% of the PES as glucose, maltose and other oligosaccharides. This mixture was then further treated by hydrolysis in 1M H_2SO_4 to convert maltose and the other oligosaccharides to glucose. The glucose was then reduced to sorbitol and acetylated to given sorbitol hexa-acetate. The identity of the sorbitol hexa-acetate was confirmed by NMR. Based on the amounts of degradation products formed, it was estimated that the barley grain PES contained 32% ^{14}C-labelled starch, derived from the re-incorporation of fluazinam residues.

Samples were stored at ≤-10 °C. To confirm the stability of the residues during the course of the investigations, an immature lettuce sample from the 30 DAT planting was re-extracted after more than two years of storage using identical extraction conditions to the original extraction. The distribution of radioactivity was very similar to the original extraction. Comparison of the HPLC chromatograms of the aqueous fractions from the re-extraction with those originally obtained were also very similar. In addition, re-analysis of the original aqueous fractions after two years of storage at ≤5 °C showed close similarity to the original. Since these storage intervals were longer than any periods encountered in the course of the laboratory phase of the studies, the stability of the residues has been addressed.

Field Rotational Crop Studies

No field rotation crop studies have been provided.

Environmental fate in soil

The meeting received information on aerobic degradation in soil, photolysis in soil and hydrolytic degradation. Information on the calculation of persistence, modelling of half-lives and formation fractions from laboratory soil degradation studies for fluazinam and its metabolite HYPA based on FOCUS kinetics were also provided. Only the data on degradation in soil, photolysis in soil and the hydrolytic degradation, which are relevant to MRL setting, are reported here.

Route of degradation in soil

Aerobic degradation in soil

Three studies have investigated the aerobic degradation of fluazinam.

The first study (Bharti, H and Bewick, D.W, 1985) was non-GLP. Fluazinam, labelled in the phenyl or pyridyl ring, was applied to two soils and degradation in the laboratory under aerobic conditions. The soils were characterised as a sandy loam and loamy sand.

Soil Characteristic	Sandy loam	Loamy sand
pH	6.9	6.4
% coarse sand	22	28
% fine sand	39	51
% silt	17	13
% clay	22	8
% organic matter	4.4	1.7
CEC (meq/100 g)	16	5

The soils were maintained at 40% moisture holding capacity and incubated at 20 °C in the dark. Fluazinam was applied to each soil type at a rate of 1 kg ai/ha or 5 kg ai/ha. Volatiles were collected in trapping solutions. In addition, samples were also prepared to cover sterilised soil. Samples of soil were removed for analysis from 7 to 361 DAT.

The soils were extracted with acetonitrile, filtered and the debris refluxed with acetonitrile for 3 hours. Extracted soil debris was analysed by combustion and LSC. Un-extracted soil residue was further characterised by refluxing the soil for 3 hours in 0.1M $Na_4P_2O_7$ and then partitioning the extract with a series of organic solvents. Analysis were carried out by TLC as well as HPLC and GC-MS.

The volatile radioactivity was almost entirely attributed to CO_2. In the two soils incubated aerobically for 361 days the level of CO_2 collected ranged from 1.8% to 6.3% of the AR. The level of CO_2 was similar for the two labels.

Anaerobic conditions reduced mineralization whereas mineralisation was negligible under sterile conditions. The amount of radioactivity extracted from the soil was >90% of the AR at the start of the study and gradually decreased over the incubation period, except for sterile soils. The un-extracted radioactivity reached 41.4% to 42.2% of the AR in the sandy loam soil and 26.1%

to 27.9% in the loamy sand soil after 361 days. The majority of the AR was extracted in the 1st extract with the second extraction procedure releasing up to another 20% of the AR.

The distribution of radioactivity in soil is outlined in Table 46 and the radioactivity identified is outlined in Table 47.

Table 46 Distribution of radioactivity

DAT	1st extract		2nd extract		NER		$^{14}CO_2$		Recovery	
	Phenyl-label	Pyridyl-label	Phenyl-label	Pyridyl-label	Phenyl-label	Pyridyl-label	Phenyl-label	Pyridyl-label	Phenyl-label	Pyridyl-label
Sandy loam _ aerobic–20 ° C–1 kg ai/ha										
0	88.7	101.5	na	na	4.0	1.5	na	na	92.6	103.0
7	83.1	79.0	na	4.5	12.4	9.1	0.1	<0.1	95.6	92.7
14	72.3	68.7	2.9	3.3	16.4	16.8	0.2	0.1	91.9	88.9
30	66.1	64.0	3.2	2.4	23.8	21.9	0.5	0.3	93.6	88.6
60	56.5	48.5	2.8	3.2	29.0	32.5	1.4	0.7	89.7	84.9
90	49.6	48.3	4.0	4.1	37.1	35.5	2.2	1.2	92.9	89.1
180	39.7	37.4	2.4	3.4	43.8	47.2	4.4	2.4	90.2	88.0
361	23.1	19.4	14.6	15.6	42.2	41.4	6.3	4.7	86.5	83.3
Sandy loam _ aerobic–20 ° C–*5 kg ai/ha*										
0	94.8	96.1	na	na	2.0	2.1	na	na	96.8	98.2
7	84.2	85.2	na	Na	9.8	10.8	0.1	<0.1	94.0	96.0
14	76.7	78.3	3.0	3.5	14.3	15.1	0.2	0.1	94.2	97.0
30	73.3	75.6	2.6	3.2	19.0	18.8	0.4	0.2	95.3	97.8
60	Na	Na	na	na	na	na	na	na	na	na
90	61.5	66.1	4.1	3.5	26.7	28.0	0.9	0.4	93.2	98.1
180	43.0	53.7	3.8	3.3	36.0	34.9	1.6	1.0	89.4	93.3
361	na	na	na	na	na	na	na	na	na	na
Sandy loam _ aerobic–20 ° C–1 kg ai/ha-*sterile*										
0	102.6	-	na	-	1.5	-	na	-	104.1	-
7	104.6	-	na	-	4.6	-	<0.1	-	109.1	-
14	96.8	-	3.7	-	5.6	-	<0.1	-	105.0	-
30	98.0	-	2.7	-	6.2	-	<0.1	-	106.9	-
60	na	-	na	-	na	-	na	-	na	-
90	na	-	na	-	na	-	na	-	na	-
180	na	-	na	-	na	-	na	-	na	-
361	na	-	na	-	na	-	na	-	na	-
Sandy loam _ aerobic–*10 ° C*–1 kg ai/ha										
0	99.0	96.1	na	na	2.2	2.2	na	na	101.2	98.3
7	91.1	82.3	na	na	6.8	6.5	<0.1	<0.1	97.9	88.7
14	96.8	82.3	3.2	2.1	11.2	9.3	<0.1	<0.1	101.2	93.9
30	82.7	81.1	2.2	2.1	13.4	13.0	0.1	<0.1	98.5	96.1
60	72.8	69.0	3.2	3.5	21.3	19.4	0.5	0.2	97.8	92.1
90	na	na	na	na	na	na	na	na	na	na
180	na	na	na	na	na	na	na	na	na	na
361	na	na	na	na	na	na	na	na	na	na
Loamy sand _ aerobic–20 ° C–1 kg ai/ha										
0	90.7	101.8	na	na	1.5	1.3	na	na	92.3	103.1
7	89.3	92.3	na	na	6.1	6.3	0.1	<0.1	95.4	98.7
14	86.2	86.7	2.1	2.4	7.1	7.4	0.2	0.1	95.6	96.5
30	84.2	82.9	1.9	2.1	26.4	9.8	0.4	0.1	112.9	94.9
60	na	na	na	na	na	na	na	na	na	na
90	72.6	69.6	3.4	3.8	16.7	15.8	1.2	0.4	93.9	89.6
180	51.7	57.6	2.0	2.8	21.2	25.4	2.7	1.3	78.0	88.5
361	38.2	35.3	18.6	19.6	27.9	26.1	3.5	1.8	88.2	84.0

Na not analysed

Table 47 radioactive residues in soil extracts

		Radioactive Residues [% of Applied]					
Treatment, Sample Day	Extract	Fluazinam	MAPA	DAPA	HYPA	Baseline	Others
Sandy loam soil/Aerobic/ 1kg ai/ha /20°/Phenyl-label							

		Radioactive Residues [% of Applied]					
Treatment, Sample Day	Extract	Fluazinam	MAPA	DAPA	HYPA	Baseline	Others
0	1	77.0	< 0.5	< 0.5	3.3	4.5	3.9
7	1	67.2	1.8	1.0	5.1	6.0	2.1
14	1	53.7	1.8	0.3	8.2	7.0	1.3
30	1	48.0	1.2	0.6	7.8	6.5	2.0
60	1	40.7	1.6	0.2	6.5	5.4	2.0
90	1	30.6	2.1	1.2	7.7	7.3	0.8
90	2	1.2	0.4	0.1	0.4	0.8	1.1
180	1	26.1	1.2	< 0.5	5.1	5.0	1.9
361	1	9.0	1.0	< 0.5	5.0	5.6	2.5
361	2	0.5	0.4	< 0.5	1.2	10.4	1.2
Sandy loam soil/Aerobic/ 1kg ai/ha /20º/Pyridyl-label							
0	1	88.9	< 0.5	< 0.5	3.2	5.2	4.3
7	1	58.2	< 0.5	< 0.5	6.1	9.3	5.4
14	1	51.2	1.9	0.9	8.0	5.5	1.2
30	1	46.7	0.8	0.8	6.4	5.5	3.7
60	1	25.0	1.0	1.3	7.7	10.7	2.8
90	1	33.2	1.5	1.5	6.5	4.6	0.8
90	2	1.3	0.4	0.1	0.6	0.5	1.1
180	1	20.2	0.1	< 0.5	8.2	4.9	2.2
180	2	1.0	0.3	< 0.5	1.1	0.4	0.5
361	1	6.4	1.1	< 0.5	5.3	3.8	2.9
361	2	0.4	0.5	< 0.5	1.7	9.7	1.6
Sandy loam soil/Aerobic/ 1kg ai/ha /20º/ Phenyl-label-Sterile							
0	1	99.8	<0.5	<0.5	<0.5	0.8	2.0
7	1	88.3	<0.5	<0.5	<0.5	9.6	6.7
14	1	95.2	<0.5	<0.5	<0.5	0.5	<0.5
30	1	92.1	<0.5	<0.5	<0.5	5.0	0.5
Sandy loam soil /Aerobic/ 1kg ai/ha /10º/ Phenyl-label							
0	1	90.7	<0.5	<0.5	1.6	3.2	3.5
7	1	80.3	0.5	1.2	3.7	5.0	0.4
14	1	79.8	<0.5	<0.5	2.1	2.0	2.8
30	1	67.8	<0.5	0.3	5.1	8.8	0.1
60	1	42.3	1.4	0.8	7.8	17.0	3.6
Sandy loam soil /Aerobic/ 1kg ai/ha /10º/ Pyridyl-label							
0	1	88.5	<0.5	1.0	1.7	2.6	2.3
7	1	72.3	<0.5	1.0	3.7	4.4	0.9
14	1	74.2	0.6	<0.5	2.4	2.6	2.6
30	1	66.1	0.5	0.3	4.0	6.5	2.1
60	1	54.1	1.0	<0.5	6.9	5.6	1.4
Loamy sand soil/ Aerobic/ 1kg ai/ha /20º/ Phenyl-label							
0	1	85.3	<0.5	<0.5	<0.5	1.8	3.7
7	1	72.2	1.5	0.8	5.9	6.3	2.6
14	1	66.5	<0.5	<0.5	3.6	3.6	12.6
30	1	77.5	<0.5	<0.5	2.1	3.2	<0.5
90	1	66.1	<0.5	<0.5	2.7	2.8	1.0
90	2	2.3	<0.5	<0.5	<0.5	<0.5	<0.5
180	1	45.3	<0.5	<0.5	2.9	2.0	1.5
361	1	28.8	0.5	<0.5	2.8	3.1	2.9
361	2	1.4	0.4	<0.5	1.8	13.1	1.6
Loamy sand soil /Aerobic/ 1kg ai/ha /20º/ Pyridyl-label							
0	1	97.0	<0.5	<0.5	<0.5	1.0	3.9
7	1	85.1	<0.5	1.0	3.1	3.6	<0.5
14	1	74.2	<0.5	<0.5	5.6	4.3	2.7
30	1	77.6	<0.5	<0.5	2.1	3.2	<0.5
90	1	63.0	<0.5	<0.5	3.0	1.8	1.9
90	2	2.4	<0.5	<0.5	<0.5	<0.5	<0.5
180	1	44.3	0.8	<0.5	4.8	5.1	2.7
361	1	23.9	0.6	<0.5	3.9	2.8	3.9
361	2	0.8	0.5	<0.5	1.6	13.8	2.0
Loamy sand soil /Aerobic/ 5kg ai/ha /20º/ Phenyl-label							

		Radioactive Residues [% of Applied]					
Treatment, Sample Day	Extract	Fluazinam	MAPA	DAPA	HYPA	Baseline	Others
0	1	91.3	< 0.5	0.5	0.8	0.9	1.3
7	1	72.4	1.4	<0.5	4.8	5.3	0.3
14	1	52.8	2.0	1.9	9.0	5.0	3.6
30	1	46.5	1.0	1.5	11.4	10.3	2.5
90	1	47.4	2.2	0.6	6.8	3.8	2.7
90	2	2.1	0.3	0.1	0.8	0.5	0.3
180	1	40.5	0.8	0.1	3.8	1.8	1.0
180	2	0.7	< 0.5	<0.5	1.2	0.4	1.0
Sandy loam soil/Aerobic/ 5kg ai/ha /20º/ Pyridyl-label							
0	1	91.3	<0.5	0.5	1.1	1.9	1.3
7	1	72.2	1.0	1.3	3.9	5.2	1.6
14	1	56.4	1.6	0.9	10.6	6.6	2.1
30	1	52.4	2.2	0.5	10.6	6.7	2.3
90	1	53.4	1.3	0.6	6.2	2.7	0.5
90	2	1.6	0.3	<0.5	0.8	0.5	0.2
180	1	39.8	0.6	0.3	8.1	3.8	1.1
180	2	0.8	0.4	0.1	1.0	0.4	0.6

The identity of the degradation products MAPA and HYPA were confirmed by mass spectrometry. The metabolite DAPA was identified only by co-chromatography. At the rate of 1 kg ai/ha, none of the metabolites were present at levels greater than 10% of applied fluazinam. HYPA was the only metabolite present at levels greater than 10% during the course of the study and only at the higher application rate of 5 kg ai/ha.

The main metabolic pathway is the formation of bound residues (up to 47.2% after 180 days under). Metabolites, which would indicate cleavage of the bridging amino group of fluazinam, were not found. Mineralisation (formation of CO_2) amounted for up to 6% AR after one year under standard conditions. Under aerobic conditions HYPA was the major metabolite which is formed by de-chlorination and subsequent hydroxylation of the phenyl ring of fluazinam. Reduced metabolism of fluazinam was observed at the lower temperature and for the higher application rate.

The half-time (DT_{50}) were determined using single first order kinetics (SFO), and where this was not a good fit they were determined using double first order in parallel kinetics (DFOP). The DT_{50} values determined are outlined in Table 48

The results of the sterilised control samples showed that the degradation of fluazinam was mainly microbiological.

Table 48 Summary of DT50 values for fluazinam and HYPA under aerobic conditions

Soil	DT_{50} (days)	
	Fluazinam	HYPA
Sandy loam (1 kg ai/ha, 20 °C)	17.83	257.21
Sandy loam (5 kg ai/ha, 20 °C)	55.6	165.8
Sandy loam (5 kg ai/ha, 10 °C)	60	-
Loamy sand	211.7	-

-Could not be calculated

The degradation rate of fluazinam was also investigated in a further study (Ryan, J and Sapiets, A, 1992) in which unlabelled fluazinam was applied at a rate of 750 g ai/ha to a standard Speyer 2.2 soil.

Soil type	Speyer 2.2
Sand content	87%
Silt content	7%
Clay content	6%
Organic matter content	3%
pH	5.4

The soil was maintained at 40% moisture holding capacity and incubated at 20 °C for 1 year in the dark. At intervals over the range of 0-364 days, soil samples were extracted with acetonitrile and analysed for fluazinam by GC-ECD. In parallel untreated control samples were established which were analysed at the same time points as the treated soil samples. Freshly fortified control

samples were also analysed and gave acceptable procedural recoveries. The results from the analysis of the samples are shown in Table 49.

Table 49 Fluazinam residues in soil

Sampling Interval (Days)	Fluazinam Residue	
	[mg/kg]	[% applied]
0	0.69	92
7	0.63	84
14	0.59	79
28	0.38	51
56	0.32	43
85	0.23	31
182	0.13	17
287	0.09	12
331	0.08	11
364	0.09	12

The half-time (DT_{50}) determined using DFOP kinetics resulted in a value of 42.9 days.

In the third study (Maward, N, 2003) a mixture of fluazinam labelled in the phenyl and pyridyl ring was applied to a sandy loam soil under laboratory conditions at a rate of 0.99 mg ai/kg dry soil (equivalent to 0.74 kg ai/ha if a soil depth of 5 cm and a soil density of 1.5 g/cm^3 are assumed).

Soil type	Sandy loan
Sand content	71.1%
Silt content	21.9%
Clay content	7 %
Organic matter content	1.1%
Maximum water holding capacity	54.4 g/100 g soil
Cation exchange capacity	6.9 meq/100 g
pH	7.1

The soils were maintained at 40% moisture holding capacity and incubated at 20 °C in the dark.

The flasks containing the soil samples were continuously ventilated with moistened air, and the outgoing air was passed through a trapping system designed to capture organic volatiles and CO_2. Individual soil samples were taken for analysis from 0 to 158 days after treatment.

Samples were extracted (methanol: phosphoric acid 99.5:0.5 v/v) and the extracts analysed by HPLC and/or TLC to characterise and identify the components. Residual soil was exhaustively extracted using Soxhlet extraction (either methanol: phosphoric acid 99.5:0.5 v/v or acetonitrile: water 4:1 v/v). The remaining soil was combusted to determine the amount of bound radioactivity. The microbial viability of the soil was determined prior to treatment and at 120 and 217 days of incubation.

Extraction with methanol/phosphoric acid up to four times, recovered the majority of extractable radioactivity from the soil sample. Soxhlet extraction contributed a maximum of 8.4% of the AR (day 70). The total extractable radioactivity steadily decreased over time to 55.2% AR on day 48. After this time point the total extractable radioactivity continued to decrease to 49.1% on day 70 and to 43.4% of the AR after 158 days.

The amount of unextractable radioactivity was high increasing from 3.7% of the AR on day 0 to 43-46% of the AR between 70 and 158 DAT.

The mineralization of fluazinam to CO_2 accounted for a maximum of 4.2% of the AR. Other volatile compounds collected did not exceed 0.4% of the AR. One major metabolite was detected which was characterized as HYPA. The maximum amount of HYPA (13.9% of the AR) was reached after 48 days of incubation. Up to 14 minor degradation products were detected with none of these individually exceeding 4.7% of the AR during the whole incubation period. The results are summarized in Table 50.

Table 50 Distribution of radioactivity (% AR) after aerobic incubation of fluazinam

DAT	Extractable	Unextacted	CO_2	Other volatiles	Recovery	Fluazinam	HYPA
0	96.0	3.7	NA	NA	99.7	96.0	ND
2	93.3	6.8	<0.1	<0.1	100.1	93.3	ND
7	84.1	13.2	0.3	<0.1	97.6	72.8	6.5
14	77.7	20.7	0.7	0.1	99.2	57.5	10.8
28[a]	58.9	35.0	2.0	0.3	96.2	26.3	9.1
48	55.2	38.8	3.3	0.2	97.5	16.4	13.9
70	49.1	43.0	4.2	0.4	96.8	7.8	11.3
120	44.2	46.4	2.7	0.1	93.3	4.5	10.6
158	43.3	43.4	3.3	0.4	90.6	2.9	8.3

[a] duplicate samples

NA not analysed

ND not detected

Fluazinam rapidly degraded in this sandy loam soil, having a DT_{50} of 16.6 days at 20 °C based on first order kinetics. The only metabolite formed in any significant amount was HYPA, which reached a maximum of 13.9% of the applied radioactivity at day 48. The calculated DT_{50} for HYPA was 109 days based on first order kinetics. All other extractable metabolites/degradates were minor, with none present above 5%. Mineralisation was a minor pathway, as carbon dioxide formation was limited to 4.2% of the applied radioactivity. Incorporation into bound soil matter was the major route of degradation, with 43.4% bound by the end of the study.

Photolysis–Soil

The photo-degradation of phenyl and pyridyl labelled fluazinam was investigated under laboratory conditions (Lentz, N.R and Korsch, B.H, 2001). The soil was a loamy sand soil.

Soil	% sand	% silt	% clay	% organic matter	pH	Cation exchange capacity (meq/100g)	Bulk density (g/cm^3)
Loamy sand	76.4	17.2	6.4	2.19	7.0	6.37	1.35

The test substance was applied at a rate of 3.55 and 3.32 mg/kg for the phenyl and pyridyl labels respectively. The soils were exposed to simulated sunlight (xenon arc lamp with filters) with a 12-hour light/12-hour dark cycle for 30 days at 25 ± 2 °C.

Volatiles were collected in trapping solutions. Duplicate soil samples were extracted 3 times with acetone: 0.1 M HCl (90:10 v/v). Attempts were made to release additional radioactivity from the post extracted solids of day 28 and day 30 samples. The PES were extracted with 0.1 M sodium pyrophosphate solution.

Light-exposed and dark control samples were analysed by radio-HPLC. Identification was established using GC-MS.

The photolysis half-lives on soil were 32.1 and 21.2 days for the phenyl ring labelled and the pyridyl ring labelled fluazinam, respectively. The half-lives in the dark controls were 68.6 days and 69.3 days. The rates of conversion of fluazinam to extractable degradation products, bound residues and to CO_2 were all more rapid for light-exposed soil than for the dark controls.

In the organic extractable fraction, fluazinam was found at a level of 35.7% (phenyl label) and 32.7% (pyridyl label) of the AR after 30 days. Two other components were identified by GC-MS. The largest component was identified as HYPA accounting for an average of 6.2% of the AR. AMPA was also identified in amounts of 4.3% and 5.1% of the AR for the phenyl and the pyridyl labels respectively after 30 days.

In the dark control samples fluazinam was present at 66.4% (phenyl label) and 71.4% (pyridyl label) of the AR after 30 days. HYPA in the dark control samples accounted for 4.9% (phenyl label) and 3.9% (pyridyl label) of the AR after 30 days. For the dark control samples AMPA represented less than 1% of the AR after 30 days.

Additionally one polar fraction and two other not identified minor fractions were found in the soil samples. In the light exposed samples these fractions amounted individually up to 2.5% of the AR. In the dark control samples these fractions amounted individually up to 0.7% of the AR.

The amount of bound residues accounted for 26.5% (phenyl label) and 16.8% (pyridyl label) of the AR after 30 days. In the dark control samples after 30 days the amounts were 10.8% (phenyl label) and 9.0% (pyridyl label) of the AR. By day 30 the

amount of CO_2 accounted for an average of 2.4% of the AR in the light-exposed samples and 0.2% of the AR in the dark control samples.

The distribution of the radioactivity after the application of fluazinam on soil is summarized in Table 51.

Table 51 Distribution of radioactivity (% AR) after application of fluazinam on soil (photolysis)

DAT	Extractable	Un-extractable	CO_2	Recovery	Fluazinam	HYPA	AMPA
Light							
0	98.8 / 99.3[a]	1.2 / 0.7	NA / NA	100.0	94.7 / 96.3	1.0 / 0.4	0.8 / 0.4
3	89.1 / 92.2	7.0 / 5.7	0.2 / 0.3	96.3 / 98.2	79.8 / 83.7	2.2 / 1.3	2.1 / 1.8
5	81.9 / 81.5	11.1 / 8.8	0.3 / 0.5	93.3 / 90.8	69.5 / 70.1	3.1 / 1.6	2.8 / 2.4
7	80.5 / 80.1	13.5 / 12.1	0.5 / 0.8	94.5 / 93.0	63.7 / 65.5	5.2 / 3.4	2.9 / 3.1
10	78.5 / 71.2	10.1 / 13.7	0.7 / 1.1	89.3 / 86.0	66.3 / 58.5	3.2 / 3.3	2.7 / 2.6
14	70.2 / 71.5	20.0 / 15.1	1.0 / 1.4	91.2 / 88.0	49.9 / 57.0	5.3 / 3.9	4.1 / 2.9
21	69.8 / 69.5	16.3 / 17.2	1.5 / 2.0	87.6 / 88.7	49.6 / 47.8	5.4 / 5.6	3.8 / 4.4
28	61.6 / NA	24.5 / NA	2.2 / NA	88.3 /NA	36.2 / NA	6.8 / NA	4.5 / NA
30	69.8 / 66.1	26.5 / 16.8	2.2 / 2.5	98.5 / 85.4	35.7 / 32.7	6.2 / 6.1	4.3 / 5.1
Dark							
0	98.8 / 99.3	1.2 / 0.7	NA / NA	100.0	94.7 / 96.3	1.0 / 0.4	0.8 / 0.4
3	87.0 / 99.0	3.5 / 4.0	0.1 / 0.0	90.6 / 103.0	82.6 / 94.6	1.3 / 1.0	0.8 / 0.5
5	91.1 / 94.1	4.9 / 4.6	0.1 / 0.1	96.1 / 98.7	86.0 / 89.8	1.5 / 1.1	0.8 / 0.5
7	90.0 / 94.4	4.8 / 5.0	0.1 / 0.1	94.9 / 99.5	85.2 / 89.9	1.8 / 1.3	0.7 / 0.5
10	88.8 / 82.0	6.3 / 6.9	0.2 / 0.1	95.3 / 89.0	82.3/ /77.1	2.6 / 1.8	0.9 / 0.6
14	74.2 / 83.1	7.2 / 7.5	0.2 / 0.1	81.6 / 90.7	86.4 / 77.9	2.3 / 1.7	0.8 / 0.7
21	83.2 / 85.8	10.5 / 8.1	0.3 / 0.1	94.0 / 94.0	75.0 / 78.3	4.1/ 3.3	0.9 / 0.8
28	77.5 / 79.2	9.6 / 8.3	0.3 / 0.1	87.4 / 87.6	68.7 / 71.9	4.5 / 3.5	0.9 / 0.6
30	78.5 / 78.4	9.0 / 10.8	0.3 / 0.1	87.8 / 89.3	66.4 / 71.4	4.9 / 3.9	0.9 / 0.7

Day 0 samples were set to 100%

[a] phenyl ring label / pyridyl ring label

NA Not analysed

Photolysis significantly increases the degradation rate of fluazinam on soil at 25 ± 2°C relative to the dark control samples. The half-lives for the dark controls averaged 69 days versus 22.2 days for the light exposed samples.

Under both light and dark conditions, conversion to bound residue was the main pathway for degradation of fluazinam. Conversion to bound residue, however, was more extensive for the light-exposed samples. In general, photolysis appears to accelerate reactions that also occur in soil under dark conditions.

The presence of HYPA at comparable levels in the dark control and the light-exposed samples suggests it is a product of soil metabolism. AMPA, however, is found in the light-exposed samples at levels of up to 5% versus levels of less than 1% in the dark control samples.

Photolysis–Aqueous Solution

The abiotic hydrolysis of phenyl and pyridyl labelled fluazinam (concentration: 0.04–0.05 mg/L) was investigated in sterile aqueous buffer solutions at pH 4, 7 and 9 (Van der Gaauw, A, 2003). The following conditions were investigated:

Experiment	pH	Temperature (°C)	Time (days)
1	4	50	5
2	7	25	29
3	7	50	56
4	9	25	29
5	9	50	56

During the incubation time, periodically the pH of each buffer solution was recorded and test samples were taken and analysed by LSC, HPLC and TLC.

All test solutions remained sterile and no significant variation of temperature and pH value was observed throughout the study. Mean recoveries of total radioactivity for both labels were between 95.8 ± 5.0% (pH 4, 50 °C) and 103.6 ± 2.4% (pH 7, 50 °C).

At pH 4 fluazinam was found to be hydrolytically stable.

At pH 7, fluazinam was rapidly hydrolysed. CAPA was the only hydrolysis product formed at 25 °C, representing 92.3% (phenyl label) and 95.1% (pyridyl label) of the applied radioactivity after 29 days. At 50 °C the major metabolite CAPA was steadily hydrolysed to DCPA with a DT_{50} value of about 32 days. At the end of incubation DCPA was found in amounts of 70.9% (phenyl label, day 56) and 38% (pyridyl label, day 28) of the applied radioactivity. Degradation of DCPA was not observed. For both labels, an additional minor hydrolysis product was detected at a maximum level of 5% of the AR on day 29.

At pH 9, hydrolysis of fluazinam was comparable to that observed at pH 7. CAPA was again the major hydrolysis product formed at 25 °C, representing 94.0% (phenyl label) and 102.6% (pyridyl label) of the applied radioactivity at the end of incubation (day 29). At 50 °C CAPA was steadily hydrolysed to DCPA with a DT_{50} value of about 8 days. DCPA represented 95.5% and 95.4% of the applied radioactivity for the phenyl label and pyridyl label, respectively, at day 29. No further degradation of this major metabolite was observed.

The balance and distribution of the radioactivity for the photolysis studies conducted at pH 7 and 9 and at 25 °C are summarized in Table 52.

Table 52 Balance and distribution of radioactivity in the buffer solutions (in percent AR) at 25 °C (phenyl label/pyridyl label)

Days	pH 7			Days	pH 9			
	Fluazinam	CAPA	Total		Fluazinam	CAPA	DCPA	Total
0	94.0 / 100.0	nd / nd	94.0 / 100.0	0	97.4 / 100.0	2.6 / nd	nd	100.0 / 100.0
2	55.5 / -	38.9 / -	94.4 / -	1	77.2 / 88.6	23.9 / 12.2	nd	101.1 / 100.9
5	40.9 / 27.5	57.6 / 72.3	98.5 / 99.9	2	69.5 / -	30.6 / -	nd	100.1 / -
10 / 15	31.4 / 5.2	64.5 / 94.5	96.0 / 99.6	5	36.8 / 39.7	63.0 / 62.0	nd	99.8 / 101.7
20	3.1 / -	93.9 / -	96.9 / -	20/15	4.3 / 6.5	96.5 / 94.7	nd	100.8 / 101.2
29	5.8 / 6.1	92.3 / 95.1	98.1 / 101.2	29	2.7 / nd	94.0 / 102.6	5.5	102.2 / 102.6

nd not detected

The half-time (DT_{50}) and DT_{90} values for fluazinam, calculated on the basis of first order kinetics, are shown in table 53.

Table 53 DT_{50} and DT_{90} values (days) for fluazinam hydrolysis at three different pH

	[^{14}C]-phenyl-fluazinam					[^{14}C]-pyridyl-fluazinam				
	pH 4	pH 7		pH 9		pH 4	pH 7		pH 9	
	50 °C	25 °C	50 °C	25 °C	50 °C	50 °C	25 °C	50 °C	25 °C	50 °C
DT_{50} [days]	Stable	4.5	0.1	3.5	0.2	Stable	2.7	0.2	3.9	0.1
DT_{90} [days]	Stable	14.8	0.4	11.6	0.6	Stable	9.1	0.6	13.0	0.3

RESIDUE ANALYSIS

Analytical methods

Data collection methods–plant commodities

Analytical method 1 (Apple Trials conducted in 1992-1996, grapes, bulb onion, broccoli, snap beans lima beans, peanut nutmeat and tea)

Residues of fluazinam, MAPA, CAPA and HYPA were extracted using methanol: acetic acid (100:2, v/v) followed by filtration. For fluazinam and MAPA the extract was acidified with HCl and partitioned with hexane. The hexane phase was partitioned with 0.2-0.5M NaOH. The aqueous phase was acidified and extracted with hexane. The hexane phase was concentrated and cleaned up using a Florisil column.

For CAPA and HYPA the initial extract was partitioned with chloroform. The organic phase was partitioned with 0.2 M NaOH. The aqueous phase was acidified and extracted with chloroform. The chloroform was evaporated, taken up in phosphoric acid: methanol (10:90, v/v) and residues methylated using diazomethane. Residues were partitioned into hexane and cleaned up using SEP-PAK Florisil columns.

Final determination was by GC-ECD.

Residues of AMGT were extracted using acetonitrile: water (4:1, v/v) followed by filtration. Aqueous sodium sulfate was added, extracts partitioned with methylene chloride, pH adjusted to 1 with HCl and partitioned twice with ethyl ether: ethyl acetate

(1:1, v/v). The organic phase was partitioned with 0.5% sodium carbonate. The organic phase was evaporated to near dryness, and re-dissolved in acetonitrile: water (32.5:67.5, v/v).

Final determination was by HPLC-UV with quantification at 254 nm.

Both methods were validated within the residue trial studies prior to sample analysis or with concurrent recoveries being analysed. The linearity of the detector response covered a working range of 0.005–0.05 µg/mL and 0.05–2 µg/mL for fluazinam and AMGT respectively. For MAPA, CAPA and HYPA the concentration ranged covered was not clear.

The specific LOQ validated for each analyte/commodity combination is reported with the residue trials.

The recovery data obtained from each study are summarised in Table 54.

Table 55 Recovery data for analytical method 1 used to determine residues of fluazinam and AMGT in apple, grapes, bulb onion, broccoli, snap beans, lima beans peanut nutmeat and tea.

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
Apple 5347-92-0245-CR-001 McFall, D.D. 1996a	Fluazinam	0.01	88, 83	83-88	86	-
		0.04	78	-	-	-
		0.1	88, 93	88-93	91	-
		0.2	90	-	-	-
		0.5	96	-	-	-
	AMGT	0.01	113	-	-	-
		0.2	82	-	-	-
		0.5	104	-	-	-
		1	97	-	-	-
Apple 5878-93-0345-CR-001 Fitzgerald, T.J. and McFall, D.D. 1995 and 5878-93-0345-CR-001 Fitzgerald, T.J. and McFall, D.D. 1995	Fluazinam	0.01	112, 82, 79, 71, 112, 110, 72	71-112	91	21
		0.1	101, 98	98-101	100	-
		0.2	85	-	-	-
		0.3	83	-	-	-
		0.4	83	-	-	-
		0.5	96	-	-	-
		1	1	-	-	-
	AMGT	0.01	70, 76	70-76	73	-
		0.025	92	-	-	-
		0.04	93	-	-	-
		0.05	82	-	-	-
		0.07	87	-	-	-
		0.2	109, 93	93-109	101	-
		0.25	85	-	-	-
		0.30	93	-	-	-
		0.40	95	-	-	-
		0.5	97, 110, 97	97-110	101	74
		1	82	-	-	-
Apple 6103-95-0025-CR-001 Fitzgerald, T.J. and McFall. D.D. 1996b	Fluazinam	0.01	86, 93, 94, 88, 112	86-112	95	11
		0.03	97	-	-	-
		0.04	95	-	-	-
		0.05	106	-	-	-
		0.1	102	-	-	-
		0.4	102	-	-	-
	AMGT	0.01	86	-	-	-
		0.02	95	-	-	-
		0.05	78	-	-	-
		0.06	95	-	-	-
		0.08	98	-	-	-
		0.1	91	-	-	-
		0.2	93, 93	93	93	-
		0.25	106	-	-	-
		0.40	95	-	-	-
		0.45	102	-	-	-

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
		0.50	91	-	-	-
		0.80	88	-	-	-
Grape 2127-91-0434-CR-001 McFall, D.D. 1996a	Fluazinam	0.01	110	-	-	-
		0.05	102	-	-	-
		0.1	95	-	-	-
		0.8	100	-	-	-
		1.0	118	-	-	-
		2.5	96	-	-	-
Grape 2106-91-0309-CR-001-001 Kenyon R.G. 1992a	Fluazinam	0.01	80, 108, 77, 100	77-108	91	17
		0.1	83	-	-	-
		0.2	95	-	-	-
		0.4	78	-	-	-
		0.8	84	-	-	-
		1.0	120	-	-	-
Grape 6245-95-0001-CR-001 Jablonski, J.E. 1995b	AGMT	0.01	100	-	-	-
		0.05	92	-	-	-
		0.1	76	-	-	-
		0.2	84	-	-	-
		0.5	86, 75	75-86	81	-
Bulb onion IR-4 PR No. 07092 Carpenter, D.H. 2008a	Fluazinam	0.01	115, 117, 110	110-115	114	3
		0.1	113, 113, 110	110-113	112	2
		1	113, 100, 111	100-113	108	7
Broccoli AAFC03-018 Ure G.B. 2006	Fluazinam	0.01	94, 99, 91	91–99	95	4
		0.02	88, 92, 73	73–92	84	12
		0.1	70, 70, 74	70-74	71	3
		0.01 (Concurrent)	95, 95, 75, 80, 82, 80	75–95	85	10
		0.1 (Concurrent)	62	-	-	-
Snap bean IR-4 PR No. 07602 Starner, V.R. 2006a	Fluazinam	0.02	105, 118, 119, 107, 105, 103	103-119	110	7
		0.1	102, 92, 107	92-107	100	7
		1	88, 87, 97	88-97	91	6
Lima beans IR-4 PR No. 08798 Starner V.R. 2006b	Fluazinam	0.02	71, 78, 81	71-81	77	7
		0.1	75, 79, 72	72-79	75	5
		1	84, 88, 93	84-93	88	5
Peanut nutmeat 5879-93-0335-CR-001 Hayes, P.C. Jr. and Kenyon, R.G. 1994	Fluazinam	0.01	88, 75, 91, 92	75–92	87	9
		0.05	72, 74, 82	72–82	76	7
		0.1	98	-	-	-
		1.0	107	-	-	-
Peanut hulls 5879-93-0335-CR-001 Hayes, P.C. Jr. and Kenyon, R.G. 1994	Fluazinam	0.01	78, 97, 91, 86	78-97	88	9
		0.03	80, 97	80-97	89	-
		0.05	88	-	-	-
		0.1	99	-	-	-
		1.0	92	-	-	-
Peanut hay 5879-93-0335-CR-001 Hayes, P.C. Jr. and Kenyon, R.G. 1994	Fluazinam	0.01	76, 96, 75, 87	75-96	83.5	11.9
		0.05	82	-	-	-
		0.1	87	-	-	-
		0.2	90, 75			
		0.5	68	-	-	-
		1.0	84, 98			

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
		2.0	89	-	-	-
Peanut nutmeat 2105-91-0307-CR-001 Kenyon, R.G. 1992b	Fluazinam	0.01	78, 90, 93, 119	78-119	95	18.2
		0.06	82	-	-	-
		0.1	84	-	-	-
		0.2	90	-	-	-
		0.3	90	-	-	-
		0.5	90	-	-	-
		1.0	95	-	-	-
Peanut nutmeat 6107-95-0013-CR-001 McFall, D.D. 1995	Fluazinam	0.01	105, 101	101 -105	103	-
		0.1	89, 76	76–89	83	11
		0.2	97	-	-	-
		0.4	97	-	-	-
		1.0	93	-	-	-
		0.01 (concurrent)	99, 100, 93	93–100	97	4
		0.1 (concurrent)	93, 91, 91	91-93	92	1
Peanut shells 6107-95-0013-CR-001 McFall, D.D. 1995	Fluazinam	0.01	111, 112	111-112	112	-
		0.1	108, 101	101–108	105	-
		0.2	100	-	-	-
		0.4	90	-	-	-
		1.0	107	-	-	-
		0.01 (concurrent)	108, 83, 120	83–120	104	19
		0.1 (concurrent)	110, 93, 108	93–110	104	9
Peanut hay 6107-95-0013-CR-001 McFall, D.D. 1995	Fluazinam	0.01	119, 94	94-119	107	-
		0.05	112	-	-	-
		0.5	100	-	-	-
		1.0	74, 78	74-78	76	-
		2.0	73	-	-	-
		5.0	75	-	-	-
		7.0	72	-	-	-
		10	68	-	-	-
		15	92	-	-	-
		0.01 (concurrent)	125, 108, 81	81–125	105	22
		0.05 (concurrent)	100, 98, 76	76-100	91	13
Tea Ohyama, J. 1993	Fluazinam	0.02	83, 81	81-83	82	-
		0.1	85, 70	70-85	78	-
	MAPA	0.02	78, 76	76-78	77	-
		0.1	79, 85	79-85	77	-
	HYPA	0.02	85, 83	83-85	84	-
		0.1	86, 73	73-86	80	-
Tea Kondo, K. 1997	Fluazinam	0.02	86, 82, 88, 80, 100, 92	82-100	88	7
		1	76, 73, 83, 80, 77, 75	73-83	77	4
	MAPA	0.02	100, 90, 83, 80, 89, 85	80 -100	88	7
		0.2	75, 71, 85, 82, 88, 84	71-88	81	7
	HYPA	0.02	77, 73, 76, 75, 76, 70	70-77	75	3
		0.2	75, 71, 72, 70, 74, 71	70-75	72	2
Tea Kato, S. 1987	Fluazinam	0.4	88, 88	88	88	-
		20	91, 93	92	91-93	-
	MAPA	0.8	71, 72	72	71-72	-
	HYPA	0.4	69, 73	71	69-73	-

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
	CAPA	0.8	76, 77	77	76-77	-
Tea Hagi, I. 1986	Fluazinam	0.02	84, 93	88	84-93	-
		0.2	87, 93	90	87-93	-
	MAPA	0.04	83, 89	86	83-89	-
		0.2	88, 94	91	88-94	-
	HYPA	0.02	82, 85	84	82-85	-
		0.2	83, 88	86	83-88	-
	CAPA	0.04	78, 85	82	78-85	-
		0.2	82, 84	83	82-84	-

Analytical method 2 (Apple Trials conducted in 2008)

Residues of fluazinam were extracted using methanol. The methanol extract was partitioned with 2M HCl followed by hexane. The hexane phase was partitioned with 5M NaOH. The alkaline layer was acidified to pH 1 and partitioned with hexane. Hexane extracts were evaporated to dryness and the residue re-dissolved in acetone. Final determination was by GC-ECD.

Residues of AMGT were extracted using methanol. The methanol extract was evaporated to near dryness and re-dissolved in 2% sodium sulfate that was partitioned with methylene chloride. The aqueous phase was acidified to pH 1 and partitioned with ethyl acetate: ethyl ether (1: 1, v/v). The organic layer was evaporated to dryness and dissolved in 30% aqueous acetonitrile. Final determination was by HPLC -UV with quantification at 254 nm.

Both methods were validated prior to the sample analysis. Concurrent recoveries ranging from 0.01–1 mg/kg, were also analysed with the samples. The linearity of the detector response covered a working range of 0.005–0.25 µg/mL and 0.005–1 µg/mL for fluazinam and AMGT respectively.

The LOQ validated for both Fluazinam and AMGT in apples was 0.01 mg/kg.

The recovery data obtained from each study are summarised in Table 55.

Table 55 Recovery data for analytical method 2 used to determine residues of fluazinam and AMGT in apples

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
Apple IB-2006-JLW-002-00-01 Wiedmann, J.L. 2008a	Fluazinam	0.01	121, 109, 97, 90	90-121	104	13
		0.1	126, 117	117-126	122	-
		1	100, 96	96-100	98	-
	AMGT	0.01	109, 55, 90	55-109	85	32
		0.1	100, 66	66-100	83	-
		1	100, 100	100	100	-

Analytical method 3 (Grape trials conducted in 1994-1997 and blueberries)

Residues of fluazinam were extracted using methanol: acetic acid (100:2, v/v) followed by filtration. The extract was acidified with HCl and partitioned with petroleum ether. The aqueous phase was discarded. The petroleum ether phase was evaporated to dryness, re-dissolved in hexane and cleaned up using a Florisil column. Final determination was by GC-ECD.

Residues of AMGT were extracted using acetonitrile: water (4:1, v/v) followed by filtration. Aqueous sodium sulphate was added, extracts partitioned with methylene chloride, pH adjusted to 1 with HCl and partitioned twice with ethyl ether: ethyl acetate (1:1, v/v). The organic phase was partitioned with 0.5% sodium carbonate. The aqueous phase was acidified to pH 1 and partitioned with ethyl ether: ethyl acetate (1:1, v/v). The organic phase was evaporated to near dryness, and either re-dissolved in methanol and cleaned up by solid phase extraction using ENVI-Carb columns or re-dissolved in water and cleaned-up using t-C18 Sep-Pak columns. Final determination was by HPLC-UV with quantification at 254 nm.

Both methods were validated within the residue trial studies prior to sample analysis or with concurrent recoveries being analysed. The linearity of the detector response covered a working range of 0.005–2 µg/mL and 0.05–2 µg/mL for fluazinam and AMGT respectively.

The specific LOQ validated for each analyte/commodity combination is reported with the residue trials.

The recovery data obtained from each study are summarised in Table 56.

Table 56 Recovery data for analytical method 3 used to determine residues of fluazinam and AMGT in grapes and blueberries

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
Grape 6106-95-0012-CR-001 Jablonski, J.E.1995a	Fluazinam	0.01	110, 110, 110	110	110	0
		0.02	105	-	-	-
		0.05	110, 112, 100	100 -112	107	6
		0.10	99, 96	96–99	98	-
		0.25	98	-	-	-
		0.50	111, 99	99–111	105	-
		1.0	123, 126, 102	102–128	118	12
		3.0	113	-	-	-
	AMGT	0.01	90, 100, 80	80–100	90	11
		0.02	60	-	-	-
		0.05	84, 74, 104	74–104	87	18
		0.10	72, 94, 71, 86	71–94	81	14
		0.20	92, 93	92-93	93	-
		0.50	84, 83, 80, 89, 82	80–89	84	4
Grape EA950132 Grolleau, G. and Kenyon, R.G. 1996 Validation	Fluazinam	0.01	80, 90	80–90	85	-
		0.1	110, 100	900–110	105	-
		1.0	112, 114	112–114	113	-
	AMGT	0.01	100, 100, 100, 100	100	100	0
		0.10	110, 87, 88	87–110	95	14
		1.0	101, 88, 68, 77	68–101	84	17
Grape EA950132 Grolleau, G. and Kenyon, R.G. 1996 Chilean trials	Fluazinam	0.01	100	-	-	-
		0.1	100	-	-	-
		0.5	120	-	-	-
		1.0	106	-	-	-
	AMGT	0.01	100, 100	100	100	-
		0.05	126, 110	110–126	118	-
		0.1	110	-	-	-
		0.5	86	-	-	-
Grape EA950132 Grolleau, G. and Kenyon, R.G. 1996 EU trials	Fluazinam	0.01	90, 100	90-100	95	-
		0.02	85	-	-	-
		0.05	88	-	-	-
		0.08	86	-	-	-
		0.1	99, 89	89–99	94	-
		0.15	87, 107	87–107	97	-
		0.2	80	-	-	-
		0.25	108	-	-	-
		0.50	100, 100	100	100	-
		0.75	96	-	-	-
		1.0	99	-	-	-
		1.5	111	-	-	-
		2.0	101	-	-	-
		2.5	101	-	-	-
		3.0	111	-	-	-
		5.0	109	-	-	-
	AMGT	0.01	80, 90	80-90	85	-
		0.03	67	-	-	-
		0.04	93	-	-	-
		0.05	90, 70	70-90	80	-
		0.07	97	-	-	-
		0.1	96, 90, 83, 90	83-96	90	6
		0.2	75	-	-	-
		0.4	83	-	-	-
		0.5	80, 94	80–94	87	-
		0.7	90	-	-	-
		1.0	77, 114	71–114	96	-

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
Grape 7074-96-0287-CR-001 Kenyon, R.G. 1997a	Fluazinam	0.01	80, 90	80-90	85	-
		0.05	108	-	-	-
		0.1	99, 100, 100	99-100	100	0.6
		0.2	115	-	-	-
		0.5	94, 94	94	94	-
		1.0	128, 95	95-128	112	-
		3.0	90	-	-	-
Grape 7074-97-0059-CR-001 Kenyon, R.G. 1997b	AMGT	0.01	80, 70	70-80	75	-
		0.03	103	-	-	-
		0.05	86	-	-	-
		0.08	81	-	-	-
		0.1	110, 89, 74	74-110	91	20
		0.2	90	-	-	-
		0.3	87	-	-	-
		0.5	82, 102	82- 102	92	-
Grape 6649-96-0022-CR-001 Dvorak, R.S. and Kenyon, R.G. 1996	Fluazinam	0.01	90, 120	90-120	96	-
		0.5	108	-	-	-
		1.0	102, 108	102-108	105	-
		8.0	104	-	-	-
	AMGT	0.01	110, 90	90-110	100	-
		0.1	85, 110	85-110	98	-
		0.5	90	-	-	-
		1.0	76	-	-	-
Grape 6245-95-0001-CR-003 Jablonski, J.E. 1995c	Fluazinam	0.01	130	-	-	-
		0.02	110	-	-	-
		0.05	96	-	-	-
		0.1	117	-	-	-
		0.2	113, 104	104-113	109	-
		0.5	106	-	-	-
		1.0	106	-	-	-
	AMGT	0.01	60	-	-	-
		0.02	70	-	-	-
		0.05	80	-	-	-
		0.1	80	-	-	-
		0.2	108, 69, 83	69-108	89	
		0.5	87	-	-	-
		1.0	82	-	-	-
Blueberry IR-4 PR No. 06129 Thompson, D.C. 2006a	Fluazinam	0.01	100, 110, 98	98-110	103	6
		0.10	120, 120, 120	120	120	0
		1.0	130, 140, 120, 120, 120, 120, 110	110-140	123	8
		0.01 (concurrent)	100, 84, 85, 140, 110, 60, 66, 81, 120, 140, 95	60–140	98	28
		0.10 (concurrent)	90, 90, 88, 83, 110, 100, 98, 100, 74, 110	74–110	94	12
		1.0 (concurrent)	110, 110, 96, 86, 140, 100, 110	86–140	107	16
		3.0 (concurrent)	97, 80, 80	80-97	86	12
	AMGT	0.02	110, 95, 100	95-110	102	8
		0.10	95, 81, 94	81-95	90	9
		1.0	110, 110, 110	110	110	0
		0.02 (concurrent)	125, 65, 70, 70, 110	65-125	88	31
		0.10 (concurrent)	93, 76, 81, 82, 71, 58, 81, 82,78, 79, 74, 72, 100, 73, 70, 88, 68, 68, 110, 70, 80, 90, 89, 83, 84, 82, 88	58-110	80	13
		0.2	95, 80	80-95	88	-

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
		(concurrent)				
		1.0 (concurrent)	77	-	-	-

Analytical method 4 [PPRAM 87] (Grape trials conducted in 1990)

Residues of fluazinam were extracted using methanol. The extract was evaporated to give the aqueous phase which was then partitioned with dichloromethane and cleaned by adsorption chromatography on a silica cartridge. Final determination was by GC-ECD.

The method was validated within the residue trial studies prior to sample analysis or with concurrent recoveries being analysed.

The linearity of the detector response was not reported.

The recovery data obtained from each study are summarised in Table 57.

Table 57 Recovery data for analytical method PPRAM 87 used to determine residues of fluazinam in grapes.

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
Grape M53785 Ryan, J. and Sapiets, A. 1991a	Fluazinam	0.02–0.1	-	-	87	15
Grape M5377B Ryan, J. and Sapiets, A. 1991b	Fluazinam	0.05–0.2	-	-	86	12
Grape RJ1107B Burke, S.R. and Sapiets, A. 1991a	Fluazinam	0.1–0.2	-	-	91	9
Grape RJ1133B Burke, S.R. and Sapiets, A. 1992b	Fluazinam	0.02–0.5	-	-	87	11
Grape RJ1147B Burke, S.R. and Sapiets, A. 1992c	Fluazinam	0.02–0.5	-	-	90	11
Grape 6936-96-0228-CR-001 Kenyon, R.G. 1996	Fluazinam	0.2	93, 87, 93, 89, 91, 83	83-93	91	3
		0.5	94, 81, 83, 95, 101, 88	81-101	91	9
Grape RJ1112B Ryan, J. and Sapiets, A. 1992b	Fluazinam	0.1	-	-	86	6

Analytical method 5 (Swiss grape trials conducted in 1990)

Residues of fluazinam were extracted using acetone and hydrochloric acid and filtered. Saturated sodium chloride solution was added and the extracts were partitioned with toluene. The organic phase was evaporated to dryness, re-dissolved in hexane and cleaned up by solid phase extraction using silica gel columns. Final determination was by GC-ECD.

The method was validated within the residue trial studies with concurrent recoveries being analysed. The linearity of the detector response was not reported. The recovery data obtained from each study are summarised in Table 58.

Table 58 Recovery data for analytical method PPRAM 87 used to determine residues of fluazinam in grapes

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
Grape 343631 Schanné C. 1994	Fluazinam	0.02	95	-	-	-
		0.1	113	-	-	-

Analytical method 6 (Grape)

Residues of fluazinam were extracted using methanol. The methanol extract was partitioned with 2M HCl followed by hexane. The hexane phase was partitioned with 5M NaOH. The alkaline layer was acidified to pH 1 and partitioned with hexane. Hexane extracts were evaporated to dryness and the residue re-dissolved in acetone. Final determination was by GC-ECD.

Residues of AMGT were extracted using acetonitrile: water (4:1, v/v) followed by filtration. Aqueous sodium sulphate was added, extracts partitioned with methylene chloride, pH adjusted to 1 with HCl and partitioned twice with ethyl acetate. The organic phase was evaporated to near dryness, re-dissolved in water and cleaned-up using a C18 Sep-Pak column.

Final determination was by HPLC-UV with quantification at 254 nm.

Both methods were validated within the residue trial studies with concurrent recoveries being analysed. The linearity of the detector response covered a working range of 0.005–0.05 µg/mL and 0.01 –1 µg/mL for fluazinam and 0.5 –5 µg/mL for AMGT respectively. The LOQ validated was 0.01 mg/kg for both Fluazinam and AMGT.

The recovery data obtained from each study are summarised in Table 59.

Table 59 Recovery data for analytical method 6 used to determine residues of fluazinam in grapes.

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
Grape 604372 Schulz, M. and Ullrich-Mietzel, A. 1996	Fluazinam	0.01	88, 107, 111	88-111	102	12
		0.10	116, 72, 98, 76	72–116	91	23
		4.0	81, 101	81-101	91	-
	AMGT	0.01	74, 106	74-106	90	-
		0.10	84	-	-	-

Method 7 (Identical to AGR/MOA/FLZ-7 monitoring method)

Residues of fluazinam, AMPA and AMGT were extracted from crop samples by shaking with methanol: acetic acid (98:2 v/v). After Celite filtration and dilution, an aliquot was purified using an Oasis HLB cartridge. The residues were eluted with acetonitrile: ultrapure water (80:20, v/v) and then analysed by LC-MS/MS. Quantitation and confirmation was performed using the following mass transitions:

Analyte	Quantitation	Confirmation
Fluazinam	463→416	463→398
AMPA	433→397	433→303
AMGT	681→431	681→327

This method is identical to the monitoring method that was validated in the representative crop matrices of potato and grape by Heilaut, 2008 [Ref: ISK/FLU/08002] and in onion, dry beans and oilseed rape seed by Gemrot, 2011 [Ref: S10-03542]. In addition concurrent recoveries were analysed validated within the residue trial studies.

The recovery data obtained from these studies are summarised in Table 60. Recovery data were only reported for the ion transition used for quantification.

Table 60 Recovery data for analytical method 7 used to determine residues of fluazinam and its metabolites in grapes and cabbage.

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
Grape S10-02337 Gemrot, F. 2011c	Fluazinam	0.01	92	-	-	-
		1.0	95	-	-	-
	AMPA	0.01	94	-	-	-
		1.0	96	-	-	-
	AMGT	0.01	106	-	-	-
		1.0	100	-	-	-
Grape S10-02338 Gemrot, F. 2011d	Fluazinam	0.1	95, 96, 97	95–97		
		0.1	90, 89	89-90	90	-
		1.0	90	-	-	-
	AMPA	0.01	98, 100, 97	97–100	98	
		0.1	90, 90	-	90	-
		1.0	93	-	-	-
	AMGT	0.01	90, 110, 88	88-110	96	
		0.1	88, 87	87-88	88	-
		1.0	90	-	-	-
Grape ISK/FLU/08001 Heilaut, C. 2009	Fluazinam	0.01	103	-	-	-
		0.1	106	-	-	-
	AMPA	0.01	92	-	-	-
		0.1	98	-	-	-
	AMGT	0.01	89	-	-	-
		0.1	95	-	-	-
Grape S10-00193 Gemrot, F. 2011b	Fluazinam	0.01	99	-	-	-
		10	97	-	-	-
	AMPA	0.01	100	-	-	-
		10	100	-	-	-
	AMGT	0.01	104, 76, 100	76–104	93	
		0.1	95, 96	95-96	96	-
		10	96	-	-	-
Cabbage IR-4 PR No. 07093 Barney, W.P. 2014a	Fluazinam	0.01	89, 91, 86	86–91	91	3
		0.1	91, 91, 89	89–91	91	1
		1.0	87, 89, 89	87–89	89	1
		0.01 (concurrent)	96, 83, 84	83–96	88	8
		0.1 (concurrent)	92, 91, 88, 86	86–92	89	3
		1.0 (concurrent)	87, 89	87–89	88	2
		10 (concurrent)	89, 87, 85	85–89	87	2
	AMGT	0.01	87, 82, 89	82–89	89	4
		0.1	94, 84, 89	84–94	94	6
		1.0	97, 95, 101	95-101	98	3
		0.01 (concurrent)	89, 70, 90	70–90	83	14
		0.1 (concurrent)	97, 91, 93, 94	91–97	94	3
		1.0 (concurrent)	82, 90	82-90	86	7
		10 (concurrent)	99, 95, 94	94-99	98	3

Method 8 (grapes, cabbage, mustard greens, lettuce)

Residues of fluazinam were extracted from crop samples by shaking with methanol: acetic acid (98:2 v/v). After Celite filtration and dilution, an aliquot was acidified and partitioned twice with hexane; the organic phase was subsequently partitioned with 0.5M

sodium hydroxide. The aqueous phase was adjusted to pH 1 using HCl and then portioned twice with hexane. The organic phase was concentrated to dryness, re-dissolved in hexane and purified using either:

A Florisil column eluting with hexane: ethyl acetate water (95:5, v/v), evaporating to dryness and re-dissolving in dodecane: acetone (9:1 v/v).

A C18 Sep-Pak column eluting with hexane: dichloromethane (50:50, v/v), evaporating to dryness and re-dissolving in hexane.

Residues were determined by GC-ECD.

The methods were validated within the residue trial studies with concurrent recoveries being analysed. The linearity of the detector response covered a working range of 0.01–0.5 µg/mL for fluazinam. The recovery data obtained from each study are summarised in Table 61.

Table 61 Recovery data for analytical method 8 used to determine residues of fluazinam in brassicas and leafy crops

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
Grape 734387 Wais, A. 2000	Fluazinam	0.01	95	-	-	-
		0.1	107	-	-	-
Cabbage IR-4 PR No. 08796 Thompson, D.C. 2006c	Fluazinam	0.01	62, 50, 85, 85, 80	50–85	72	22
		0.1	82, 93, 82	82–93	86	7
		0.01 (concurrent)	82, 83, 103, 95, 82, 80, 85, 85, 100, 78, 82	78–103	87	10
		0.1 (concurrent)	113, 66, 101, 106, 91, 125, 113, 89, 99, 91, 99	66–125	99	16
Cabbage AAFC03-066R Ballantine, J. 2006	Fluazinam	0.01	74, 78, 81	74–81	78	5
		0.02	93, 76, 84	76 –93	84	10
		0.10	108, 110, 113	108-113	110	2
		0.05 (concurrent)	99	-	-	-
		0.1 (concurrent)	72, 89, 85, 72, 77, 72, 70, 82, 72, 77, 71, 86, 107, 115, 75	70-115	81	17
Mustard Greens IR-4 PR No. 08797 Thompson, D.C. 2006d	Fluazinam	0.01	93, 77, 70	70–93	80	15
		0.1	109, 95, 80	80–109	95	15
		0.01 (concurrent)	104, 90, 94, 77, 64, 97, 75, 76, 109, 103	64–109	89	17
		0.1 (concurrent)	87, 118, 99, 90, 80, 71, 104, 87, 87, 85, 97, 95	71–118	92	13
Lettuce IR-4 PR No. 06892 Carpenter, D.H. 2008b	Fluazinam	0.01	90, 101, 98, 96, 85, 106, 105, 101	85–106	98	7
		0.1	105, 108, 110	105–110	108	2
		1.0	101, 102, 99	99–102	101	2
		3.0	110, 105, 108	105–110	108	2
		0.01 (concurrent)	82, 96, 114, 111, 109, 105, 108, 99, 90	82–114	102	11
		0.02 (concurrent)	98, 85, 86	85–98	90	8
		0.2 (concurrent)	119, 91	91–119	105	-
		1.0 (concurrent)	118, 117	117-118	118	-

Analytical method 9 (Broccoli Trials conducted in 2004)

Residues of fluazinam were extracted using methanol: acetic acid (125:2, v/v) followed by filtration. The extract was acidified with 0.2N HCl and partitioned twice with hexane. The hexane phase was partitioned with 0.5M NaOH. The aqueous phase was acidified and partitioned twice with hexane. The hexane phase was concentrated and analysed by GC-ECD.

The methods were validated within the residue trial studies prior to sample analysis or with concurrent recoveries being analysed. The linearity of the detector response covered a working range of 0.005–0.025 µg/mL for fluazinam. The recovery data obtained from each study are summarised in Table 62.

Table 62 Recovery data for analytical method 9 used to determine residues of fluazinam in broccoli.

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
Broccoli IR-4 PR No. 08795 Thompson, D.C. 2006b Validation	Fluazinam	0.01	90, 80, 82, 82, 72, 81	72–90	81	7
		0.10	68, 83, 88	68–88	80	13
		0.01 (concurrent)	57, 65, 58, 86, 100, 70, 81, 72, 110	57–110	78	24
		0.10 (concurrent)	88, 76, 77, 72, 73, 88, 72, 90	72-90	80	10

Analytical method 10 (Melon, cucumber, summer squash, pepper, soya bean)

Residues of fluazinam and AMGT were extracted using methanol: acetic acid (98:2, v/v). The extract was cleaned-up by polymeric SPE and diluted with water. Final determination was by LC-MS/MS.

The method was validated for each commodity prior to the sample analysis. The linearity of the detector response covered a working range of 0.1–2 µg/mL for both fluazinam and AMGT. The recovery data are summarised in Table 63.

Table 63 Recovery data for analytical method 10 used to determined residues of fluazinam and AMGT in cantaloupe melon, cucumber and summer squash

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
Cantaloupe melon IR-4 PR No. 07097 Thompson, D.C. 2011a	Fluazinam	0.01	83, 81, 77	77-83	84	3.7
		0.1	82, 85, 85	82-85	84	2.1
		1	92, 90, 88	88-92	90	2.2
	AMGT	0.01	83, 92, 88	83-92	88	5.1
		0.1	87, 90, 88	87-90	88	1.7
		1	97, 95, 96	95-97	96	1.0
Cucumber IR-4 PR No. 09238 Barney, W.P. 2014b	Fluazinam	0.01	93, 96, 91	91-96	93	2.7
		0.1	93, 91, 89	89-93	91	2.2
		1	94, 89, 94	89-94	92	3.1
	AMGT	0.01	98, 94, 99	94-99	97	2.7
		0.1	89, 86, 89	86-89	88	2.0
		1	87, 78, 96	78-96	87	10
Pepper IR-4 PR No. 09556 Thompson, D.C. 2011b	Fluazinam	0.01	85, 89, 84	84-89	86	3.1
		0.1	90, 87, 88	87-90	88	1.7
		1	86, 84, 87	84-87	86	1.8
	AMGT	0.01	83, 97, 94	83-97	91	8.1
		0.1	88, 93, 93	88-93	91	3.2
		1	88, 92, 91	88-92	90	2.3
Soya bean seeds IB-2010-JLW-006-00-01	Fluazinam	0.01	97, 93, 98	93-98	96	3
		0.1	108, 104, 109	104-109	107	3
	AMGT	0.01	83, 104, 76	76-104	88	17
		0.1	109, 104, 106	104-109	106	2
Soya bean forage IB-2010-JLW-006-00-01	Fluazinam	0.01	92, 79, 89	79-92	86	8
		0.1	94, 106, 99	94-106	100	6
	AMGT	0.01	105, 109, 102	102 -109	105	3
		0.1	102, 106, 105	102-106	104	2
Soya bean hay IB-2010-JLW-006-00-01	Fluazinam	0.01	86, 84, 88	84-88	86	2
		0.1	97, 94, 102	94-102	97	4
	AMGT	0.01	118, 108, 102	76-104	96	3
		0.1	110, 112, 109	104 -109	107	3

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
Soya bean grain dust	Fluazinam	0.01	72, 72, 76	72-76	73	3
		0.1	91, 118, 85	85-118	98	18
IB-2010-JLW-006-00-01	AMGT	0.01	108, 107, 86	86-108	100	12
		0.1	109, 108, 113	108-113	110	2
Soya bean hulls	Fluazinam	0.01	78, 77, 76	76-78	77	1
		0.1	118, 121, 116	116-121	118	2
IB-2010-JLW-006-00-01	AMGT	0.01	97, 100, 92	92-100	96	4
		0.1	118, 117, 115	115-118	116	2
Soya bean meal	Fluazinam	0.01	111, 101, 107	101 -111	106	5
		0.1	120, 102, 109	102-120	110	8
IB-2010-JLW-006-00-01	AMGT	0.01	94, 84, 96	84-96	91	7
		0.1	104, 103, 107	103-107	105	2
Soya bean refined oil	Fluazinam	0.01	79, 89, 85	79-89	84	6
		0.1	92, 73, 101	73-101	89	16
IB-2010-JLW-006-00-01	AMGT	0.01	100, 106, 102	100-106	103	3
		0.1	110, 107, 114	107-110	110	3

Analytical method 11 (Dry bean)

Residues of fluazinam were extracted using methanol: acetic acid (100:2, v/v) followed by filtration. The extract was acidified with 0.2N HCl and partitioned twice with hexane. The hexane phase was partitioned with 0.5M NaOH. The aqueous phase was acidified and partitioned twice with hexane. The hexane phase was concentrated and analysed by GC-ECD.

The methods were validated within the residue trial studies prior to sample analysis or with concurrent recoveries being analysed. The linearity of the detector response covered a working range of 0.002–0.012 µg/mL for fluazinam. The recovery data obtained from each study are summarised in Table 64.

Table 64 Recovery data for analytical method 11 used to determine residues of fluazinam in dried beans

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
Dry beans IR-4 PR No. 06369 Thompson, D.C. 2006e	Fluazinam	0.01	102, 88, 71	71–102	87	18
		0.10	102, 100, 94	94–102	99	4
		1.0	98, 95, 87	87–98	93	6
		0.01 (concurrent)	88, 72, 74, 88, 76, 79, 83, 71, 79, 100, 86, 108, 77	72-108	83	13
		1.0 (concurrent)	96	-	-	-

Analytical method 12 (Tea)

Residues of Fluazinam, MAPA and HYPA were extracted using methanol: phosphoric acid followed by filtration. The extract was partitioned with hexane followed by acetonitrile partitioning. The extract was concentrated and cleaned up using Florisil columns followed by silica gel column chromatography. Residues were analysed by GC-ECD. For HYPA, an additional methylation step using diazomethane was necessary before GC-ECD analysis. The linearity of the detector response covered a working range of 0.005–0.2 µg/mL for fluazinam, MAPA and HYPA. The recovery data are summarised in Table 65.

Table 65 Recovery data for analytical method 12 used to determined residues of fluazinam, MAPA and HYPA in tea

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
Tea	Fluazinam	0.4	91, 90	90-91	91	-
		40	91, 85	85-91	88	-
Komatsu, K. and	MAPA	0.4	85, 77	77-85	81	-

Crop/ Study reference	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD
Yabusaki. T. 1993	HYPA	0.4	78, 74	74-78	76	-
Tea	Fluazinam	0.2	96, 93, 94, 92, 99, 97	92-99	95	3
	MAPA	0.2	84, 75, 81, 80, 79, 73	73-84	79	5
Komatsu, K. and Yabusaki. T. 1997	HYPA	0.2	76, 73, 77, 76, 82, 76	73-82	77	4

Data collection methods–animal commodities

Method IB-2007-JLW-004-00-01

This method is presented in the enforcement methods section in the study by Weidmann, 2008b [Ref: IB-2007-JLW-004-00-01].

Enforcement methods–plant matrices

Method AGR/MOA/FLZ-7 (potato, grape, onion, dry beans, oilseed rape seed)

Residues of fluazinam, AMPA and AMGT were extracted from crop samples by shaking with methanol: acetic acid (98:2 v/v). After Celite filtration and dilution, an aliquot was purified using an Oasis HLB cartridge. The residues were eluted with acetonitrile: ultrapure water (80:20, v/v) and then analysed by LC-MS/MS. Quantitation and confirmation was performed using the following mass transitions:

Analyte	Quantitation	Confirmation
Fluazinam	463→416	463→398
AMPA	433→397	433→303
AMGT	681→431	681→327

The method was validated in the representative crop matrices of potato (high starch) and grape (high acid content) by Heilaut, 2008 [Ref: ISK/FLU/08002] and in onion (high water content), dry beans (high protein content) and oilseed rape seed (high oil content) by Gemrot, 2011 [Ref: S10-03542].

The method showed good linearity in the range of 0.1-5 ng/mL for all analytes (correlation coefficients >0.99 and no significant interferences were noted at the retention times corresponding to the analytes in any control samples (the response in control samples at the relevant retention times for fluazinam, AMPA and AMGT always corresponded to less than 30% of the limit of quantification). The mean recoveries for all matrices tested at all fortification levels ranged from 78 to 104%, within the acceptable range, with relative standard deviations of <20%. The limits of quantitation (LOQs) were 0.01 mg/kg for all matrices tested.

Table 66 Method AGR/MOA/FLZ-7 analytical recovery rates for fluazinam and its metabolites in crop matrices

Matrix	Fortification level (mg/kg)	Number of tests	% Recovery	Average % recovery	% RSD	Reference
Fluazinam, m/z 463 →416						
Potato	0.01	5	89, 89, 88, 91, 91	90	1	ISK/FLU/08002
	0.1	5	93, 92, 92, 87, 91	91	3	
Grape	0.01	5	98, 97, 103, 93, 95	97	4	
	0.1	5	98, 97, 102, 105, 92	99	5	
Onion	0.01	5	99, 88, 96, 96, 92	94	5	S10-03542
	0.1	5	83, 83, 79, 79, 78	80	3	
Dry bean	0.01	5	95, 93, 98, 98, 95	96	2	
	0.1	5	83, 82, 79, 82, 83	82	2	
Oilseed rape seed	0.01	5	93, 90, 98, 96, 94	94	3	
	0.1	5	78, 79, 81, 84, 80	80	3	
Fluazinam, m/z 463 →398						
Potato	0.01	5	90, 90, 93, 91, 92	91	1	ISK/FLU/08002

Matrix	Fortification level (mg/kg)	Number of tests	% Recovery	Average % recovery	% RSD	Reference
	0.1	5	93, 92, 91, 88, 91	91	2	
Grape	0.01	5	97, 96, 102, 95, 96	97	3	
	0.1	5	98, 96 ,102, 105, 91	98	5	
Onion	0.01	5	97, 88, 94, 94, 91	93	4	S10-03542
	0.1	5	84, 85, 78, 81, 78	81	4	
Dry bean	0.01	5	94, 93, 100, 100, 94	96	4	
	0.1	5	84, 82, 80, 84, 83	83	2	
Oilseed rape seed	0.01	5	92, 89, 96, 95, 93	93	3	
	0.1	5	79, 79, 81, 85, 80	81	3	
AMPA, m/z 433 →397						
Potato	0.01	5	78, 82, 79, 82, 84	81	3	ISK/FLU/08002
	0.1	5	89, 99, 92, 89, 88	92	5	
Grape	0.01	5	98, 103, 115, 99, 103	104	6	
	0.1	5	104, 99, 101, 101, 104	102	2	
Onion	0.01	5	98, 87, 93, 93, 92	92	4	S10-03542
	0.1	5	84, 83, 78, 80, 78	81	3	
Dry bean	0.01	5	97, 90, 98, 97, 97	96	3	
	0.1	5	82, 81, 79, 82, 83	81	2	
Oilseed rape seed	0.01	5	93, 91, 97, 96, 92	94	3	
	0.1	5	78, 79, 82, 84, 80	81	3	
AMPA, m/z 433 →303						
Potato	0.01	5	80, 82, 83, 87, 88	84	4	ISK/FLU/08002
	0.1	5	101, 101, 96, 95, 100	99	3	
Grape	0.01	5	99, 105, 109, 96, 104	102	5	
	0.1	5	105, 99, 99, 99, 104	101	3	
Onion	0.01	5	98, 88, 97, 98, 91	94	5	S10-03542
	0.1	5	86, 84, 78, 81, 79	82	4	
Dry bean	0.01	5	96, 91, 97, 97, 94	95	3	
	0.1	5	82, 81, 79, 83, 85	82	3	
Oilseed rape seed	0.01	5	94, 91, 99, 95, 94	94	3	
	0.1	5	79, 80, 81, 85, 81	81	3	
AMGT, m/z 681 →431						
Potato	0.01	5	90, 93, 86, 92, 105	93	8	ISK/FLU/08002
	0.1	5	86, 81, 81, 77, 78	81	4	
Grape	0.01	5	89, 88, 92, 86, 88	89	3	
	0.1	5	98, 91, 95, 100, 91	95	5	
Onion	0.01	5	89, 65, 87, 83, 93	83	13	S10-03542
	0.1	5	79, 80, 76, 78, 78	78	2	
Dry bean	0.01	5	107, 92, 101, 106, 88	99	8	
	0.1	5	81, 81, 81, 79, 83	81	2	
Oilseed rape seed	0.01	5	85, 96, 108, 104, 108	100	10	
	0.1	5	80, 84, 91, 89, 89	87	5	
AMGT, m/z 681 →327						
Potato	0.01	5	83, 87, 86, 96, 82	87	6	ISK/FLU/08002
	0.1	5	85, 84, 78, 77, 76	80	5	
Grape	0.01	5	98, 95, 94, 87, 95	94	4	
	0.1	5	96, 94, 98, 104, 89	96	6	
Onion	0.01	5	99, 89, 108, 100, 95	98	7	S10-03542
	0.1	5	81, 80, 76, 78, 74	78	4	
Dry bean	0.01	5	96, 94, 103, 101, 97	98	4	
	0.1	5	87, 81, 81, 84, 83	83	3	
Oilseed rape seed	0.01	5	105, 84, 88, 103, 102	97	10	
	0.1	5	81, 83, 88, 88, 87	82	4	

No consideration of the extraction efficiency of this method was provided. Instead extraction efficiency for “Method 1” used in support of residue trials for peanuts was provided [Ref: 6574-95-0257-EF-001]. In “Method 1” samples are extracted by homogenisation with methanol: acetic acid (98:2 v/v), however the subsequent clean-up, which includes additional acidification and solvent partition steps, and the measurement technique are different.

An independent laboratory validation of Method AGR/MOA/FLZ-7 for residues of fluazinam, AMPA and AMGT in potato, grapes, onion, dry beans and oilseed rape seed was conducted by Eichler, 2010 and 2011 [Refs: 59321101 and59322101] and reported good linearity in the range of 0.1-5 ng/mL for all analytes (correlation coefficients >0.99) and no significant interferences at the relevant retention times. The mean recoveries for all matrices tested at all fortification levels ranged from 73 to 100% (RSDs <20%) with the exception of dry beans (high protein content) for AMPA and AMGT where average recoveries were in the range 67–93% with RSDs of 11-27%. The limits of quantitation (LOQs) were 0.01 mg/kg for all matrices tested.

Table 67 Method AGR/MOA/FLZ-7: Independent validation recovery rates for fluazinam and its metabolites in crop matrices

Matrix	Fortification level (mg/kg)	Number of tests	% Recovery	Average % recovery	% RSD	Reference
Fluazinam, m/z 463 →416						
Potato	0.01	5	70, 67, 80, 77, 76	74	7.2	59321101
	0.1	5	84, 73, 77, 82, 83	80	5.8	
Grape	0.01	5	76, 79, 79, 79, 78	78	1.7	
	0.1	5	85, 83, 81, 85, 84	84	2.0	
Onion	0.01	5	85, 84, 83, 82, 77	82	3.8	59322101
	0.1	5	78, 72, 67, 74, 74	73	5.5	
Dry bean	0.01	5	104, 89, 87, 99, 88	93	8.2	
	0.1	5	93, 96, 92, 74, 91	89	9.8	
Oilseed rape seed	0.01	5	87, 91, 89, 86, 86	88	2.5	
	0.1	5	82, 86, 83, 83, 82	83	2.0	
Fluazinam, m/z 463 →398						
Potato	0.01	5	77, 72, 79, 78, 81	77	4.3	59321101
	0.1	5	85, 71, 73, 83, 84	80	7.6	
Grape	0.01	5	77, 82, 82, 78, 82	80	3.1	
	0.1	5	86, 83, 81, 85, 83	84	2.3	
Onion	0.01	5	98, 93, 93, 91, 86	92	4.7	59322101
	0.1	5	79, 71, 68, 77, 73	74	6.0	
Dry bean	0.01	5	99, 87, 91, 90, 93	92	4.9	
	0.1	5	95, 99, 90, 70, 93	89	12.7	
Oilseed rape seed	0.01	5	75, 67, 77, 74, 73	73	5.1	
	0.1	5	84, 91, 84, 85, 80	85	4.7	
AMPA, m/z 433 →397						
Potato	0.01	5	90, 90, 86, 91, 90	89	2.2	59321101
	0.1	5	96, 83, 92, 93, 92	91	5.3	
Grape	0.01	5	87, 97, 95, 91, 90	92	4.3	
	0.1	5	97, 93, 95, 94, 94	95	1.6	
Onion	0.01	5	103, 93, 97, 103, 100	99	4.3	59322101
	0.1	5	89, 82, 83, 84, 86	85	3.3	
Dry bean	0.01	5	82, 85, 135, 85, 79	93	25.2	
	0.1	5	95, 91, 91, 71, 94	88	11.2	
Oilseed rape seed	0.01	5	82, 84, 85, 83, 79	83	2.8	
	0.1	5	92, 82, 81, 79, 83	83	6.0	
AMPA, m/z 433 →303						
Potato	0.01	5	88, 89, 89, 89, 91	89	1.2	59321101
	0.1	5	96, 83, 95, 94, 92	92	5.7	
Grape	0.01	5	87, 93, 95, 94, 93	92	3.4	
	0.1	5	96, 93, 95, 96, 94	95	1.4	
Onion	0.01	5	97, 96, 104, 102, 101	100	3.4	59322101
	0.1	5	88, 83, 90, 86, 89	87	3.2	
Dry bean	0.01	5	97, 85, 134, 84, 87	97	21.7	
	0.1	5	92, 91, 91, 72, 92	88	10.0	
Oilseed rape seed	0.01	5	84, 87, 89, 80, 81	84	4.6	
	0.1	5	95, 80, 80, 78, 82	83	8.3	
AMGT, m/z 681 →431						
Potato	0.01	5	92, 100, 96, 95, 87	94	5.2	59321101

Matrix	Fortification level (mg/kg)	Number of tests	% Recovery	Average % recovery	% RSD	Reference
	0.1	5	85, 74, 84, 84, 82	82	5.5	
Grape	0.01	5	79, 95, 101, 91, 81	89	10.4	
	0.1	5	92, 86, 91, 85, 85	88	3.9	
Onion	0.01	5	74, 81, 96, 78, 108	87	15.1	59322101
	0.1	5	90, 84, 96, 83, 81	87	7.1	
Dry bean	0.01	5	88, 75, 75, 63, 90	78	14.1	
	0.1	5	84, 134, 83, 68, 89	92	27.3	
Oilseed rape seed	0.01	5	75, 74, 56, 71, 73	70	11.3	
	0.1	5	77, 74, 71, 71, 75	71	3.5	
AMGT, m/z 681 →327						
Potato	0.01	5	75, 77, 74, 82, 82	78	4.9	59321101
	0.1	5	83, 71, 79, 79, 85	79	6.8	
Grape	0.01	5	77, 82, 87, 79, 76	80	5.5	
	0.1	5	89, 86, 86, 84, 80	85	3.9	
Onion	0.01	5	107, 86, 68, 95, 87	89	16.1	59322101
	0.1	5	93, 91, 97, 79, 81	88	8.9	
Dry bean	0.01	5	73, 52, 66, 92, 53	67	24.5	
	0.1	5	83, 130, 88, 65, 82	90	27.0	
Oilseed rape seed	0.01	5	102, 99, 99, 79, 99	96	9.8	
	0.1	5	80, 77, 77, 76, 79	78	2.1	

Method US FDA PAM 1

The analytical characteristics of fluazinam, AGMT and AMPA when subject to analysis by US FDA Multi-Residue Protocols A, C, D, E, and F (third edition 1/94) were investigated in three separate studies by Rhodes, 1995 [Ref: 6582-95-0190-EF], 1996a [Ref: 6582-95-0191-EF], 1996b [Ref: 6582-95-0192-EF]. The analytical characteristics of the metabolite DAPA when subjected to the same methods were investigated by Robaugh, 2011 [Ref: 6582-95-0192-EF].

Briefly, Protocol A evaluates tests substances for fluorescence, to determine if LC analysis is appropriate and if required Protocol C evaluates the GC profile of tests substances using a variety of column polarity and detection systems. Protocols D, E and F evaluate various extractions and clean-up procedures, using test materials in solvent and in fortified food matrices (fatty and non-fatty).

A summary of the method parameters evaluated and the results for each compound is given in Table 70. The FDA PAM 1 methods could be used to determine residues of fluazinam in high water and high fat content commodities and residues of AMPA in high fat content commodities. The methods are not suitable for the determination of AGMT or DAPA.

Table 68 Summary of the evaluation of US FDA PAM 1 for the determination of fluazinam and its metabolites

Analyte	Fluazinam	AMGT	AMPA	DAPA
Protocol A	Not fluorescent. No further work required	Not fluorescent. No further work required	Not fluorescent. No further work required	Not fluorescent. No further work required
Protocol B	Not applicable	GC analysis of methylated derivatives was successful.	Not applicable	Not applicable
Protocol C	Response for both GC-ECD and GC-NPD. GC-ECD 10× better sensitivity	GC-NPD selected.	GC-NPD selected.	GC-NPD selected.
Protocol D	Validated in grapes. Mean recovery 72.7% ± 22% (n = 4) at 0.05 and 0.5 mg/kg	Validated in grapes No recovery data obtained due to matrix interference.	Validated in wine. Mean recovery 82% ± 12% (n = 4) at 0.05 and 0.25 mg/kg	Validated in potatoes. Mean recovery 29% (n = 2) at 0.05 mg/kg Mean recovery 19% (n = 2) at 0.25 mg/kg
Protocol E	Validated in grapes Clean-up 303 C1: Mean recovery 114% ± 73% (n = 4) at 0.05 and 0.5 mg/kg Clean-up 303 C2: Mean recovery 76% ± 15% (n = 4) at 0.05 and 0.5 mg/kg	AMGT was not recovered from Florisil columns. No further work required.	Validated in wine Clean-up 303 C1: Mean recovery 37% ± 22% (n = 4) at 0.05 and 0.5 mg/kg Clean-up 303 C2: Mean recovery 20% ± 22% (n = 4) at 0.05 and 0.5 mg/kg	DAPA was not recovered from Florisil columns. No further work required.

Analyte	Fluazinam	AMGT	AMPA	DAPA
Protocol F	Validated in peanut. Clean-up 304 C1: Mean recovery 71% ± 9% (n = 4) at 0.05 and 0.5 mg/kg Clean-up 304 C2: Mean recovery 87% ± 60% (n = 4) at 0.05 and 0.5 mg/kg	AMGT was not recovered from Florisil columns. No further work required.	Validated in milk. Clean-up 304 C1: Mean recovery 82% ± 10% (n = 4) at 0.05 and 0.5 mg/kg Clean-up 304 C2: Mean recovery 59% ± 6% (n = 4) at 0.05 and 0.5 mg/kg	DAPA was not recovered from Florisil columns. No further work required
Study reference	6582-95-0190-EF	6582-95-0191-EF	6582-95-0192-EF	6582-95-0192-EF

Enforcement methods–animal commodities

Method ISK-0504V

The determination of residues of fluazinam in animal matrices was investigated and validated by Lakaschus, 2006 [Ref: ISK-0504V].

Meat, milk and fat were extracted with methanol: acetic acid (100:2, v/v) in the presence of Celite 545 and filtered. The extract was acidified with HCl and partitioned with hexane. The hexane phase was reduced to near dryness, taken up in ethyl acetate: cyclohexane (1:1, v/v) and cleaned-up using gel permeation chromatography and silica column chromatography.

Liver and eggs were extracted under acidic conditions with a mixture of hydrochloric acid, sodium chloride and ethyl acetate. After homogenization and centrifugation, the organic phase is isolated, concentrated and cleaned up by gel permeation chromatography and silica column chromatography.

The final extracts were diluted in toluene and analysed by gas-liquid chromatography with GC-ECD. A further aliquot was diluted with methanol and acetic acid for confirmatory analysis by LC-MS/MS, using the mass transitions m/z 463→416 for quantification and m/z 463→398 for confirmation).

The method showed good linearity in the range of 10-500 ng/mL for GC-ECD and in the range of 0.25-20 ng/mL for LC-MS/MS (correlation coefficients >0.99) and no significant interferences were noted at the retention times corresponding to the analytes in any control samples. The mean recoveries for all matrices tested at all fortification levels ranged from 71 to 100%, within the acceptable range, with relative standard deviations of <20%. The limit of quantitation (LOQ) was 0.01 mg/kg for all matrices tested.

Table 69 Method ISK-0504V analytical recovery rates for fluazinam in animal matrices

Matrix	Fortification level (mg/kg)	Number of tests	% Recovery	Average % recovery	% RSD	Reference
Fluazinam (GC-ECD)						
Meat	0.01	5	86, 80, 84, 70, 80	80	7.8	ISK-0504V
	0.1	5	73, 56, 76, 77, 73	71	12	
Liver	0.01	5	73, 78, 86, 64, 80	76	1	
	0.1	5	72, 77, 91, 68, 105	83	18	
Milk	0.01	5	86, 69, 76, 92, 85	82	11	
	0.1	5	87, 80, 80, 82, 85	83	3.7	
Eggs	0.01	5	105, 104, 102, 96, 105	102	3.7	
	0.1	5	128, 109, 80, 92, 93	100	19	
Fat	0.01	5	118,101, 103, 89, 87	100	13	
	0.1	5	87, 77, 87, 67, 97	83	14	
Fluazinam (LC MS/MS m/z 463→416)						
Meat	0.01	3	89, 81, 91	87	6.1	ISK-0504V
	0.1	3	74, 72, 77	74	3.4	
Liver	0.01	3	79, 87, 84	83	4.8	
	0.1	3	83, 77, 85	82	5.1	
Milk	0.01	3	85, 77, 90	84	7.9	
	0.1	3	76, 80, 82	79	3.9	
Eggs	0.01	3	80, 85, 85	83	3.5	
	0.1	3	80, 73, 74	76	5.0	
Fat	0.01	3	84, 78, 83	82	3.9	
	0.1	3	82, 82, 85	83	2.0	
Fluazinam (LC MS/MS m/z 463→398)						
Meat	0.01	3	87, 82, 92	88	4.1	ISK-0504V

Matrix	Fortification level (mg/kg)	Number of tests	% Recovery	Average % recovery	% RSD	Reference
	0.1	3	73, 72, 77	74	3.5	
Liver	0.01	3	78, 86, 86	83	5.5	
	0.1	3	83, 76, 86	82	6.2	
Milk	0.01	3	81, 82, 85	83	2.5	
	0.1	3	73, 82, 80	78	6.0	
Eggs	0.01	3	79, 85, 84	83	3.9	
	0.1	3	80, 73, 74	76	3.8	
Fat	0.01	3	83, 78, 81	81	3.1	
	0.1	3	83, 80, 86	83	3.6	

Method IB-2007-JLW-004-00-01 (method also used in feeding study)

The determination of residues of fluazinam, AMPA and DAPA in animal matrices was investigated and validated by Weidmann, 2008b [Ref: IB-2007-JLW-004-00-01].

Milk was extracted with acidified methanol in the presence of Celite 545 and filtered. The extract was concentrated, water and NaCl added and partitioned with hexane. The hexane was evaporated to near dryness and taken up in acetonitrile: water (1:1, v/v) for LC-MS/MS analysis of fluazinam and AMPA or taken up in toluene for GC/MS analysis of DAPA.

Muscle was extracted with acidified acetonitrile: water (1:1, v/v) in the presence of Celite 545 and filtered. The extract was concentrated and cleaned-up using Extrelut QE column and analysed using LC-MS/MS.

Fat was extracted with acidified acetonitrile in the presence of Celite 545 and filtered. The extract was partitioned with acetonitrile saturated cyclohexane. The cyclohexane phase was discarded, and the acetonitrile evaporated to near dryness and taken up in acetonitrile: water (1:1, v/v). Analysis was by LC-MS/MS.

Liver and kidney were extracted with acidified acetonitrile: water (liver: 3:1, v/v; kidney 1:1, v/v) in the presence of Celite 545 and filtered. The acetonitrile was evaporated, water and NaCl added and the extract partitioned with dichloromethane. The dichloromethane was evaporated to near dryness and residues taken up in acetonitrile and analysed by LC-MS/MS.

In addition, to determine conjugates in kidney and liver, samples were extracted with acetonitrile: water (1:1, v/v) and an additional hydrolysis step with HCl at 37 °C for 1 hour was added. The extract was partitioned with hexane (liver) or ethyl acetate (kidney). The organic phase was evaporated to near dryness and residues taken up in acetonitrile and analysed by LC-MS/MS.

Quantitation and confirmation was performed using the following mass transitions for LC-MS/MS:

Analyte	Quantitation	Confirmation
Fluazinam	465→373	465→338
AMPA	435→373	435→354
DAPA	405→353	405→333

Validation data were only generated for the ion transition used for quantification.

For the GC-MS determination of DAPA the ion m/z 369 was used for quantification, with m/z 388 and m/z 404 used as qualifier ions.

The method showed good linearity in the range of 0.1- 40 ng/mL for all analytes (correlation coefficients >0.99) and no significant interferences were noted at the retention times corresponding to the analytes in any control samples. Validation data for the quantitation ion/mass transition only were provided and recovery data were provided for two fortification levels for each analyte/matrix combination (n = 3 per level). The mean recoveries for milk, muscle and fat for fluazinam, AMPA and DAPA were generally within or just outside the range of 70-120% with relative standard deviations of <20%, although it is noted that analysis of DAPA residue in milk by GC gave better recovery values.

In liver (without hydrolysis) mean recoveries for fluazinam and AMPA were within or just outside the range of 70-120% with relative standard deviations of <20%. Recoveries for DAPA were poor: with individual and mean recoveries less than 30%.

In kidney without the additional hydrolysis step mean recoveries for fluazinam, AMPA and DAPA were in the range 42-63%; the relative standard deviations were <20% with the exception of fluazinam in kidney for the lowest fortification level.

The additional hydrolysis step (validation data reported in table 71) did not lead to improved recovery data.

The limits of quantitation (LOQs) were stated to be 0.01 mg/kg for all matrices tested.

Table 70 Method IB-2007-JLW-004-00-01 analytical recovery rates for fluazinam and its metabolites in animal matrices (no hydrolysis step)

Matrix	Fortification level (mg/kg)	Number of tests	% Recovery	Average % recovery	% RSD	Reference
Fluazinam (m/z 465 → 373)						
Milk	0.01	3	91, 93, 99	94	4.5	IB-2007-JLW-004-00-01
	0.1	3	71, 74, 80	75	6.1	
Muscle	0.01	3	79, 84, 83	82	3.2	
	0.05	3	64, 70, 65	66	4.8	
Liver	0.01	3	83, 80, 79	81	2.6	
	0.05	3	70, 64, 68	67	4.6	
Kidney	0.01	3	54, 58, 33	48	28	
	0.05	3	49, 41, 35	42	17	
Fat	0.01	3	96, 96, 101	98	3.0	
	0.02	3	99, 103, 103	102	2.3	
	0.1	3	98, 98, 101	99	1.7	
AMPA (m/z 435→373)						
Milk	0.01	3	93, 93, 89	92	2.5	IB-2007-JLW-004-00-01
	0.1	3	80, 86, 91	86	6.4	
Muscle	0.01	3	97, 92, 98	96	3.3	
	0.05	3	94, 96, 96	95	1.3	
Liver	0.01	3	72, 69, 61	67	8.5	
	0.05	3	62, 65, 62	63	2.7	
Kidney	0.01	3	61, 72, 57	63	12	
	0.05	3	50, 49, 44	48	6.7	
Fat	0.01	3	102, 106, 106	105	2.2	
	0.02	3	105, 109, 104	106	2.5	
	0.1	3	101, 104, 104	103	1.7	
	0.5	3	99, 103, 104	102	2.5	
DAPA (m/z 405 →353)						
Milk	0.01	3	58, 69, 65	64	8.8	IB-2007-JLW-004-00-01
	0.1	3	74, 74, 60	69	12	
Muscle	0.01	3	98, 83, 90	90	8.3	
	0.05	3	93, 96, 96	95	1.8	
Liver	0.01	3	25, 24, 24,	24	2.5	
	0.05	3	24, 23, 23	23	2.6	
Kidney	0.01	3	63, 73, 52	63	17	
	0.05	3	65, 65, 60	63	4.6	
Fat	0.01	3	64, 72, 71	69	6.4	
	0.02	3	65, 70, 72	69	5.2	
	0.1	3	88, 78, 74	80	9.0	
	0.5	3	75, 77, 73	75	2.7	
DAPA (GC-MS)						
Milk	0.01	3	105, 109, 108	107	2.0	IB-2007-JLW-004-00-01
	0.1	3	98, 106, 105	103	4.3	

Table 71 Method IB-2007-JLW-004-00-01 analytical recovery rates for fluazinam and its metabolites including conjugates in liver and kidney (hydrolysis step)

Matrix	Fortification level (mg/kg)	Number of tests	% Recovery	Average % recovery	% RSD	Reference
Fluazinam (including conjugates) (m/z 465 →373)						
Liver (hydrolysis method)	0.01	3	83, 84, 93	87	6.3	
	0.05	3	81, 83, 74	79	5.9	
Kidney (hydrolysis method)	0.01	3	96, 95, 98	96	1.6	
	0.05	3	69, 61, 58	63	9.0	

Matrix	Fortification level (mg/kg)	Number of tests	% Recovery	Average % recovery	% RSD	Reference
AMPA (including conjugates) (m/z 435 → 373)						
Liver (hydrolysis method)	0.01	3	71, 68, 66	68	3.7	
	0.05	3	60, 68, 66	65	6.5	
Kidney (hydrolysis method)	0.01	3	62, 83, 79	75	15	
	0.05	3	30, 26, 60	39	48	
DAPA (including conjugates) (m/z 405 →353)						
Liver (hydrolysis method)	0.01	3	43, 35, 36	38	12	
	0.05	3	37, 34, 32	34	7.4	
Kidney (hydrolysis method)	0.01	3	17, 33, 43	31	42	
	0.05	3	12, 8, 7	9	2.6	

No consideration of the extraction efficiency of this method was provided.

First independent laboratory validation (ILV)

An independent laboratory validation of Method IB-2007-JLW-004-00-01 for residues of fluazinam, AMPA and DAPA in beef liver, fat and milk was conducted by Smith and Perez, 2009 [Ref: 2K8-ADPEN-023-0808], although alternations to the methods were required in order to achieve acceptable recoveries. The validation data for the unchanged method is presented in Table 72.

Table 72 Method IB-2007-JLW-004-00-01: Independent validation recovery rates for fluazinam and its metabolites in animal matrices (original method)

Matrix	Fortification level (mg/kg)	Number of tests	% Recovery	Average % recovery	% RSD	Reference
Fluazinam (m/z 465 → 373)						
Milk–Original method	0.01	5	109, 62, 74, 72, 62	76	25.5	2K8-ADPEN-023-0808
	0.1	5	76, 97, 111, 113, 118	103	16.5	
Fat-Original method	0.01	5	74, 62, 79, 60, 61	67	12.6	
	0.1	5	60, 59, 59, 61, 53	58	5.3	
Fat-Original method (repeated)	0.01	5	85, 85, 88, 69, 70	79	11.3	
	0.1	5	77, 91, 90, 60, 65	76	18.3	
Liver–Original method	0.01	5	70, 48, 80, 46, 61	62	23.7	
	0.1	5	34, 37, 25, 23, 37	31	20.8	
AMPA (435 → 373)						
Milk–Original method	0.01	5	79, 64, 58, 71, 65	67	11.6	2K8-ADPEN-023-0808
	0.1	5	77, 91, 88, 85, 96	87	8.2	
Liver–Original method	0.01	5	88, 77, 86, 82, 78	82	5.8	
	0.1	5	86, 97, 81, 67, 81	82	13.2	
DAPA (405–353)						
Milk–Original method	0.01	5	65, 63, 91, 78, 54	70	20.6	2K8-ADPEN-023-0808
	0.1	5	67, 28, 33, 41, 33	34	13.6	
Fat-Original method	0.01	5	76, 72, 88, 82, 13	66	46.3	
	0.1	5	27, 42, 88, 64, 19	48	58.7	
Fat -Original method (repeated)	0.01	5	79, 80, 78, 82, 83	80	2.5	
	0.1	5	62, 72, 80, 77, 80	74	10.2	
Liver–Original method	0.01	5	20, 47, 40, 39, 31	36	28.7	
	0.1	5	41, 46, 41, 4, 32	33	51.8	

The mean recoveries for milk for fluazinam, AMPA and DAPA and for fat for fluazinam and DAPA were generally within or just outside the range of 70-120% with relative standard deviations of <20%. In liver (without hydrolysis) mean recoveries for AMPA were within the range of 70-120% with relative standard deviations of <20%, however the mean recoveries for fluazinam and DAPA were in the range 31-62% with relative standard deviations of >20% (range of RSDs 21–52%).

The additional hydrolysis step (validation data reported in Table 73) did not lead to improved recovery data.

Table 73 Method IB-2007-JLW-004-00-01: Independent validation recovery rates for fluazinam and its metabolites including conjugates in liver (hydrolysis step)

Matrix	Fortification level (mg/kg)	Number of tests	% Recovery	Average % recovery	% RSD	Reference
Fluazinam (m/z 465 → 373)						
Liver (hydrolysis method)	0.01	5	30, 47, 33, 30, 39	36	20.4	2K8-ADPEN-023-0808
	0.1	5	32, 30, 37, 38, 42	36	13.4	
AMPA (m/z 435 → 373)						
Liver (hydrolysis method)	0.01	5	33, 39, 32, 45, 29	36	17.6	2K8-ADPEN-023-0808
	0.1	5	58, 27, 42, 49, 45	44	25.8	
DAPA (405 →353)						
Liver (hydrolysis method)	0.01	5	15, 14, 11, 12, 13	13	11.9	2K8-ADPEN-023-0808
	0.1	5	17, 7, 10, 15, 12	12	32.5	
Liver (hydrolysis method)-repeated	0.01	5	16, 5, 19, 11, 14	13	43.1	
	0.1	5	14, 11, 12, 9,7	10	24.5	

The following alternations to the method were made as a result of the poor recoveries:

For milk, samples were extracted with HCl and methanol in the presence of Celite 545 and filtered. The concentration and partitioning steps from the primary method were removed and the extract was made up to volume in methanol, filtered, then diluted in acetonitrile: water (1:1, v/v) for LC-MS/MS analysis for all compounds.

For liver, samples were extracted with acidified acetonitrile: water (3:1, v/v) in the presence of Celite 545 and filtered. The concentration and partitioning steps from the primary method were removed and the extract was diluted with acetonitrile: water (3:1, v/v) and filtered for LC-MS/MS analysis.

The validation data for the modified method is presented in table 74.

Table 74 Method IB-2007-JLW-004-00-01: Validation recovery rates for fluazinam and its metabolites in animal matrices for the modified method (method modifications made within the ILV)

Matrix	Fortification level (mg/kg)	Number of tests	% Recovery	Average % recovery	% RSD	Reference
Fluazinam (m/z 465 → 373)						
Milk–Modified method	0.01	5	98, 101, 97, 88, 94	95	5.3	2K8-ADPEN-023-0808
	0.1	5	73, 83, 79, 85, 81	80	5.5	
Liver–Modified method	0.01	5	113, 70, 81, 84, 84	86	18.6	
	0.1	5	82, 89, 90, 82, 92	87	5.4	
AMPA (435 → 373)						
Milk–Modified method	0.01	5	93, 91, 82, 94, 88	89	5.6	2K8-ADPEN-023-0808
	0.1	5	73, 85, 81, 77, 81	79	6.1	
Liver–Modified method	0.01	5	85, 117, 86, 91, 97	95	13.8	
	0.1	5	72, 90, 90, 76, 89	84	10.4	
DAPA (405 →353)						
Milk–Modified method	0.01	5	106, 34, 107, 80, 24	70	56.0	2K8-ADPEN-023-0808
	0.1	5	89, 120, 122, 55, 77	93	30.8	
Liver–Modified method	0.01	5	107, 109, 104, 101, 110	106	3.4	
	0.1	5	72, 77, 83, 79, 84	79	6.2	

The modified methods showed good linearity in the range of 0.1-40 ng/mL for all analytes (correlation coefficients >0.99) and no significant interferences at the relevant retention times.

The mean recoveries for milk and liver for fluazinam, AMPA and DAPA were within the range of 70-120% with relative standard deviations of <20%; with the exception of analysis of DAPA in milk where the relative standard deviations were

significantly higher than 20%. The ILV used LC-MS/MS for determination of DAPA residues and as already observed in the primary validation analysis of DAPA residue in milk by GC gives better recovery values.

Second ILV

Method IB-2007-JLW-004-00-01 was further independently validated in kidney, liver and fat by Schoenau, 2010 [Ref: 100342] using the modified extraction procedures for liver and kidney as given in the first ILV. Modifications to the original extraction procedures for fat were also made.

An outline of the method extraction is as follows:

Liver and kidney were extracted with acidified acetonitrile: water (3:1 v/v) in the presence of Celite 545 and filtered. In the primary method kidney was extracted using acidified acetonitrile: water (1:1 v/v). The concentration and partitioning steps from the primary method were removed and the extract was diluted with acetonitrile: water (3:1, v/v) and filtered for LC-MS/MS analysis.

Fat was extracted with acidified acetonitrile in the presence of Celite 545 and filtered. The extract was partitioned with acetonitrile saturated cyclohexane. The cyclohexane phase was discarded, and the acetonitrile evaporated to low volume, instead of near dryness as in the original method. The extract was filtered and diluted in acetonitrile: water (1:1, v/v). Analysis was by LC-MS/MS.

Quantitation and confirmation was performed using the following mass transitions for LC-MS/MS:

Analyte	Quantitation	Confirmation
Fluazinam	465→373	465→338
AMPA	435→373	435→354
DAPA	405→353	405→333

The modified method showed good linearity in the range of 0.1-40 ng/mL for all analytes (correlation coefficients >0.99) and no significant interferences at the relevant retention times. The mean recoveries for all matrices tested using the modified methods at all fortification levels ranged from 74 to 94% (RSDs <20%) with the exception of kidney for fluazinam where the RSD was 23%. The limits of quantitation (LOQs) were 0.01 mg/kg for all matrices tested.

Table 75 Method IB-2007-JLW-004-00-01: Independent validation recovery rates for fluazinam and its metabolites in animal matrices for the modified extraction procedure

Matrix	Fortification level (mg/kg)	Number of tests	% Recovery	Average % recovery	% RSD	Reference
Fluazinam m/z 465→373						
Liver	0.01	5	62, 88, 79, 70, 72	74	13.2	100342
	0.1	5	100, 98, 84, 92, 93	93	6.5	
Kidney	0.01	5	99, 88, 74, 76, 52	78	22.7	
	0.1	5	98, 88, 90, 86, 93	91	5.4	
Fat	0.01	5	87, 93, 92, 85, 92	90	3.8	
	0.1	5	88, 88, 92, 93, 87	90	3.3	
Fluazinam m/z 465→338						
Liver	0.01	5	52, 85, 83, 76, 79	75	17.8	100342
	0.1	5	101, 96, 84, 96, 88	93	7.5	
Kidney	0.01	5	94, 90, 80, 79, 55	80	19.1	
	0.1	5	99, 85, 89, 80, 94	89	8.3	
Fat	0.01	5	85, 93, 88, 86, 90	89	3.6	
	0.1	5	85, 86, 91, 92, 85	88	4.1	
AMPA m/z 435→373						
Liver	0.01	5	87, 88, 87, 90, 87	88	1.5	100342
	0.1	5	93, 94, 93, 96, 94	94	1.3	
Kidney	0.01	5	92, 95, 92, 88, 86	91	3.9	
	0.1	5	96, 94, 95, 90, 96	94	2.6	
Fat	0.01	5	86, 91, 87, 86, 87	88	2.6	
	0.1	5	94, 93, 94, 98, 89	94	3.5	
AMPA m/z 435→354						
Liver	0.01	5	91, 92, 90, 90, 86	90	2.5	100342
	0.1	5	91, 92, 92, 92, 94	92	1.1	

Matrix	Fortification level (mg/kg)	Number of tests	% Recovery	Average % recovery	% RSD	Reference
Kidney	0.01	5	94, 94, 92, 91, 86	91	3.6	
	0.1	5	95, 92, 96, 86, 96	93	4.7	
Fat	0.01	5	86, 92, 88, 87, 90	89	2.8	
	0.1	5	94, 92, 93, 95, 90	93	2.3	
DAPA m/z 405→353						
Liver	0.01	5	80, 79, 86, 81, 79	81	3.5	100342
	0.1	5	81, 78, 74, 78, 77	78	3.5	
Kidney	0.01	5	97, 89, 92, 87, 89	91	4.1	
	0.1	5	94, 95, 96, 90, 94	94	2.4	
Fat	0.01	5	83, 86, 82, 83, 83	84	1.4	
	0.1	5	90, 91, 89, 94, 86	90	3.3	
DAPA m/z 405→333						
Liver	0.01	5	79, 78, 84, 83, 80	81	3.2	100342
	0.1	5	83, 79, 75, 81, 80	80	3.5	
Kidney	0.01	5	94, 92, 88, 88, 90	91	2.8	
	0.1	5	93, 96, 93, 91, 97	94	2.4	
Fat	0.01	5	84, 85, 83, 83, 85	84	1.1	
	0.1	5	90, 91, 92, 92, 89	91	1.4	

Stability of residues in stored analytical samples

The meeting received freezer storage stability data for fluazinam in a variety of plant and animal matrices. The meeting also received data to support the stability of AMGT and AMPA in various matrices.

Plant commodities

Coffee, potato, onion, grape and wine

Samples of coffee, potato, onion and grape were homogenised, fortified at 0.1 mg/kg with fluazinam and stored frozen (<-15 °C). Samples of wine were fortified at 0.05 mg/L. Samples were analysed at different time points for up to two years after storage.

At each time point samples were freshly fortified with fluazinam to serve as procedural recovery samples. After extraction the methanol extracts were stored at < 8 °C for various periods of time prior to analysis. Final determination was achieved using analytical method 2. The results are summarised in Table 76.

Table 76 Storage stability data for fluazinam residues in frozen plant matrices

Matrix	Sample storage interval (days)	Storage interval of extracts (days)	% remaining after storage	Procedural recoveries (%)
Grape	5	13	91, 126, 99 (105)	Recoveries were undertaken in the range of 0.1–0.2 mg/kg (0.05 - 0.2 mg/L for wine) at each time point for each crop. The recoveries obtained were all acceptable [a]
	113	10	111, 111 (111)	
	243	8	92, 109 (101)	
	428	8	95, 94 (95)	
	786	4	111, 119 (115)	
Potato	1	10	118, 117 (118)	
	118	1	81, 87 (84)	
	218	1	83, 95 (89)	
	363	15	89, 93 (91)	
	758	14	73, 78 (76)	
Onion	5	6	97, 105 (101)	
	118	16	77, 77 (77)	
	218	1	98, 112 (105)	
	481	1	74, 88 (81)	
	768	1	93, 83 (88)	
Coffee	1	10	109, 108 (109)	
	107	25	101, 100 (101)	
	217	8	88, 98 (93)	
	425	37	107, 99 (103)	
	790	8	99, 100 (100)	
Wine	5	10	80, 78 (79)	
	111	0	92, 100 (96)	

Matrix	Sample storage interval (days)	Storage interval of extracts (days)	% remaining after storage	Procedural recoveries (%)
	222	3	100, 101 (101)	
	377	1	90, 100 (95)	
	777	7	94, 92 (93)	

Values in parentheses = mean recovery of stored samples

[a] Based on data presented it is not possible to determine which procedural recoveries relate to specific time points.

Residues of fluazinam were shown to be stable in potatoes (high starch content commodity) for up to 25 months, in coffee (high oil content commodity), onion (high water content commodity), grapes (high acid content commodity) and wine for up to 26 months after frozen storage.

Grapes

Samples of untreated homogenised grapes were fortified with fluazinam at 0.25 mg/kg. Control matrices/procedural recovery samples were also prepared. Samples were analysed after 0, 31, 63, 94 and 183 days of frozen storage.

At each time point freshly fortified samples were fortified with fluazinam to serve as procedural recovery samples. Final determination was achieved using analytical method 1. The results are summarised in Table 77.

Table 77 Storage stability data for fluazinam residues in frozen grapes

Matrix	Sample storage interval (days)	% Remaining after storage	Procedural recoveries (%)
Grapes	0	96, 96, 104 (99)	108, 104
	31	92, 96, 88 (92)	84, 100
	63	112, 108, 104 (108)	112, 112
	94	100, 92, 104 (99)	104, 100
	183	112, 88, 88 (96)	88, 100

Values in parentheses = mean recovery of stored samples

Residues of fluazinam were shown to be stable in grapes (high acid content commodity) for at least 6 months in freezer storage.

Potatoes and processed fractions of potatoes

Samples of whole potatoes were chopped and homogenised with dry ice pellets using a Hobart chopper. Potato chips were crushed and mixed by hand. Potato wet peels and granules were mixed by hand to homogenise. Samples were fortified with fluazinam at 0.5 mg/kg and stored frozen for up to three years (-18°C to -20°C). At specified sampling intervals, four replicate fortified (stored) samples were analysed for residues of fluazinam along with one control sample and two concurrent (fresh) fortification samples. Final determination was achieved using analytical method 1. The results are summarised in Table 78.

Table 78 Storage stability data for fluazinam residues in frozen potato and processed fractions

Matrix	Sample storage interval (days)	% Remaining after storage	Procedural recoveries (%)
Potato	0	110, 110, 108, 100 (107)	116, 106
	1	92, 98, 96, 100 (97)	100, 92
	21	96, 98, 102 , 96 (98)	108, 114
	49	90, 92, 90, 86 (90)	100, 122
	90	88, 84, 84, 88 (87)	112, 108
	181	98, 100, 104, 102 (101)	112, 118
	363	78, 78, 82, 82 (80)	106, 108
	547	72, 74, 66, 74 (72)	108, 110
	767	72, 70, 70, 64 (69)	102, 102
	924	64, 50, 60, 72 (59)	98, 94
	1096	62, 64, 68, 56 (63)	104, 108
Potato chips	0	108, 90, 94, 86 (97)	90, 100
	1	88, 94, 90, 94 (92)	90, 94
	21	78, 84, 86, 90 (85)	84, 78
	49	98, 86, 90, 84 (90)	124, 102
	91	74, 76, 76, 76 (76)	82, 78
	182	78, 86, 88, 92, (86)	114, 114

Matrix	Sample storage interval (days)	% Remaining after storage	Procedural recoveries (%)
	365	72, 66, 64, 66 (67)	72, 76
	546	84, 62, 62, 66 (69)	90, 84
	764	88, 82, 86, 82 (85)	92, 88
	912	68, 62, 64, 66 (65)	72, 74
	1146	72, 76, 74, 76 (75)	94, 96
Potato granules	0	112, 122, 124 (119)soil	118, 114
	1	90, 92, 96, 92 (93)	94, 96
	21	116, 106, 98, (105)	104, 110
	49	66, 66,66, 62 (65)	102, 104
	57	92, 92, 90, 90 (91)	98, 106
	91	64, 64, 64, 66 (65)	116, 116
	99	66, 64, 72, 66 (67)	100, 106
	183	48, 44, 46, 53 (48)	100, 100
	196	38, 40, 40, 40 (40)	92, 90
	364	58, 60, 64, 64 (62)	106, 100
	547	30, 28, 34, 28, (30)	100, 104
	766	54, 46, 54, 52 (52)	90, 88
	910	44, 56, 62, 56 (55)	112, 100
	1094	36, 38, 34, 34, (36)	114, 124
Wet potato peels	0	84, 86, 79, 78 (82)	84, 88
	1	86, 92, 86, 86 (88)	84, 94
	21	76, 80, 72, 72 (75)	82, 84
	50	78, 76, 84, 84 (81)	86, 88
	91	70, 70, 64, 66 (68)	82, 94
	138	58, 56, 54, 52 (55)	92, 90
	182	84, 70, 68, 76 (75)	90, 94
	365	70, 74, 74, 72 (73)	108, 112
	534	62, 68, 52, 62 (61)	104, 106
	728	66, 64, 70, 80 (70)	116, 118
	917	64, 52, 54, 66, (59)	110, 116
	1107	54, 56, 70, 54 (59)	100, 104

Values in parentheses = mean recovery of stored samples

Residues of fluazinam were shown to be stable in potatoes (high starch content commodity) for up to 26 months, in potato chips for up to 38 months and in potato granules for up to 2 months. With respect to wet potato peel, the data shows a degradation at 91 days and 138 days. The recoveries then obtained from the stored samples after 182 days and 365 days were above 70% before a decline was seen after this time period. Overall the data supports the stability for a 12 month period.

Grapes

Samples of untreated homogenised grapes and wine were fortified separately with fluazinam, AMGT and AMPA (wine only) at 0.5 mg/kg. Samples were stored frozen (-22 to -12 °C) and analysed after intervals up to 36 months of storage. At each sampling point, four stored samples were analysed along with a control and two untreated crop samples freshly fortified with analytes at 0.5 mg/kg to act as procedural recoveries. Final determination was achieved using analytical method 1. The results are summarised in Tables 79, 80 and 81 for fluazinam, AMGT and AMPA respectively.

Table 79 Storage stability data for fluazinam residues in grapes and wine

Matrix	Sample storage interval (days)	% Remaining after storage	Procedural recoveries (%)
Red grapes	0	92, 92, 92, 92 (92)	88, 86
	1	92, 90, 78, 84 (86)	90, 82
	21	92, 90, 78, 84 (86)	100, 102
	49	94, 96, 86, 76 (88)	90, 84
	90	84, 70, 66, 82 (76)	72, 70
	181	92, 94, 92, 98 (94)	96, 84
	363	86, 88, 84, 90 (87)	98, 90
	553	110, 108, 96, 102 (104)	116, 108
	714	82, 78, 70, 84 (79)	88, 86
	918	86, 90, 90, 88 (89)	90, 94

Matrix	Sample storage interval (days)	% Remaining after storage	Procedural recoveries (%)
	1148	80, 90, 80, 84 (84)	82, 82
White grapes	0	88, 86, 90, 88 (88)	92, 84
	1	88, 88, 88, 90 (89)	90, 86
	21	88, 90, 96, 90 (91)	88, 104
	50	84, 90, 84, 82 (85)	86, 92
	91	88, 86, 76, 86 (84)	86, 84
	182	94, 94, 102, 102 (98)	96, 98
	361	98, 86, 100, 104 (97)	96, 108
	550	92, 98, 102, 100 (98)	96, 98
	709	86, 92, 88, 92, (90)	90, 86
	914	92, 88, 92, 92 (91)	92, 96
	1144	90, 96, 90, 96 (93)	88, 96
Red wine	0	104, 100, 104, 106 (104)	100, 102
	1	102, 102, 102, 106 (103)	102, 102
	21	102, 100, 100, 104 (102)	108, 106
	49	82, 90, 112, 90 (94)	108, 98
	91	90, 88, 100, 88, (92)	90, 78
	183	116, 112, 118, 118 (116)	116, 116
	357	116, 112, 116, 118 (116)	110, 118
	553	98, 96, 100, 92 (97)	96, 100
	721	94, 94, 94, 100 (96)	98, 100
	920	98, 94, 102, 90 (96)	98, 100
	1155	90, 96, 92, 96 (94)	96, 94
White wine	0	104, 102, 104. 98 (102)	102, 100
	1	108, 106, 108, 108 (108)	106, 106
	21	104, 102, 102, 102 (103)	106, 106
	49	90, 98, 110, 110 (102)	106, 90
	90	96, 94, 108,114 (103)	124, 92
	182	108, 104, 110, 108 (108)	112, 106
	354	92, 104, 100, 98 (99)	108, 100
	550	100, 94, 94, 100 (97)	98, 92
	718	90, 92, 88, 94 (91)	96, 92
	917	96, 104, 104 (101)	94
	1152	92, 98, 96, 98 (96)	88, 88

Values in parentheses = mean recovery of stored samples

Table 80 Storage stability data for AMGT residues in grapes and wine

Matrix	Sample storage interval (days)	% Remaining after storage	Procedural recoveries (%)
Red grapes	0	86, 90, 92, 92 (90)	84, 90
	1	82, 90, 94, 90 (89)	106, 98
	21	84, 90, 92, 94 (90)	94, 102
	49	98, 98, 98, 94 (97)	98, 108
	90	98, 110, 124, 110 (111)	112, 114
	181	92, 90, 82, 92 (89)	114, 106
	370	86, 88, 92, 74 (85)	96, 96
	615	74, 74, 72, 74 (74)	92, 94
	728	76, 82, 80, 76 (79)	88, 92
	930	74, 72, 72, 74 (73)	82, 82
	1162	84, 82, 86, 86 (85)	96, 98
White grapes	0	94, 94, 90, 90 (92)	92, 94
	1	98, 102, 94, 100 (99)	90, 106
	21	80, 88, 60, 80 (77)	92, 88
	50	90, 94, 98, 100 (96)	102, 104
	91	80, 84, 88, 72 (81)	94, 92
	182	72, 92, 74, 88 (82)	102, 96
	367	70, 80, 74, 74 (75)	82, 82
	616	66, 68, 72, 62 (67)	90, 90

Matrix	Sample storage interval (days)	% Remaining after storage	Procedural recoveries (%)
	724	78, 78, 76 74 (77)	88, 84
	927	62, 74, 72, 70 (70)	78, 76
	1155	74, 80, 82, 76 (78)	96, 98
Red wine	0	94, 92, 90, 108 (96)	88, 88
	1	92, 96, 94, 96 (95)	98, 94
	21	78, 82, 80, 92 (83)	98, 08
	49	94, 96, 88, 88 (92)	102, 106
	91	84, 86, 86, 84 (85)	92, 92
	183	90, 94, 84, 80 (87)	106, 90
	380	80, 80, 82, 78 (80)	88, 94
	563	78, 78, 80, 74 (78)	104, 100
	728	96, 94, 98 (96)	106
	934	82, 76, 74, 74 (77)	90, 84
	1165	86, 90, 92, 90 (90)	98, 112
White wine	0	92, 84, 98 (91)	96, 80
	1	62, 76, 82, 84 (76)	66, 74
	21	88, 92, 98, 92 (93)	100, 94
	49	100, 100, 106, 105 (103)	82, 108
	90	84, 96, 98, 98 (94)	98, 94
	181	92, 92, 96, 98 (95)	98, 98
	375	94, 96, 96, 98 (96)	92, 86
	557	78, 80, 78, 78 (79)	88, 84
	728	94, 90, 88, 106 (95)	98, 100
	935	90, 80, 88, 84 (86)	84, 88
	1163	96, 92, 92, 92 (93)	98, 94

Values in parentheses = mean recovery of stored samples

Table 81 Storage stability data for AMPA residues in wine

Matrix	Sample storage interval (days)	% Remaining after storage	Procedural recoveries (%)
Red wine	0	104, 104, 108, 104 (105)	102, 106
	1	104, 108, 108, 110 (108)	104, 106
	21	100, 106, 106, 104 (104)	98, 100
	49	100, 114, 112, 112 (110)	102, 104
	90	82, 96, 90, 96 (91)	92, 88
	186	114, 114, 110, 114 (113)	114, 112
	362	98, 100, 98, 100 (99)	96, 98
	553	80, 92, 100, 92 (91)	98, 98
	727	92, 84, 86, 76 (85)	80, 90
	901	110, 118, 116, 118, (116)	108, 112
	1142	98, 98, 104, 98 (100)	100, 100
White wine	0	106, 108, 106, 108 (107)	106, 104
	1	108, 110, 112, 112 (111)	102, 102
	21	98, 98, 100, 102 (100)	90, 96
	49	88, 98, 84, 100 (93)	94, 98
	90	80, 109, 96, 96 (95)	90, 94
	181	100, 102, 108,100 (103)	98, 102
	357	98, 102, 106, 107 (103)	100, 102
	549	90, 86, 96, 86 (90)	90, 94
	722	88, 86, 80, 96 (88)	84, 94
	896	102, 100, 106, 106 (104)	102, 102
	1140	94, 96, 104, 104 (100)	94, 92

Values in parentheses = mean recovery of stored samples

Residues of fluazinam were shown to be stable in grapes (high acid content commodity) and wine for at least 38 months after frozen storage. Residues of AMGT were shown to be stable in grapes (high acid content commodity) and wine for at least 39 months after frozen storage. Residues of AMPA were shown to be stable in wine for at least 38 months after frozen storage.

Peanut nutmeats

Samples of untreated peanut nutmeat were fortified with fluazinam at 0.25 mg/kg. Samples were stored frozen (-23 to -12 °C) and analysed at different intervals up to 190 days of storage. At each sampling point, three stored samples were analysed along with a control and two untreated crop samples freshly fortified with fluazinam at 0.25 mg/kg to act as procedural recoveries. Final determination was achieved using analytical method 1. The results are summarised in Table 82.

Table 82 Storage stability data for fluazinam residues in peanut nutmeats

Matrix	Sample storage interval (days)	% Remaining after storage	Procedural recoveries (%)
Peanut nutmeats	0	104, 88, 80 (91)	92, 92
	29	76, 72, 88 (79)	84, 68
	60	84, 76, 88 (83)	84, 96
	88	80, 84, 80 (81)	96, 104
	102	88, 88, 80 (85)	100, 104
	190	80, 76, 84 (80)	100, 108

Values in parentheses = mean recovery of stored samples

Residues of fluazinam were shown to be stable in peanut nutmeat (high oil content commodity) for at least 6 months in freezer storage.

Peanuts and processed fractions

Homogenised samples of peanut nutmeat, hay, hull, meal and oil were fortified with fluazinam at 0.5 mg/kg (0.4 mg/kg for peanut oil). Samples were stored frozen (-22 to -12 °C) and analysed after different intervals up to 36 months of storage. At each sampling point, four stored samples were analysed along with a control and two untreated crop samples freshly fortified with analytes at 0.5 mg/kg to act as procedural recoveries. Final determination was achieved using analytical method 1. The results are summarised in Table 83.

Table 83 Storage stability data for fluazinam residues in peanut fractions

Matrix	Sample storage interval (days)	% Remaining after storage	Procedural recoveries (%)
Peanut hay	0	84, 86, 86, 84 (85)	84, 86
	1	88, 88, 88, 92 (89)	85, 85
	21	88, 84, 86, 86 (86)	77, 80
	49	84, 78, 82, 80 (81)	78, 81
	91	92, 88, 90, 90 (90)	80, 87
	184	84, 84, 80, 86 (84)	84, 88
	366	78, 80, 80, 86 (81)	77, 77
	548	80, 80, 82, 88 (83)	91, 86
	752	90, 84, 86, 88 (87)	96, 92
	924	84, 86, 74,8 8 (83)	95, 95
	1164	72, 68, 66, 70 (69)	76, 68
Peanut hulls	0	90, 92, 92, 94 (92)	103, 91
	1	102, 100, 102, 100 (101)	94, 97
	21	98, 100, 102, 102 (101)	98, 97
	49	92, 92, 94, 90 (92)	95, 92
	91	102, 102,100,98 (101)	105, 104
	181	98, 94, 94, 84 (93)	95, 94
	364	90, 92, 92, 90 (91)	86, 94
	545	86, 86, 90, 90 (88)	91, 105
	747	98, 100, 100, 100 (100)	107, 110
	916	80, 84, 88, 86 (85)	93, 93
	1156	76, 80, 82, 80 (80)	91, 91
Peanut nutmeats	0	76, 74, 72, 70 (73)	81, 61

Matrix	Sample storage interval (days)	% Remaining after storage	Procedural recoveries (%)
	1	92, 94, 90, 98 (94)	95, 96
	21	90, 88, 84, 88 (88)	91, 85
	49	88, 86, 86, 88 (87)	89, 91
	89	90, 86, 90, 92 (90)	83, 92
	181	88, 90, 86, 92 (89)	85, 90
	368	84, 84, 84, 86 (85)	87, 90
	550	86, 78, 86, 80 (83)	93, 98
	753	92, 88, 82, 86 (87)	85, 89
	924	76, 80, 76, 76 77)	90, 90
	1167	64, 68, 74, 68 (69)	86, 86
Peanut meal	0	92, 86, 82, 86 (87)	86, 86
	1	78, 78, 80, 80 (79)	83, 83
	21	70, 74, 70, 68 (71)	82, 83
	49	56, 54, 54, 60 (56)	81, 74
	71	54, 58, 54, 54 (55)	83, 70
	90	58, 60, 48, 54 (55)	80, 71
	183	48, 50, 42, 78 (55)	79, 80
	364	44, 48, 50, 50 (48)	77, 77
	546	42, 36, 40, 36 (39)	80, 76
	743	70, 68 ,62, 70 (68)	100, 105
	916	44, 46, 44, 36 (43)	81, 72
	1151	40, 32, 26, 32 (33)	77, 77
Peanut oil	0	85, 98, 98, 95 (94)	90, 93
	1	90, 95,95,102.5 (96)	92, 97
	21	102.5, 103,103,103 (103)	109, 109
	49	100, 100, 93, 100 (98)	95, 92
	90	100, 108, 108, 105 (105)	113, 117
	181	90, 88, 90, 90 (89)	102, 97
	365	93, 93, 95, 95 (94)	103, 100
	547	95, 85, 80, 85 (87)	95, 92
	738	90, 90, 93, 98 (93)	102, 105
	910	88, 88, 88, 88 (88)	103, 103
	1147	83, 78, 78, 78 (79)	94, 99

Values in parentheses = mean recovery of stored samples

Residues of fluazinam were shown to be stable in peanut nutmeat (high oil content commodity), hulls, and hay for at least 39 months, in peanut oil for at least 38 months and in peanut meal for up to 21 days after frozen storage.

Apple and processed commodities

Samples of apples were chopped and homogenised using a Hobart chopper. The homogenised apple, wet apple pomace and apple juice samples were fortified with fluazinam and AMGT separately at 0.5 mg/kg and stored frozen for up to three years (-18 °C to -20 °C). At specified sampling intervals, four replicate fortified (stored) samples were analysed for residues of fluazinam along with one control sample and two concurrent (fresh) fortification samples. Final determination was achieved using analytical method 1. The results are summarised in Tables 84 and 85 for fluazinam and AMGT respectively.

Table 84 Storage stability data for fluazinam in apples and processed apple fractions

Matrix	Sample storage interval (days)	% Remaining after storage	Procedural recoveries (%)
Apple	0	90 ,84 ,94 ,82(88)	104, 92
	1	86 ,82 ,92(87)	90, 104
	21	100 ,100 ,92 ,92 (96)	94, 110
	57	96 ,92 ,86 ,88(90.5)	88, 98
	93	114 ,108 ,112 ,118 (113)	120, 118
	181	98 ,100 ,108 ,98 (101)	108, 108
	366	88 ,92 ,92 ,90 (91)	96, 98
	547	106 ,94 ,104 ,98 (101)	100, 98
	733	116 ,114 ,114 ,104 (112)	124, 114

Matrix	Sample storage interval (days)	% Remaining after storage	Procedural recoveries (%)
	915	106 ,114 ,106 ,106 (108)	114, 100
	1097	92 ,102 ,100 ,98 (98)	118, 112
Apple juice	0	76 ,82 ,86 ,90 (85)	78, 70
	1	92 ,90 ,90 ,86 (90)	98, 98
	21	78 ,84 ,86 ,84 (83)	90, 90
	50	96 ,86 ,88 ,88(90)	92, 104
	92	104 ,96 ,98 ,96 (99)	100, 104
	182	102 ,104 ,102 ,102 (103)	102, 100
	364	82 ,92 ,90 ,86 (88)	88, 90
	546	86 ,80 ,90 ,84(85)	98, 84
	731	104 ,102 ,106 ,110 (106)	96, 98
	912	92 ,100 ,98 ,104 (99)	104, 88
	1094	92 ,88 ,94 ,92 (92)	92, 94
Apple wet pomace	0	118 ,110 ,122 (117)	114, 122
	1	114 ,114 ,110 ,108 (112)	110, 116
	21	84 ,88 ,88 ,78 (85)	86, 86
	49	100 ,88 ,94 ,96(95)	92, 100
	92	76 ,86 ,94 ,90 (87)	92, 92
	181	100 ,96 ,96 ,98 (98)	100, 108
	363	84 ,92 ,88 ,86 (88)	80, 78
	546	90 ,86 ,82 ,88 (87)	92, 88
	729	118 ,118 ,126 ,120 (121)	112, 112
	916	110 ,108 ,114 ,108 (110)	114, 118
	1096	88 ,88 ,86 ,86 (87)	90, 90

Values in parentheses = mean recovery of stored samples

Table 85 Storage stability data for AMGT in apples and processed apple fractions

Matrix	Sample storage interval (days)	% Remaining after storage	Procedural recoveries (%)
Apple	0	80, 88, 90, 82 (85)	86, 88
	1	112, 96, 94, 88 (98)	100, 92
	21	88, 100, 80, 98 (92)	96, 84
	49	98, 90, 90, 106 (96)	100, 94
	90	60, 52, 70, 68 (63)	86, 86
	94	96, 100, 100, 110 (102)	112, 108
	180	86, 82, 86, 78 (83)	106, 86
	360	74, 74, 74, 76 (75)	84, 72
	559	68, 68, 62, 68 (67)	88, 90
	566	66, 66, 64, 68 (66)	94, 90
	714	76, 70, 70, 70 (72)	92, 104
	932	62, 78, 66, 70 (69)	90, 104
	1108	62, 70, 64, 68 (66)	88, 104
Apple juice	0	98, 94, 86, 88 (92)	96, 96
	1	94, 98, 98, 104 (99)	98, 90
	21	98, 94, 90, 92 (94)	92, 94
	49	100, 88, 104, 96 (97)	64, 80
	90	76, 82, 82, 82 (81)	82, 84
	180	100, 98, 84, 106 (97)	116, 108
	360	90, 80, 88, 88 (87)	90, 88
	581	82, 78, 80, 70 (78)	92, 90
	735	70, 68, 62, 70 (68)	74, 74
	960	68, 76, 66, 72 (71)	80, 102
	1114	66, 60, 62, 64 (63)	70, 90
Apple wet pomace	0	84, 76, 84, 72(79)	74, 78
	1	88, 78, 86, 78 (83)	88, 90
	21	80, 76, 74, 78 (77)	78, 80
	49	76, 68, 64, 76 (71)	78, 82

Matrix	Sample storage interval (days)	% Remaining after storage	Procedural recoveries (%)
	90	72, 72, 72, 72 (72)	92, 84
	180	52, 80, 84, 46 (66)	104, 92
	360	62, 62, 60, 64 (62)	74, 72
	577	48, 54, 56, 50 (52)	84, 96
	584	52, 50, 50, 56 (52)	80, 94
	742	68, 76, 72, 58 (69)	92, 86
	959	52, 60, 50, 50 (53)	82, 94
	1128	28, 26, 28, 26(27)	88, 92

Values in parentheses = mean recovery of stored samples

Residues of fluazinam were shown to be stable in apple (high water content commodity) and apple wet pomace for at least 37 months and in apple juice for at least 36 months after frozen storage. Residues of AMGT were shown to be stable in apple (high water content commodity) up to 31 months, in apple juice for up to 32 months and in apple wet pomace for up to 3 months after frozen storage.

Various crops

The stability of residues in a number of crops were investigated within the residue trial studies.

The homogenised crop samples were fortified with fluazinam and stored frozen. Stored samples were analysed along with control and concurrent recovery samples.

In some cases the stability of AMGT was also investigated:

- For blueberries separate samples were fortified with fluazinam and AMGT
- For Cabbage (heads with wrapper leaves) samples were fortified with fluazinam and AMGT using a mixed fortification standard
- For cantaloupe samples were fortified with fluazinam and AMGT using a mixed fortification standard
- Cucumbers samples were fortified with fluazinam and AMGT using a mixed fortification standard
- For summer squash samples were fortified with fluazinam and AMGT using a mixed fortification standard
- For pepper samples were fortified with fluazinam and AMGT using a mixed fortification standard
- For soya bean separate samples were fortified with fluazinam and AMGT
- In the majority of cases the initial fortification levels in the crops were not verified by the analysis of a time zero sample.

The results are summarised in Tables 86 and 87 for fluazinam and AMGT respectively.

Table 86 Storage stability data for fluazinam in various crops

Matrix	Analytical method number	Storage temperature [b] (°C)	Fortification level (mg/kg)	Sample storage interval (days)	Stored recoveries (%)	Procedural recoveries (%)
Blueberries	1	-21	0.1	203	98, 99, 84 (94)	100, 100
Onion	1	-40 to -6	1	429	99, 94, 100 (98)	114
Broccoli	1	-21	0.1	182†	39, 52, 50 (47)	43, 46, 38,77, 72,73,49,82
				205†	66, 68, 68 (67)	88, 72, 90
Broccoli	1	-20	0.1	232	50, 52, 42 (52)	67
Cabbage	1	-22 to -4	0.1	560	26, 27, 33 (29)	99
Cabbage head with wrapper leaves	1	-22 to -4	0.1	0	77, 76, 77 (77)	72
				1	76, 79, 70 (75)	89
				4	72, 69, 62 (68)	85
				7	64, 59, 63 (59)	72
				10	61, 58, 53(57)	77
				14	62, 57, 56 (58)	72
				21	55, 55, 53 (54)	70
				28	59, 62, 60 (60)	82

Matrix	Analytical method number	Storage temperature [b] (°C)	Fortification level (mg/kg)	Sample storage interval (days)	Stored recoveries (%)	Procedural recoveries (%)
				35	46, 47, 47 (47)	72
				42	58, 53, 55 (55)	77
				65	46, 40, 44 (43)	71
				95	59, 44, 54 (52)	86
				125	76, 78, 69 (74)	107
				155	81, 79, 87 (82)	115
				185	54, 54, 54 (54)	75
Cabbage head with wrapper leaves	3	-20	0.1	435	69, 71, 70 (70)	88, 86
Cantaloupe	10	-20	0.1	1184	78, 77, 75 (77)	69, 68
Cucumber	10	-20	0.1	477	80, 84, 86 (83)	91, 90
Summer squash	10	-20	0.1	425	70, 72, 72 (72)	90, 89
Pepper	10	-20	1	1241	61, 73, 70 (68)	See table 67 [c]
Mustard greens	1	-22 to -4	0.1	590	44, 52, 40 (45)	See table 65 [c]
Lettuce	1	-40 to 0	1	414	75, 70 (72)	118
				419	85, 87 (86)	117
Succulent bean (bean without a pod)–snap bean	1	-38 to -1	1	377	59, 58, 69 (62)	75
Lima bean	1	-38 to -1	1	455	51, 46, 57 (51)	78
Soya bean forage	10	<-10	0.1	0	97, 103, 96 (98)	91, 114
				37	79, 86, 72 (79)	89, 101
				62	76, 76, 67 (73)	82, 102
				125	84, 86, 104 (91)	109, 107
				153	82, 87, 102 (90)	102, 106
Soya bean hay	10	<-10	0.1	0	120, 119, 107 (115)	116, 120
				37	72, 86, 70 (76)	85, 92
				62	73, 68, 77 (73)	106, 116
				125	69, 79, 82 (77)	107, 95
				153	78, 81, 80 (79)	108, 97
Soya bean seed	10	<-10	0.1	0	85, 87, 86 (86)	84, 83
				37	78, 73, 87 (79)	105, 119
				62	76, 65, 65 (69)	91, 105
				125	82, 81, 80 (81)	105, 95
				153	74, 73, 73 (73)	106, 88
Bean (dry)	1	-22 to -4	1	307	58, 46, 59 (54)	96
Carrot	1	-22 to -4	1	469	79, 70, 80 (76)	83, 105
Ginseng	1	-21	1	332	49, 46, 42 (46)	75, 75, 80
				344	64, 59, 56 (60) [a]	77, 76, 78

Values in parentheses = mean recovery of stored samples

[a] Storage stability repeated for broccoli as first set of data at 182 days showed poor recoveries from the storage and freshly prepared samples. The extraction procedure of the analytical method was modified slightly for the samples analysed at 205 days. Storage stability was also repeated for ginseng due to the low recoveries obtained in the first set of data.

[b] The trial samples were subject to the same temperature ranges during storage prior to analysis

[c] Freshly fortified samples were not prepared for peppers and mustard greens. The recovery data generated for the analytical methods were generated at the same time as the analysis of the stored samples. The data in the tables indicated are applicable.

Table 87 Storage stability data for AMGT in various crops

Matrix	Analytical method Number	Storage temperature [a] (°C)	Fortification level (mg/kg)	Sample storage interval (days)	Stored recoveries (%)	Procedural recoveries (%)
Blueberries	1	-21	0.1	251-259	70, 140, 34, 110, 60 (89)	120, 65, 94, 62, 70, 64
			0.2	219	75, 75, 75 (75)	85
Cabbage head with wrapper leaves	3	-20	0.1	435	54, 53, 52 (53)	93, 94
Cantaloupe	3	-20	0.1	1035	86, 85, 82 (84)	98, 72
				1055	78, 81, 88 (82)	70
Cucumber	3	-20	0.1	477	84, 88, 92 (88)	98, 97
Summer squash	3	-20	0.1	425	79, 86, 81 (82)	96, 90
Pepper	3	-20	1	1106	72, 78, 74 (75)	See Table 67 [b]
Soya bean forage	3	<-10	0.1	0	108, 120, 118 (115)	98, 104
				37	87, 87, 79 (84)	1115, 112
				62	93, 86, 82 (87)	104, 111
				125	84, 81, 93 (85)	108, 110
				153	86, 88, 95 (89)	113, 110
Soya bean hay	3	<-10	0.1	0	118, 116,118 (117)	87, 98
				37	87, 77, 74 (76)	109, 111
				62	77, 71, 76 (75)	97, 112
				125	86, 86, 85 (85)	102, 98
				153	80, 91, 82 (84)	111, 101
Soya bean seed	3	<-10	0.1	0	119, 118, 119 (118)	87, 104
				37	71, 76, 71 (72)	100, 118
				62	54, 57, 59 (56)	98, 111
				125	73, 75, 72 (73)	113, 98
				153	75, 70, 72 (94)	89, 98

Values in parentheses = mean recovery of stored samples

[a] The trial samples were subject to the same temperature ranges during storage prior to analysis

[b] Freshly fortified samples were not prepared for peppers. The recovery data generated for the analytical methods were generated at the same time as the analysis of the stored samples. The data in the tables indicated are applicable.

Tea

Separate samples of non-milled untreated dried tea leaves were fortified with fluazinam, MAPA and HYPA at 2.0 mg/kg and stored frozen (-20 °C). Stored samples were analysed along with control samples. No procedural recoveries were analysed. Final determination was achieved using analytical methods 1 or 12. The results are summarised in Table 88.

Table 88 Storage stability data for tea

Analyte	Sample storage interval (days)	Stored recoveries (%)	Procedural recoveries (%)
Fluazinam	32	77, 70 (74)	No procedural recovery samples were given
	77	87, 79 (84)	
	156	89, 88, 94, 86, 97, 95 (92)	
MAPA	32	77, 76 (77)	
	77	86, 81 (84)	
	156	75, 72, 72, 70, 74, 73 (73)	
HYPA	32	77, 69 (83)	
	77	88, 86 (87)	
	170	65, 62, 69, 65, 68, 65 (67)	

Animal commodities

Separate control samples of milk and tissues of muscle, liver, fat and kidney were fortified with fluazinam, AMPA and DAPA at 0.1 mg/kg and stored frozen. Unfortified samples to serve as control and procedural recovery samples were also prepared.

At specified sampling intervals stored samples and freshly fortified samples were analysed for residues of fluazinam, AMPA and DAPA. Final determination was achieved using analytical method IB-2007-JLW-004-00-01. The method used for milk included a hydrolysis step to extract any sulfamate conjugates that may be present. For kidney and liver samples two sets of analysis were undertaken; extraction with acetonitrile: water, and extraction with acetonitrile: water followed by a hydrolysis step with HCl. The results are summarised in Table 89.

Table 89 Storage stability data for fluazinam, AMGT and DAPA in animal matrices

Analyte	Matrix	Sample storage interval (days)	Stored recoveries (%)	Procedural recoveries (%)
Fluazinam	Milk	183	79, 87, 95 (87)	79, 87
	Fat	205	84, 84, 87 (85)	90, 91
	Muscle	1	62, 66, 62 (63)	68, 64
		164	43, 44, 47 (45)	83, 79
	Liver (non-hydrolysis method)	209	28, 24, 29 (27)	76, 82
	Liver (hydrolysis method)	210	18, 20, 19 (21)	68, 76
	Kidney (non-hydrolysis method)	210	19, 23, 21(35)	42, 53
	Kidney (hydrolysis method)	218	34, 36, 34 (35)	86, 85
AMPA	Milk	183	87, 87, 98 (91)	92, 96
	Fat	205	92, 90, 91 (91)	108, 106
	Muscle	2	109, 113, 107 (110)	103, 90
		161	42, 38, 34 (38)	95, 91
	Liver (non-hydrolysis method)	209	42, 43, 48 (44)	45, 55
	Liver (hydrolysis method)	210	31, 43, 38 (37)	47, 48
	Kidney (non-hydrolysis method)	218	31, 43, 38 (37)	60, 68
	Kidney (hydrolysis method)	218	26, 37, 33 (32)	57, 64
DAPA	Milk	183	90, 87, 88 (88)	76, 78
	Fat	205	62, 57, 59 (59)	92, 92
	Muscle	2	102, 98, 90 (97)	104, 93
		161	11, 10, 9 (10)	97, 95
	Liver (non-hydrolysis method)	209	14, 19, 22 (18)	31, 53
	Liver (hydrolysis method)	210	17, 18, 20 (18)	33, 36
	Kidney (non-hydrolysis method)	218	13, 23 ,18 (18)	68, 77
	Kidney (hydrolysis method)	218	4, 6, 7 (6)	20, 45

Values in parentheses = mean recovery of stored samples

Stability of residues in samples extract

The storage stability of sample extracts was addressed by the analysis of procedural recovery samples which were prepared, stored and analysed concurrently with the samples from the residue trials.

USE PATTERNS

Table 90 represents a summary of the GAPs submitted for consideration in this Meeting.

Table 90 List of uses of fluazinam submitted for this meeting

Crop	Country	Indoor/ outdoor	Type	Timing of application	Rate per appl'n (kg ai/ha)	Total appl'n (kg ai/ha)	No. of appl'n (interval)	PHI (days)
Apple	USA	Outdoor	Foliar	Not stated	0.504	5.045	Must not exceed 10 (7–10 days)	28
Wine grape	Hungary	Outdoor	Foliar	BBCH 79	0.750	3.75	5 (7 -14 days)	21
Wine grape	Italy	Outdoor		Not stated	0.5	0.5	1	22
Table grape				At the end of flowering	0.5	0.5	1	Defined by appl'n timing
Wine grape	Chile	Outdoor		Not stated	1.2	3.6	3 (not specified)	22
Blueberries and other bush berries	USA	Outdoor	Foliar	Some fruit ripened	0.730	4.38	Not specified (7–10 days)	30

Crop	Country	Indoor/ outdoor	Type	Timing of application	Rate per appl'n (kg ai/ha)	Total appl'n (kg ai/ha)	No. of appl'n (interval)	PHI (days)
Bulb onions	USA	Outdoor	Foliar	Not stated	0.584	3.51	Must not exceed 6 (7–10 days)	7
Broccoli	USA	Outdoor	Soil drench	At or after transplanting	1.52	1.52	1	50
Cabbage	USA	Outdoor	Soil drench Plus Foliar	At or after transplanting	Soil drench: 0.025 kg/hL (100 mL of soln per plant i.e. 0.025 kg ai/1000 plants) Foliar: 0.561	Soil drench: 0.025 kg/hL (100 mL soln per plant i.e. 0.025 kg ai/1000 plants Foliar: 3.36	Not specified (7 days)	7
Mustard greens	USA	Outdoor	Soil drench	At or after transplanting	0.025 kg/hL (100 mL soln per plant i.e. 0.025 kg ai/1000 plants)	0.025 kg/hL (100 mL of soln per plant i.e. 0.025 kg ai/1000 plants)	Not specified (7 days)	20
Lettuce	USA	Outdoor	Foliar	-	0.874	0.874 [b]	-	30
Melon	USA	Outdoor	Foliar	Defined by PHI	0.876	5.26	Not specified (7–10 days)	30
Cucumber, summer squash	USA	Outdoor	Soil drench followed by Foliar	For soil drench BBCH 00-10	0.876	4.38	4 [a] (7–10 days for foliar appl'n)	7
Bell pepper and non-bell pepper	USA	Outdoor	Soil drench followed by Foliar	For soil drench 7 days after transplant	0.876	5.26	Not specified (7–14 days for foliar appl'n)	30
Snap beans and other Edible podded beans (Bean with pod)	USA	Outdoor	Foliar		0.497	1.02	Not specified (7–10 days) [c]	14
Succulent beans, including Lima beans (Bean without a pod)	USA	Outdoor	Foliar		0.497	1.02	Not specified (7–10 days) [c]	30
Soya bean [d]	USA	Outdoor		Early pod formation (R3)	0.583	1.17	Not specified (10–14 days)	Defined by appl'n timing
Dry beans	USA	Outdoor	Foliar		0.497	1.02	Not specified (7–10 days) [c]	30
Carrot	USA	Outdoor	Foliar		0.583	2.33	4 (7–14 days)	7
Potato	USA	Outdoor	Foliar		0.293	2.04	Not specified (7– 10 days)	14
Ginseng	USA	Outdoor	Foliar		0.876	3.51	Not specified (7– 14 days)	30
Peanuts [e]	USA	Outdoor	Foliar		0.874	2.34	Not specified	30

Crop	Country	Indoor/ outdoor	Type	Timing of application	Rate per appl'n (kg ai/ha)	Total appl'n (kg ai/ha)	No. of appl'n (interval)	PHI (days)
							(21– 28 days)	
Tea	Japan	Outdoor	Foliar	-	1	1	-	14

[a] Only 4 applications at 0.876 kg ai/ha are permitted. The first application at 0.876 kg ai/ha may be made as soil drench at transplantation or when the plants have the first true leaves. The critical GAP is therefore four foliar applications.

[b] The total application rate per crop cycle is 0.874 kg ai/ha with no more than 4 crop cycles permitted per year and a total application rate of 3.51 kg ai/ha/year

[c] The total application rate per crop cycle is 1.02 kg ai/ha with no more than 3 crop cycles permitted per year and a total application rate of 3.07 kg ai/ha/year

[d] For soya bean livestock are not permitted to graze treated areas and treated hay must not be fed to livestock

[e] For peanut livestock are not permitted to graze in treated area and treated hay and threshings must not be fed to livestock

RESIDUES RESULTING FROM SUPERVISED TRIALS

Apple

Twenty three residue trials were conducted in Canada and the USA in 1992-1994 and 2006.

Seven-twelve foliar applications were made using an SC formulation at application rates in the range of 0.482–1.009 kg ai/ha.

Samples of apple were collected 28–90 days after the last treatment.

Samples were immediately frozen and maintained in frozen storage for periods of up to 287 days for fluazinam and 1187 days for AMGT prior to extraction and analysis.

Residues of fluazinam and AMGT in apple were determined using the two analytical methods outlined above. Procedural recovery samples were analysed with the residue trial samples. For fluazinam the recoveries were at fortification levels of 0.01-1 mg/kg with recoveries in the range of 71–126%. For AMGT the recoveries were at fortification levels of 0.01-1 mg/kg with recoveries in the range of 55–130%.

Table 91 Residues in Apple from supervised trials in Canada and the USA involving 7-12 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP USA	MID: 0.504 MTD: 5.045 †	7-10	-	28	-	-		-
Kenly, NC, USA 1992 Apple/Starspur	0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 [5.045]	-- 7 10 14 14 15 14 12 15 13	Fruit less than 8.9 cm diameter	30	Apple	0.05, 0.03 (0.04)	<0.01	5347-92-0245-CR-001 McFall, D.D. 1996a Kenly, NC

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Conklin, MI, USA 1992 Apple/ Miller	0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 [6.053]	-- 7 7 7 10 10 10 13 15 13 15 14	Apples are 5.7-6.4 cm diameter	30	Apple	0.05, 0.06 (0.06)	0.01, 0.02 (0.02)	5347-92-0245-CR-001 McFall, D.D. 1996a Conklin, M
Ephrata, WA, USA 1993 Apple/ Red delicious	0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 [3.531]	-- 8 13 10 10 10 10 10	Fruit 3.8 cm diameter	90	Apple	<0.01	0.02	5878-93-0345-CR-001 Fitzgerald, T.J. and McFall, D.D. 1995 and 5878-93-0345-CR-002 Fitzgerald, T.J. and McFall, D.D. 1996 Ephrata, WA
Watsonville, CA, USA 1993 Apple/ Fuji	0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 [3.531]	-- 7 9 11 9 11 10	60-70% cessation of terminal growth	90	Apple	<0.01	<0.01	5878-93-0345-CR-001 Fitzgerald, T.J. and McFall, D.D. 1995 and 5878-93-0345-CR-002 Fitzgerald, T.J. and McFall, D.D. 1996 Watsonville, CA
Upper black Eddy, PA, USA 1993 Apple/ Jonamac	0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 [5.045]	-- 7 7 10 12 13 14 14 14 14 14	Fruit sizing slowly due to dry weather	28	Apple	0.02, 0.04 (0.03)	0.01	5878-93-0345-CR-001 Fitzgerald, T.J. and McFall, D.D. 1995 and 5878-93-0345-CR-002 Fitzgerald, T.J. and McFall, D.D.

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
	1.009 1.009 1.009 1.009 1.009 1.009 1.009 1.009 1.009 1.009 [10.089]	-- 7 7 10 12 13 14 14 14 14	Fruit sizing slowly due to dry weather	28	Apple	0.1, 0.08 (0.09)	0.01	1996 Upper Black Eddy, PA Replicate trials–HR taken from 0.03 and 0.08
	0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 [5.045]	-- 7 7 10 12 13 14 14 14 14	Fruit sizing slowly due to dry weather	28	Apple	0.08, 0.08 (0.08)	<0.01	
Williamson, NY, USA 1993 Apple/ Twenty ounce	0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 0.504 [5.045]	-- 10 7 10 12 10 10 14 14 14	Fruit 6.3-6.6 cm diameter	29	Apple	0.02, 0.03 (0.03)	<0.01	5878-93-0345-CR-001 Fitzgerald, T.J. and McFall, D.D. 1995 and 5878-93-0345-CR-002 Fitzgerald, T.J. and McFall, D.D. 1996 Williamson, NY
Ephrata, WA, USA 1994 Apple/ Golden delicious	0.493 0.482 0.493 0.482 0.482 [2.433]	-- 4 15 10 10	Fruit diameter at 1.3 cm	90	Apple	<0.01	<0.01	6103-95-0025-CR-001 Fitzgerald, T.J. and McFall. D.D. 1996b Ephrata, WA
Winchester, VA, USA 1994 Apple/ Golden delicious	0.493 0.493 0.493 0.493 0.493 0.493 0.493 0.493 0.493 0.493 [4.932]	-- 8 8 12 15 15 14 14 15 15	not noted	31	Apple	0.03, 0.04 (0.04)	0.01	6103-95-0025-CR-001 Fitzgerald, T.J. and McFall. D.D. 1996b Winchester, VA

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Conklin, MI, USA 1994 Apple/ Paula red	0.493 0.504 0.493 0.482 0.482 0.504 0.504 0.493 0.493 [4.45]	-- 7 7 7 10 10 11 9 10	Apples are approx. 4.4 cm diameter	30	Apple	<0.01	<0.01	6103-95-0025-CR-001 Fitzgerald, T.J. and McFall. D.D. 1996b Conklin, MI
Hereford, PA, USA 2006 Apple/ Starkrims on red delicious	0.522 0.517 0.516 0.522 0.521 0.528 0.511 0.525 0.524 0.524 [5.209]	-- 7 7 6 8 7 5 8 7 7	Final Fruit Swell	28	Apple	0.16, 0.16 (0.16)	<0.01	IB-2006-JLW-002-00-01 Wiedmann, J.L. 2008 IB-2006-JLW-002-01
Shelby, MI, USA 2006Apple/ Yellow delicious	0.519 0.518 0.518 0.521 0.503 0.503 0.503 0.504 0.504 0.504 [5.099]	-- 6 7 7 7 7 7 7 7 7	BBCH 82	28	Apple	1st sample-0.04 [0.26], (0.15) 2nd sample-0.10 [0.14], (0.12) Mean = 0.14 Highest analytical result = 0.15	<0.01, 0.01, (0.01)	IB-2006-JLW-002-00-01 Wiedmann, J.L. 2008 IB-2006-JLW-002-05
Eckert, CO, USA 2006 Apple/ Red delicious	0.502 0.498 0.501 0.501 0.503 0.500 0.500 0.501 0.503 0.500 [5.01]	-- 7 7 7 7 7 7 7 6 8	BBCH 85 81 mm fruit	28	Apple	1st sample = 1.75[1.61], (1.68) 2nd sample = 1.74 [1.04], (1.39) Mean = 1.54 Highest analytical result = 1.68	<0.01	IB-2006-JLW-002-00-01 Wiedmann, J.L. 2008 IB-2006-JLW-002-06
Hickson, CA, USA 2006 Apple/ Elliot	0.501 0.506 0.501 0.504 0.502 0.503 0.515 0.518 0.518 0.513 [5.081]	-- 7 7 8 6 7 7 7 7 7	BBCH 77	28	Apple	0.05, 0.02 (0.04)	0.01, <0.01 (0.01)	IB-2006-JLW-002-00-01 Wiedmann, J.L. 2008 IB-2006-JLW-002-07

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Ephrata, WA 2006 Apple/ Braeburn	0.500 0.499 0.499 0.497 0.499 0.494 0.498 0.499 0.500 0.495 [4.979]	-- 7 7 7 7 7 7 7 7 7	BBCH 79	28	Apple	0.17, 0.09 (0.13)	<0.01	IB-2006-JLW-002-00-01 Wiedmann, J.L. 2008 IB-2006-JLW-002-08
Toppenish, WA 2006 Apple/ Red delicious	0.502 0.534 0.538 0.522 0.526 0.537 0.518 0.539 0.532 0.528 [5.277]	-- 6 8 6 7 7 8 6 7 6	Immature fruit	0 7 14 21 28	Apple	2.39[2.88], 1.45 [2.13] (2.21) 1.87, 1.80 (1.84) 1.07 [1.46], 1.95 [2.17] (1.66) 1.99, 1.69 (1.84) 1st sample-1.10 [1.51], (1.31) 2nd sample–1.38 [1.59], (1.49) Mean = 1.40 Highest analytical result = 1.49	<0.01 <0.01 <0.01 <0.01 <0.01	IB-2006-JLW-002-00-01 Wiedmann, J.L. 2008 IB-2006-JLW-002-09
Payette, ID 2006 Apple/ Law rome	0.522 0.518 0.521 0.515 0.518 0.513 0.503 0.515 0.511 0.518 [5.154]	-- 6 7 6 8 7 7 7 6 7	40% colour	28	Apple	0.13, 0.15 (0.14)	<0.01	IB-2006-JLW-002-00-01 Wiedmann, J.L. 2008 IB-2006-JLW-002-10
Hood River, OR 2006 Apple/ Jonigold	0.528 0.519 0.522 0.534 0.528 0.520 0.528 0.516 0.517 0.535 [5.246]	-- 7 6 8 7 7 7 7 7 7	Fruit coloring	28	Apple	1st sample-0.04[0.06], (0.05) 2nd sample–0.30 [0.30], (0.3) Mean = 0.18 Highest analytical result = 0.3	<0.01	IB-2006-JLW-002-00-01 Wiedmann, J.L. 2008 IB-2006-JLW-002-11

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Ephrata, WA 2006 Apple/ Red delicious	0.498 0.498 0.494 0.500 0.499 0.499 0.500 0.495 0.499 0.501 [4.983]	-- 7 7 7 7 7 7 7 7 7	BBCH 81	28	Apple	0.12, 0.17 (0.15)	<0.01	IB-2006-JLW-002-00-01 Wiedmann, J.L. 2008 IB-2006-JLW-002-12
Branchton, ON, Canada 1994 Apple/ Courtland	0.504 0.516 0.516 0.504 0.504 0.504 0.516 0.493 0.538 0.516 [5.112]	-- 8 8 9 11 10 11 21 23 14	not noted	29	Apple	<0.01	<0.01	6103-95-0025-CR-001 Fitzgerald, T.J. and McFall. D.D. 1996b Branchton, ON
Sommerset, NS, Canada 1994 Apple/ Mcintosh	0.493 0.538 0.527 0.504 0.516 0.493 0.516 0.516 0.527 0.516 [5.145]	-- 9 8 9 10 10 11 15 16 13	not noted	32	Apple	0.02, 0.04 (0.03)	<0.01	6103-95-0025-CR-001 Fitzgerald, T.J. and McFall. D.D. 1996b Sommerset, NS
St. Paul d'Abbotsford, QC, Canada‡ 2006 Apple/Lobo	0.506 0.506 0.510 0.500 0.508 0.512 0.506 0.521 0.526 0.510 [5.103]	-- 6 7 6 7 8 7 6 6 7	6-7 cm fruit	7 14 21 29	Apple	0.44, 0.34 (0.39) <0.01 <0.01, 0.01 (0.01) 0.03, 0.02 (0.03)	0.02, 0.01 (0.02) 0.02, 0.01 (0.02) 0.03, 0.01 (0.02) 0.02, 0.03 (0.03)	IB-2006-JLW-002-00-01 Wiedmann, J.L. 2008 B-2006-JLW-002-02
St. Paul d'Abbotsford, QC, Canada‡ 2006 Apple/ Empire	0.499 0.503 0.507 0.510 0.516 0.492 0.503 0.504 0.510 0.504 [5.049]	-- 8 6 7 6 8 7 8 6 7	6-7 cm fruit	28	Apple	0.09, 0.15 (0.12)	0.02, 0.01 (0.02)	IB-2006-JLW-002-00-01 Wiedmann, J.L. 2008 IB-2006-JLW-002-03

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
St. Paul d'Abbotsford, QC, Canada‡ 2006 Apple/ Paula red	0.488 0.510 0.506 0.515 0.507 0.501 0.500 0.500 0.519 0.799 [5.344]	-- 8 6 7 6 7 8 6 7 6	6-7 cm fruit, 50% red	28		0.15, 0.12 (0.14)	<0.01	IB-2006-JLW-002-00-01 Wiedmann, J.L. 2008 IB-2006-JLW-002-04

†The GAP authorised is restricted to a maximum of 10 applications

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Results in square brackets represents the re-analysis of the same analytical sample. The mean result from these duplicate analyses is given. The overall mean from the two independent samples analysed is also given as this the highest analytical result taking into account the mean of the duplicate analysis.

‡Two trials conducted on same trial site. However, the timings of all applications were >30 days apart. The third trial from Canada was in the same region but a different trial site.

Grapes

Nine residue trials were conducted in Canada and the USA in 1991 and 1994.

One to eight foliar applications were made using an SC formulation at application rates in the range of 0.751–1.121 kg ai/ha.

Samples of grapes were collected 11–21 days after the last treatment.

Samples were immediately frozen and maintained in frozen storage for periods of up to 207 days for fluazinam and AMGT prior to extraction and analysis.

Residues of fluazinam and AMGT in grapes were determined using the analytical methods 1 and 3. Procedural recovery samples were analysed with the residue trial samples. For fluazinam the recoveries were at fortification levels of 0.01-1 mg/kg with recoveries in the range of 77–126%. For AMGT the recoveries were at fortification levels of 0.01-1 mg/kg with recoveries in the range of 55–130%.

Table 92 Residues in Grapes from supervised trials in Canada and the USA involving 1-8 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP Hungary	0.750 ×5 [3.75]	7-14	Defined by PHI	21	-	-	-	-
Madera CA, USA 1991 Grape/Thompson Seedless	1.121 1.121 1.121 [3.363]	-- 14 15	Maturing fruit; Berry size 1.3 cm	11 11	Grapes	0.89, 0.63 (0.76) 2.25, 1.72 (1.99) Mean = *1.38*	n/a	2127-91-0434-CR-001 Fitzgerald, T.J. 1992
Dundee NY, USA 1991	1.121 [1.121]	--	Fully coloured, Fruit 1-2 cm	13	Grapes	0.39, 0.25 (0.32)	n/a	2106-91-0309-CR-001-001

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Grape/Concord	1.121 1.121 [2.242]	-- 32	Fully coloured, Fruit 1-2 cm	13	Grapes	0.42, 0.29 (0.36)	n/a	Kenyon R.G. 1992a
Royal City WA, USA 1991	1.121 [1.121]	--	Mature grapes	14	Grapes	0.45, 0.41 (0.43)	n/a	
Grape/Cabernet Sauvignon	1.121 1.121 [2.242]	-- 26	Mature grapes	14	Grapes	0.37, 0.35 (0.36)	n/a	
Madera CA, USA 1991	1.121 [1.121]	--	Maturing fruit	14	Grapes	0.73, 0.69 (0.71)	n/a	
Grape/Thompson Seedless	1.121 1.121 [2.242]	-- 14	Maturing fruit	14	Grapes	0.96, 0.79 (0.88)	n/a	
Fresno CA, USA 1994 Grape/Thompson Seedless	0.751 0.751 0.751 0.751 [3.004]	-- 45 14 25	-	20	Grapes	0.07 [0.09] Mean = 0.08	0.27 [0.27]	6106-95-0012-CR-001 Jablonski, J.E.1995a
George WA, USA 1994 Grape/White Riesling	0.751 0.751 0.751 0.751 [3.004]	-- 32 43 36	Grape health excellent, crop ~3 weeks from maturity	19 19	Grapes	0.17, 0.15 (0.16) 0.07, 0.08 (0.08) Mean = <u>0.12</u>	0.11, 0.09 (0.10) 0.08, 0.08 (0.08) 0.09	
Suisun CA, USA 1994 Grape/Carignane	0.751 0.751 0.751 0.751 [3.004]	-- 27 43 36	-	21	Grapes	0.11 [0.11] Mean = <u>0.11</u>	0.15 [0.13]	
Phelps NY, USA 1994 Grape/Catawba	0.751 0.751 0.751 0.751 0.751 0.751 0.751 0.751 [6.008]	-- 14 14 14 14 14 14 14	Veraison	20	Grapes	0.70, 0.82 (0.76)	0.10, 0.16 (0.13)	6106-95-0012-CR-001 Jablonski, J.E.1995a
Vineland ON, USA 1994 Grape/Riesling	0.751 0.751 0.751 0.751 0.751 0.751 0.751 0.751 [6.008]	-- 14 14 14 14 14 14 14	Ripening	17	Grapes	0.03 [0.03]	0.05 [0.05]	

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Results in square brackets represent the re-analysis of the same analytical sample

n/a = not analysed

Two residue trials were conducted in Chile in 1996.

Four foliar applications were made using an SC formulation at application rates in the range of 0.730–0.780 kg ai/ha.

Samples of grapes were collected 20–21 days after the last treatment.

Samples were immediately frozen and maintained in frozen storage for periods of up to 30 days for fluazinam and up to 70 days for AMGT prior to extraction and analysis.

Residues of fluazinam and AMGT in grapes were determined using the analytical method 3 outlined above. Procedural recovery samples were analysed with the residue trial samples. For fluazinam the recoveries were at fortification levels of 0.01-1 mg/kg with recoveries in the range of 80–120%. For AMGT the recoveries were at fortification levels of 0.01-1 mg/kg with recoveries in the range of 68–126%.

Table 93 Residues in Grapes from supervised trials in Chile involving 4 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP Hungary	0.750 [3.75]	7-14	Defined by PHI	21	-	-	-	-
Paine, Region Metropolitane, Chile, 1996 Grape/Cabernet Sauvignon	0.780 0.755 0.785 0.752 [3.072]	-- 34 24 35	Colour change	21	Grapes	0.27, 0.36 (0.32)	0.13, 0.13 (0.13)	EA950132 Grolleau, G. and Kenyon, R.G. 1996
San Juan de pirique, Region Metropolitane, Chile, 1996 Grape/Totonel	0.765 0.765 0.750 0.730 [3.01]	-- 34 44 34	Colour change	20	Grapes	0.45, 0.64 (0.55)	0.13, 0.18 (0.16)	EA950132 Grolleau, G. and Kenyon, R.G. 1996

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Results in square brackets represent the re-analysis of the same analytical sample

Sixty residue trials were conducted in Europe between 1990 and 2010. In several trials a number of replicate trials were also conducted.

One to eight foliar applications were made using either WP or SC formulations at application rates in the range of 0.250–0.870 kg ai/ha.

Samples of grapes were collected 0–111 days after the last treatment.

Samples were immediately frozen and maintained in frozen storage for periods of up to 363 days for fluazinam and 935 days for AMGT prior to extraction and analysis.

Residues of fluazinam and AMGT in grapes were determined using the analytical methods 3, 4, 5, 6 and. Procedural recovery samples were analysed with the residue trial samples. For fluazinam the recoveries were at fortification levels of 0.01-10 mg/kg with recoveries in the range of 72–130%. For AMGT the recoveries were at fortification levels of 0.01–10 mg/kg with recoveries in the range of 60–126%.

Table 94 Residues in Grapes from supervised trials in Europe involving 1-8 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP Hungary	0.750 [3.75]	7-14	Defined by PHI	21	-	-	-	-
Germany, EU (North) 1995 Grape/ Müller Thurgau	0.770 0.775 0.765 0.785 [3.095]	-- 23 22 17	BBCH 83	20	Grapes	2.22 [2.31] Mean = 2.27	0.18 [0.21] Mean = 0.20	EA950132 Grolleau, G. and Kenyon, R.G. 1996
France, EU (North) 1995 Grape/Gros Plant	0.802 0.702 0.773 0.762 [3.038]	-- 14 31 14	Colour change	7	Grapes	0.25, 0.23 (0.24)	0.10, 0.08 (0.09)	
				14	Grapes	0.17, 0.14 (0.16)	0.12, 0.05 (0.09)	
				21	Grapes	0.12, 0.15 (0.14)	0.07, 0.08 (0.08)	
				28	Grapes	0.12, 0.13 (0.13)	0.10, 0.09 (0.10)	
				35	Grapes	0.14, 0.14 (0.14)	0.07, 0.14 (0.11)	
Spain, EU (South) 1995 Grape/Garnacha Comun	0.721 0.773 0.750 0.770 [3.014]	-- 19 24 16	Colour change	22	Grapes	0.08, 0.17 (0.13)	0.06, 0.11 (0.09)	
France, EU (South) 1995 Grape/Syrah	0.778 0.752 0.744 0.760 [3.036]	-- 19 27 16	Colour change	22	Grapes	0.22 [0.21] Mean = 0.22	0.20 [0.22] Mean = 0.21	EA950132 Grolleau, G. and Kenyon, R.G. 1996
France, EU (South) 1995 Grape/Cabernet Sauvignon	0.722 0.771 0.764 0.784 [3.036]	-- 27 25 19	Colour change	21	Grapes	1.39, 1.51 (1.45)	0.16, 0.14 (0.15)	
France, EU (South) 1995 Grape/Ugni Blanc	0.742 0.728 0.777 0.737 [2.983]	-- 31 24 28	Colour change	22	Grapes	1.17, 0.77 (0.97)	0.20, 0.17 (0.19)	
Italy, EU (South) 1995 Grape/Barbera	0.753 0.728 0.727 0.747 [2.955]	-- 22 21 27	BBCH 85	21	Grapes	0.68, 0.74 (0.71)	0.14, 0.16 (0.15)	
Italy, EU (South) 1995 Grape/Riesling	0.771 0.715 0.751 0.759 [2.998]	-- 28 16 15	BBCH 85	21	Grapes	0.80, 0.42 (0.61)	0.18, 0.15 (0.17)	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Italy, EU (South) 1995 Grape/Rondinella	0.740 0.752 0.756 0.747 [2.995]	-- 20 34 22	BBCH 85	22	Grapes	2.24, 2.14 (2.19)	0.28, 0.21 (0.25)	
Italy, EU (South) 1995 Grape/Trebbiano	0.753 0.739 0.746 0.718 [2.955]	-- 9 37 22	BBCH 85	31	Grapes	1.55, 1.39 (1.47)	0.35, 0.32 (0.34)	
France, EU (North)	0.5	--	End of flowering	83	Grapes	0.02	n/a	M53785
1990	0.5	--	Bunch closure	59	Grapes	0.05	n/a	Ryan, J. and Sapiets, A. 1991a
Grape/Pinot Noir	0.5	--	Start of colour change	42	Grapes	0.08	n/a	
	0.5 0.5 [1.0]	-- 24	Bunch closure	59	Grapes	0.09	n/a	
	0.5 0.5 [1.0]	-- 41	Start of colour change	42	Grapes	0.06	n/a	
	0.5 0.5 [1.0]	-- 17	Start of colour change	42	Grapes	0.10	n/a	
	0.5 0.5 0.5	-- 24 17	Start of colour change	42	Grapes	0.20	n/a	
France, EU (North)	0.5	--	Start of fruiting	91	Grapes	0.04 [0.04] Mean = 0.04	n/a	M53785
1990	0.5	--	Bunch closure	69	Grapes	0.05 [0.04] Mean = 0.05	n/a	Ryan, J. and Sapiets, A. 1991a
Grape/Carignan	0.5	--	Start of colour change	34	Grapes	0.03 [0.05] Mean = 0.04	n/a	
	0.5 0.5 [1.0]	-- 22	Bunch closure	69	Grapes	0.03 [0.03] Mean = 0.03	n/a	
	0.5 0.5 [1.0]	-- 35	Start of colour change	34	Grapes	0.04 [0.03] Mean = 0.04	n/a	
	0.5 0.5 [1.0]	-- 57	Start of colour change	34	Grapes	0.02 [0.03] Mean = 0.03	n/a	
	0.5 0.5 0.5 [1.5]	-- 22 57	Start of colour change	34	Grapes	0.02 [0.03] Mean = 0.03	n/a	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
France, EU (North) 1990 Grape/Gamay	0.5	--	23-25 (Eichhorn-Lorenz, late flowering, 80% flower hoods fallen))	96	Grapes	<0.01	n/a	
	0.5	--	33 (Eichhorn-Lorenz, bunch closure)	68	Grapes	0.01	n/a	
	0.5	--	35 (Eichhorn-Lorenz, veraison)	53	Grapes	0.03	n/a	
	0.5 0.5 [1.0]	-- 28	33 (Eichhorn-Lorenz, bunch closure)	68	Grapes	0.01	n/a	
	0.5 0.5 [1.0]	-- 43	35 (Eichhorn-Lorenz, veraison)	53	Grapes	0.03	n/a	
	0.5 0.5 [1.0]	-- 15	35 (Eichhorn-Lorenz, veraison)	53	Grapes	0.05	n/a	
	0.5 0.5 0.5 [1.5]	-- 28 15	35 (Eichhorn-Lorenz, veraison)	53	Grapes	0.04	n/a	
France, EU (North) 1990 Grape/Chenin	0.5 0.5 [1.0]	-- 31	End of flowering	82	Grapes	0.03	n/a	M5377B Ryan, J. and Sapiets, A. 1991b
	0.5 0.5 [1.0]	-- 58	First fruits colouring	55	Grapes	0.09	n/a	
	0.5 0.5 0.5 [1.5]	-- 31 27	First fruits colouring	55	Grapes	0.06	n/a	
	0.75 0.75 0.75 0.75 [3.0]	-- 31 27 33	3 weeks before harvest	22	Grapes	0.46	n/a	
	0.5 0.5 0.5 0.5 [2.0]	-- 31 27 33	3 weeks before harvest	22	Grapes	0.32	n/a	
France, EU (North) 1990	0.5 0.5 [1.0]	-- 30	Bunch closing	68	Grapes	0.02	n/a	M5377B Ryan, J. and Sapiets, A. 1991b

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Grape/Sauvignon	0.5 0.5 [1.0]	-- 52	First berries colouring	43	Grapes	0.01	n/a	
	0.5 0.5 0.5 [1.5]	-- 30 22	First berries colouring	43	Grapes	0.07	n/a	
	0.75 0.75 0.75 0.75 [3.0]	-- 30 22 18	3 weeks before harvest	28	Grapes	0.14	n/a	
	0.5 0.5 0.5 0.5 [2.0]	-- 30 22 18	3 weeks before harvest	28	Grapes	0.07	n/a	
France, EU (North) 1990 Grape/Pinot noir	0.5 0.5 [1.0]	-- 26	Bunch closing	67	Grapes	0.04	n/a	M5377B Ryan, J. and Sapiets, A. 1991b
	0.5 0.5 [1.0]	-- 42	First berries colouring	49	Grapes	0.07	n/a	
	0.5 0.5 0.5 [1.5]	-- 26 16	First berries colouring	49	Grapes	0.10	n/a	
	0.75 0.75 0.75 0.75 [3.0]	-- 26 16 26	3 weeks before harvest	25	Grapes	0.23	n/a	
	0.5 0.5 0.5 0.5 [2.0]	-- 26 16 26	3 weeks before harvest	25	Grapes	0.44	n/a	
France, EU (North) 1990 Grape/Pinot noir	0.5 0.5 [1.0]	-- 27	Bunch closing	67	Grapes	0.04	n/a	M5377B Ryan, J. and Sapiets, A. 1991b
	0.5 0.5 [1.0]	-- 45	Many berries colouring	49	Grapes	0.03	n/a	
	0.5 0.5 0.5 [1.5]	-- 27 18	Many berries colouring	49	Grapes	0.18	n/a	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
	0.75 0.75 0.75 0.75 [3.0]	-- 27 18 21	3 weeks before harvest	25	Grapes	0.65	n/a	
	0.5 0.5 0.5 0.5 [2.0]	-- 27 18 24	3 weeks before harvest	25	Grapes	0.34	n/a	
France, EU (North) 1990 Grape/Pinot noir	0.5 0.5 [1.0]	-- 26	Bunch closing	60	Grapes	0.18	n/a	M5377B Ryan, J. and Sapiets, A. 1991b
	0.5 0.5 [1.0]	-- 42	First berries colouring	44	Grapes	0.24	n/a	
	0.5 0.5 0.5 [1.5]	-- 26 16	First berries colouring	44	Grapes	0.18	n/a	
	0.75 0.75 0.75 0.75 [3.0]	-- 26 16 26	3 weeks before harvest	18	Grapes	0.10	n/a	
	0.5 0.5 0.5 0.5 [2.0]	-- 26 16 26	3 weeks before harvest	18	Grapes	0.71 [1.1] Mean = 0.91	n/a	
France, EU (North) 1991 Grape/Pinneu d/Aunis	0.75 [0.75]	--	3-35 (Eichhorn-Lorenz scale, bunches closing and beginning to colour)	0	Grapes	1.5	n/a	RJ1107B Burke, S.R. and Sapiets, A. 1991a
				13	Grapes	0.33	n/a	
				31	Grapes	0.07	n/a	
				45	Grapes	0.05	n/a	
				60	Grapes	0.07	n/a	
France, EU (South) 1991 Grape/Carignan	0.75 [0.75]	--	3-35 (Eichhorn-Lorenz scale, bunches closing and beginning to colour)	0	Grapes	0.44	n/a	RJ1107B Burke, S.R. and Sapiets, A. 1991a
				13	Grapes	0.06 [0.07] Mean = 0.07	n/a	
				29	Grapes	0.01 [0.02] Mean = 0.02	n/a	
				46	Grapes	<0.01	n/a	
				61	Grapes	<0.01	n/a	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
France, EU (North) 1991 Grape/Pinot Noir	0.75 [0.75]	--	Bunches closing	77	Grapes	0.03	n/a	RJ1133B Burke, S.R. and Sapiets, A. 1992b
	0.75 0.75 [0.75]	-- 17	Bunches closing	77	Grapes	0.08	n/a	
	0.50 0.50 0.50 0.50 0.50 0.50 0.50 [3.50]	-- 9 10 16 9 8 11	Bunches formed	66	Grapes	0.14	n/a	
	0.75 0.75 0.75 0.75 0.75 0.75 0.75 [5.25]	-- 9 10 16 9 8 11	Bunches formed	66	Grapes	0.33	n/a	
	0.50 0.50 0.50 0.50 0.50 0.50 0.50 0.50 [4.0]	-- 9 10 16 9 8 11 16	Start of colour change	50	Grapes	0.15	n/a	RJ1133B Burke, S.R. and Sapiets, A. 1992b
	0.75 0.75 0.75 [2.25]	-- 17 27	Start of colour change	50	Grapes	0.29	n/a	
	0.75 0.75 0.75 0.75 [3.0]	-- 17 27 25	3 weeks to harvest	25	Grapes	1.3	n/a	
France, EU (North) 1991 Grape/Gamay	0.754 [0.754]	--	Bunches closing	71	Grapes	0.01	n/a	
	0.754 0.754 [1.508]	-- 9	Bunches closing	71	Grapes	0.01	n/a	
	0.503 0.501 0.502 0.508 0.505 [2.518]	-- 11 14 9 12	34 (Eichhorn-Lorenz stage scale)	59	Grapes	0.03	n/a	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
	0.784 0.750 0.754 0.765 0.747 [3.80]	-- 11 14 9 12	34 (Eichhorn-Lorenz stage scale)	59	Grapes	0.05	n/a	
	0.495 0.513 0.505 0.502 0.497 0.498 0.508 [3.517]	-- 11 14 9 12 11 11	Start of colour change	37	Grapes	0.17	n/a	
	0.750 0.758 0.761 [2.269]	-- 9 34	Start of colour change	37	Grapes	0.13	n/a	
	0.750 0.745 0.758 0.773 [3.025]	-- 9 34 21	3 weeks to harvest	16	Grapes	0.51	n/a	
France, EU (South) 1991 Grape/Gamay	0.750 [0.750]	--	Bunches closing	67	Grapes	0.02	n/a	RJ1133B Burke, S.R. and Sapiets, A. 1992b
	0.750 0.750 [1.5]	-- 17	Bunches closing	67	Grapes	0.04	n/a	
	0.50 0.50 0.50 0.50 0.50 0.50 0.50 [3.5]	-- 14 9 9 10 7 11	34 (Eichhorn-Lorenz stage scale)	56	Grapes	<0.01	n/a	
	0.750 0.750 0.750 0.750 0.750 0.750 0.750 [5.25]	-- 14 9 9 10 7 11	34 (Eichhorn-Lorenz stage scale)	56	Grapes	0.03	n/a	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
	0.50 0.50 0.50 0.50 0.50 0.50 0.50 0.50 [4.0]	-- 14 9 9 10 7 11 11	34 (Eichhorn-Lorenz stage scale)	45	Grapes	0.07	n/a	
	0.750 0.750 0.750 [2.25]	-- 17 22	34 (Eichhorn-Lorenz stage scale)	45	Grapes	0.11	n/a	
	0.750 0.750 0.750 0.750 [3.0]	-- 17 22 27	34 (Eichhorn-Lorenz stage scale)	18	Grapes	0.6	n/a	
France, EU (North) 1991 Grape/Chenin	0.50 0.50 0.50 0.50 0.50 0.50 0.50 0.50 [3.5]	-- 7 12 10 11 15 15	One week before ripening	50	Grapes	0.18	n/a	RJ1147B Burke, S.R. and Sapiets, A. 1992c
	0.75 0.75 0.75 0.75 0.75 0.75 0.75 0.75 [5.25]	-- 7 12 10 11 15 15	One week before ripening	50	Grapes	0.21	n/a	
	0.75 0.75 [1.5]	-- 22	Bunch closing	69	Grapes	0.50	n/a	
	0.75 0.75 0.75 0.75 [2.25]	-- 22 27	Beginning of ripening	42	Grapes	0.20	n/a	
	0.75 0.75 0.75 0.75 [3.0]	-- 22 27 22	50% ripe	20	Grapes	1.7	n/a	
France, EU (South) 1991 Grape/Semillon	0.50 0.50 0.50 0.50 0.50 0.50 [2.5]	-- 11 14 11 10	31 (Eichhorn-Lorenz stage scale), fruits pea size	80	Grapes	0.01	n/a	RJ1147B Burke, S.R. and Sapiets, A. 1992c

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
	0.75 0.75 0.75 0.75 0.75 [3.75]	-- 11 14 11 10	31 (Eichhorn-Lorenz stage scale), fruits pea size	80	Grapes	0.02	n/a	
	0.75 0.75 [1.5]	-- 24	31 (Eichhorn-Lorenz stage scale), fruits pea size	76	Grapes	0.06	n/a	
	0.75 0.75 0.75 [2.25]	-- 24 25	35 (Eichhorn-Lorenz stage scale)	51	Grapes	0.07	n/a	
	0.75 0.75 0.75 0.75 [3.0]	-- 24 25 24	21 days before harvest	27	Grapes	0.08	n/a	
France, EU (South) 1991 Grape/Grenache	0.5 0.5 0.5 0.5 0.5 0.5 [3.0]	-- 10 9 13 9 11	33 (Eichhorn-Lorenz stage scale), bunch closing	63	Grapes	0.09	n/a	RJ1147B Burke, S.R. and Sapiets, A. 1992c
	0.75 0.75 0.75 0.75 0.75 0.75 [4.5]	-- 10 9 13 9 11	33 (Eichhorn-Lorenz stage scale), bunch closing	63	Grapes	0.1	n/a	
	0.75 0.75 [1.5]	-- 22	31 (Eichhorn-Lorenz stage scale), fruits pea size	74	Grapes	0.17	n/a	
	0.75 0.75 0.75 0.75 [2.25]	-- 22 33	35 (Eichhorn-Lorenz stage scale), fruit colouring	41	Grapes	0.33	n/a	
	0.75 0.75 0.75 0.75 0.75 [3.0]	-- 22 33 17	37 (Eichhorn-Lorenz scale), 3 weeks before harvest	21	Grapes	0.64	n/a	
France, EU (North) 1992 Grape/Pinot Noir	0.750 [0.750]	--	25 (Eichhorn-Lorenz scale, late flowering)	91	Grapes	<0.01	0.03	6936-96-0228-CR-001 Kenyon, R.G. 1996 and 6245-95-0001-CR-001
	0.870 [0.870]	--	33 (Eichhorn-Lorenz scale, bunch closure)	70	Grapes	<0.01	0.04	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
	0.850 0.790 [1.64]	-- 21	33 (Eichhorn-Lorenz scale, bunch closure)	70	Grapes	0.04	0.11	Jablonski, J.E. 1995b
	0.810 0.820 0.860 [2.49]	-- 21 22	35 (Eichhorn-Lorenz scale, veraison)	48	Grapes	0.09	0.18	
	0.830 0.810 0.810 0.840 [3.29]	-- 21 22 24	36 (Eichhorn-Lorenz scale, berries coloured)	24	Grapes	0.28	0.09	
France, EU (North) 1992 Grape/Pinot Noir	0.680 [0.68]	--	25 (Eichhorn-Lorenz scale, late flowering)	88	Grapes	<0.01	0.08	6936-96-0228-CR-001 Kenyon, R.G. 1996 and 6245-95-0001-CR-001 Jablonski, J.E. 1995b
	0.79 [0.79]	--	33 (Eichhorn-Lorenz scale, bunch closure)	67	Grapes	0.05	0.28	
	0.710 0.770 [1.48]	-- 21	33 (Eichhorn-Lorenz scale, bunch closure)	67	Grapes	0.03	0.30	
	0.810 0.800 0.840 [2.45]	-- 21 22	35 (Eichhorn-Lorenz scale, veraison)	45	Grapes	0.22	0.20	
	0.820 0.750 0.780 0.780 [3.1]	-- 21 22 24	36 (Eichhorn-Lorenz scale, berries coloured)	21	Grapes	0.37	0.19	
France, EU (North) 1992 Grape/Chenin	0.840 [0.84]	--	25 (Eichhorn-Lorenz scale, late flowering)	99	Grapes	<0.01	0.04	6936-96-0228-CR-001 Kenyon, R.G. 1996 and 6245-95-0001-CR-001 Jablonski, J.E. 1995b
	0.870 [0.87]	--	33 (Eichhorn-Lorenz scale, bunch closure)	84	Grapes	<0.01	0.04	
	0.840 0.870 [1.71]	-- 15	33 (Eichhorn-Lorenz scale, bunch closure)	84	Grapes	<0.01	0.07	
	0.840 0.830 0.740 [2.38]	-- 15 36	35 (Eichhorn-Lorenz scale, veraison)	48	Grapes	0.23	0.09	
	0.720 0.830 0.770 0.790 [3.11]	-- 15 36 27	36 (Eichhorn-Lorenz scale, berries coloured)	21	Grapes	0.70	0.12	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
France, EU (South) 1992 Grape/Gamay	0.760 [0.76]	--	25 (Eichhorn-Lorenz scale, late flowering)	94 101	Grapes Grapes	0.02 0.01	0.05 0.05	6936-96-0228-CR-001 Kenyon, R.G. 1996 and 6245-95-0001-CR-001 Jablonski, J.E. 1995b
	0.760 [0.76]	--	33 (Eichhorn-Lorenz scale, bunch closure)	67 74	Grapes Grapes	0.04 0.03	0.22 0.16	
	0.760 0.760 [1.52]	-- 27	33 (Eichhorn-Lorenz scale, bunch closure)	67 74	Grapes Grapes	0.03 0.12	0.17 0.18	
	0.760 0.760 0.760 [2.28]	-- 27 22	35 (Eichhorn-Lorenz scale, veraison)	45 52	Grapes Grapes	0.24 0.19	0.25 0.24	
	0.760 0.760 0.760 0.760 [3.04]	-- 27 22 25	36 (Eichhorn-Lorenz scale, berries coloured)	20 27	Grapes Grapes	0.41 0.50	0.24 0.29	
Switzerland, EU (North) 1995 Grape/Riesling x Sylvaner	0.250 [0.25]	--	-	72	Grapes	0.35	n/a	343631 Schanné C. 1994
	0.250 0.250 [0.500]	-- 31	-	0 70	Grapes Grapes	6.62 0.12	n/a n/a	
France, EU (North) 1995 Grape/Pinot Blanc	0.748 0.757 0.756 0.790 [3.05]	-- 21 22 14	-	22 36	Grapes Grapes	2.034 1.213	0.237 0.22	604372 Schulz, M. and Ullrich-Mietzel, A. 1996
France, EU (North) 1995 Grape/Pinot Noir	0.747 0.750 0.747 0.750 [2.994]	-- 28 28 19	-	19	Grapes	1.881 [2.061] Mean = 1.971	0.058	604372 Schulz, M. and Ullrich-Mietzel, A. 1996
France, EU (South) 1995 Grape/Gamay	0.727 0.748 0.750 0.747 [2.971]	-- 30 22 14	-	21	Grapes	0.535	0.167	
France, EU (South) 1995 Grape/Sauvignon	0.764 0.745 0.752 0.754 [3.015]	-- 37 20 20	-	22	Grapes	0.778	0.068	
France, EU (North) 1996	0.750 [0.75]	--	BBCH 69 (end of flowering)	93	Grapes	0.01, 0.01 (0.01)	0.06, 0.05 (0.06)	7074-96-0287-CR-001 Kenyon, R.G.

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Grape/Pinot Noir	0.75 0.75 [1.5]	-- 21	BBCH 77 (bunch closure)	72	Grapes	0.02, 0.02 (0.02)	0.07, 0.08 (0.08)	1997a and 7074-97-0059-CR-001 Kenyon, R.G. 1997b
	0.75 0.75 0.75 [2.25]	-- 21 35	BBCH 81 (beginning veraison)	37	Grapes	0.21, 0.22 (0.22)	0.11, 0.10 (0.11)	
	0.75 0.75 0.75 0.75 [3.0]	-- 21 35 14	3 weeks before harvest	23	Grapes	1.13, 1.07 (1.10)	0.10, 0.11 (0.11)	
France, EU (North) 1996 Grape/Pinot Noir	0.750 [0.75]	--	BBCH 69 (end of flowering)	97	Grapes	<0.01, <0.01 (<0.01)	0.04, 0.04 (0.04)	7074-96-0287-CR-001 Kenyon, R.G. 1997a and 7074-97-0059-CR-001 Kenyon, R.G. 1997b
	0.75 0.75 [1.5]	-- 23	BBCH 77 (beg. bunch closure)	74	Grapes	0.01, 0.01 (0.01)	0.11, 0.11 (0.11)	
	0.75 0.75 0.75 [2.25]	-- 23 28	BBCH 83 (veraison)	46	Grapes	0.06, 0.05 (0.06)	0.14, 0.13 (0.14)	
	0.75 0.75 0.75 0.75 [3.0]	-- 23 28 21	BBCH 85 (veraison)	25	Grapes	0.44, 0.37 (0.41)	0.13, 0.13 (0.13)	
France, EU (South) 1996 Grape/Carignan	0.750 [0.75]	--	BBCH 69 (end of flowering)	92	Grapes	<0.01, <0.01 (<0.01)	0.03, 0.06 (0.05)	7074-96-0287-CR-001 Kenyon, R.G. 1997a and 7074-97-0059-CR-001 Kenyon, R.G. 1997b
	0.75 0.75 [1.5]	-- 12	BBCH 77 (beg. bunch closure)	80	Grapes	0.09, 0.10 (0.10)	0.11, 0.15 (0.13)	
	0.75 0.75 0.75 [2.25]	-- 12 44	BBCH 85 (veraison)	36	Grapes	0.32, 0.36 (0.34)	0.13, [0.30] 0.29, [0.15] (0.22)	
	0.75 0.75 0.75 0.75 [3.0]	-- 12 44 11	BBCH 86 (veraison)	25	Grapes	2.28, 2.53 (2.41)	0.28, 0.22 (0.25)	
France, EU (South) 1996 Grape/Merlot	0.750 [0.75]	--	BBCH 63 (Flowering)	111	Grapes	<0.01, <0.01 (<0.01)	0.02, 0.02 (0.02)	7074-96-0287-CR-001 Kenyon, R.G. 1997a and 7074-97-0059-CR-001
	0.75 0.75 [1.5]	-- 35	BBCH 79 (D. of fruits)	76	Grapes	0.06, 0.07(0.07)	0.06, 0.13 (0.10)	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
	0.75 0.75 0.75 [2.25]	-- 35 33	BBCH 85 (Ripening of berries)	73	Grapes	0.38, 0.42 (0.40)	0.25, 0.26 (0.26)	Kenyon, R.G. 1997b
	0.75 0.75 0.75 0.75 [3.0]	-- 35 33 22	BBCH 88 (Ripening of berries)	21	Grapes	1.21, 1.21 (1.21)	0.17, 0.17 (0.17)	
France, EU (North) 2010 Grape/Gamay	0.761 0.764 0.764 [2.289]	-- 14 16	BBCH 85	21	Grapes	0.02	<0.01	S10-02337 Gemrot, F. 2011c
France, EU (North) 2010 Grape/Chenin	0.778 0.787 0.752 [2.317]	-- 21 13	BBCH 85	21	Grapes	<0.01	<0.01	
France, EU (South) 2010 Grape/Carignan	0.742 0.714 0.717 [2.173]	-- 25 16	BBCH 83-85	19	Grapes	0.03	<0.01	
France, EU (South) 2010 Grape/Chardonnay	0.696 0.816 0.720 [2.231]	-- 22 22	BBCH 85	21	Grapes	<0.01	<0.01	
France, EU (North) 2010 Grape/Cabernet	0.759 0.735 0.738 [2.261]	-- 27 16	-	0	Grapes	0.40	0.03	S10-02338 Gemrot, F. 2011d
				7	Grapes	0.21	0.03	
				14	Grapes	0.20	0.03	
				21	Grapes	0.25	0.04	
				28	Grapes	0.11	0.03	
France, EU (North) 2010 Grape/Carigan	0.751 0.758 0.740 [2.249]	-- 19 9	-	0	Grapes	1.31	0.11	
				7	Grapes	1.38	0.08	
				14	Grapes	0.44	0.13	
				21	Grapes	0.19	0.07	
				28	Grapes	0.16	0.11	
France, EU (North) 2010 Grape/Chenin	0.741 0.734 0.770 [2.245]	-- 22 11	-	0	Grapes	0.23	0.01	
				7	Grapes	0.24	0.01	
				14	Grapes	0.19	0.02	
				21	Grapes	0.32	0.05	
				28	Grapes	0.12	0.02	
France, EU (North) 2010 Grape/Chardonnay	0.763 0.766 0.786 [2.315]	-- 17 7	-	0	Grapes	1.22	0.06	
				7	Grapes	1.59	0.17	
				14	Grapes	0.56	0.11	
				21	Grapes	0.51	0.15	
				28	Grapes	0.36	0.11	
France, EU (South) 2010 Grape/Cabernet Sauvignon	0.721 0.766 0.732 [2.222]	-- 27 14	-	0	Grapes	0.89	0.07	S10-02338 Gemrot, F. 2011d
				7	Grapes	0.58	0.19	
				14	Grapes	0.37	0.12	
				21	Grapes	0.09	0.08	
				28	Grapes	0.13	0.12	
France, EU (South)	0.737	--	-	0	Grapes	0.40	0.02	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
2010 Grape/Carigan	0.779 0.738 [2.254]	29 13		7 14 21 28	Grapes Grapes Grapes Grapes	0.18 0.13 0.12 0.11	0.01 0.02 0.02 0.01	
France, EU (South) 2010 Grape/Grenache Gris	0.751 0.767 0.751 [2.269]	-- 32 15	-	0 7 14 21 28	Grapes Grapes Grapes Grapes Grapes	0.45 0.10 0.12 0.05 0.03	0.05 0.04 0.05 0.05 0.06	
France, EU (South) 2010 Grape/Sauvignon	0.725 0.761 0.724 [2.21]	-- 33 10	-	0 7 14 21 28	Grapes Grapes Grapes Grapes Grapes	0.33 0.15 0.16 0.13 0.08	0.01 0.01 0.02 0.02 0.02	
France, EU (South) 1991 Grape/Pinot noir	0.750 [0.75]	--	Bunches closed	0 15 32 46 64	Grapes Grapes Grapes Grapes Grapes	1.9 0.3 0.05 0.06 0.05	n/a n/a n/a n/a n/a	RJ1112B Ryan, J. and Sapiets, A. 1992b
Greece, EU (South) 1991 Grape/Savatiano	0.723 0.756 0.748 0.742 [2.969]	-- 30 36 26	Close to ripening	22	Grapes	6.80 [7.42] Mean = 7.11	0.51 [0.58] Mean = 0.55	6649-96-0022-CR-001 Dvorak, R.S. and Kenyon, R.G. 1996
France, EU (South) 1999 Grape/Cabernet-Sauvignon	0.728 [0.729]	--	BBCH 77	81	Grapes	0.03	n/a	734387 Wais, A. 2000
France, EU (South) 1999 Grape/Grenache	0.754 [0.754]	--	BBCH 77-79	70	Grapes	0.02	n/a	
Greece, EU (South) 2008 Grape/Rhoditis	0.742 [0.742]	--	BBCH 69	92	Grapes	<0.01	<0.01	ISK/FLU/08001 Heilaut, C. 2009
Greece, EU (South) 2008 Grape/Chardonnay	0.742 [0.742]	--	BBCH 69	77	Grapes	<0.01	<0.01	
Greece, EU (South) 2010 Grape/Victoria	0.784 [0.784]	--	BBCH 69	69	Grapes	<0.01	0.05, [0.08], [0.08], <0.01	S10-00193 Gemrot, F. 2011b
Greece, EU (South) 2010 Grape/Soultania	0.768 [0.768]	--	BBCH 89	98	Grapes	<0.01	0.10, [0.17], [0.18] 0.02	
Greece, EU (South)	0.760 0.750	-- 9	Grapes just larger than	0	Grapes	1.09, 1.16 (1.13)	0.14, 0.13 (0.14)	6245-95-0001-CR-003

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
1994 Grape/Sultana	0.750 0.750 0.750 0.750 [4.51]	5 12 21 21	pea size	21	Grapes	0.06, 0.05 (0.06)	0.18, 0.18 (0.18)	Jablonski, J.E. 1995c
				30	Grapes	0.10, 0.09 (0.10)	0.17, 0.16 (0.17)	
				45	Grapes	0.11, 0.18 (0.15)	0.21, 0.21 (0.21)	
	0.500 0.500 0.500 0.500 0.500 0.500 [3.0]	-- 9 5 12 21 21	Grapes just larger than pea size	0	Grapes	1.00, 1.06 (1.03)	0.04, 0.09 (0.07)	
				21	Grapes	0.15, 0.15 (0.15)	0.30, 0.28 (0.29)	
				30	Grapes	0.09, 0.10 (0.10)	0.20, 0.19 (0.20)	
				45	Grapes	0.07, 0.07 (0.07)	0.20, 0.21 (0.21)	

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Results in square brackets represent the re-analysis of the same analytical sample

n/a = not analysed

Blueberries

Thirteen residue trials were conducted in Canada and the USA in 2003 and 2004.

Six foliar applications were made using an SC formulation at application rates in the range of 0.706–1.166 kg ai/ha.

Samples of berries were collected 23-51 days after the last treatment.

Samples were immediately frozen and maintained in frozen storage for periods of up to 162 days for fluazinam and for up to 229 days for AMGT prior to extraction and analysis.

Residues of fluazinam and AMGT in blueberries were determined using analytical method 3. Procedural recovery samples were analysed with the residue trial samples. Fortification levels for fluazinam of 0.01-3 mg/kg were made with recoveries in the range of 60–140%. Fortification levels for AMGT of 0.01-1 mg/kg were made with recoveries in the range of 58–125%.

Table 95 Residues in Blueberries from supervised trials in Canada and the USA involving 6 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP USA	MID: 0.73 MTD: 4.38	7-10	Some ripe fruit	30	-	-	-	-
Jonesboro, ME, USA 2003 Blueberry/wild low bush	0.729 0.729 0.729 0.729 0.751 0.740 [4.406]	-- 6 7 7 6 7	Vegetative, bloom	50	Berries	0.24, 0.41 (0.33)	0.060, 0.082 (0.071)	IR-4 PR No. 06129 Thompson, D.C. 2006a
	0.729 0.751 0.729 0.729 0.740 0.751 [4.428]	-- 6 7 8 7 7	Vegetative	28	Berries	0.45, 0.49 (0.47)	0.10, 0.12 (0.11)	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Chatsworth, NJ, USA 2003 Blueberry/Blue ray [a]	0.706 0.729 0.762 0.729 0.751 0.762 [4.439]	-- 7 8 7 7 7	Fruiting	47	Berries	0.066, 0.082 (0.074)	<0.02, <0.02 (<0.02)	
	0.717 0.729	-- 7	Fruiting	32	Berries	0.42, 0.68 (0.55)	0.051, 0.046 (0.049)	
	0.729 0.751 0.729 0.740 [4.394]	7 7 7 8		39	Berries	0.28, 0.26 (0.27)	0.032, 0.036 (0.034)	
Chatsworth, NJ, USA 2003 Blueberry/Blue crop [a]	0.717 0.729 0.740 0.740 0.740 0.751 [4.417]	-- 7 8 7 7 7	Fruiting	47	Berries	0.11, 0.095 (0.10)	0.020, 0.023 (0.022)	IR-4 PR No. 06129 Thompson, D.C. 2006a
	0.729 0.740	-- 7	Fruiting	32	Berries	1.2, 1.0 (1.1)	0.026, 0.042 (0.034)	
	0.740 0.740 0.740 0.751 [4.439]	7 7 7 8		39	Berries	0.34, 0.42 (0.38)	0.037, 0.043 (0.040)	
Castle Hayne, NC, USA 2003 Blueberry/Premier	0.729 0.729 0.717 0.729 0.729 0.717 [4.349]	-- 6 7 7 7 7	Fruiting	50	Berries	0.16, 0.14 (0.15)	0.054, 0.056 (0.055)	IR-4 PR No. 06129 Thompson, D.C. 2006a
	0.729 0.729 0.717 0.729 0.729 0.762 [4.349]	-- 7 7 8 8 6	Fruiting	28	Berries	0.50, 0.55 (0.53)	0.055, 0.072 (0.064)	
Fennville, MI, USA [b] 2003 Blueberry/Rubel	0.740 0.740 0.706 0.706 0.706 0.740 [4.338]	-- 6 7 7 7 7	Fruiting	50	Berries	0.042, 0.034 (0.038)	<0.02, <0.02 (<0.02)	IR-4 PR No. 06129 Thompson, D.C. 2006a
	0.706 0.717	-- 7	Fruiting	28	Berries	0.16, 0.12 (0.14)	0.13, 0.12 (0.13)	
	0.729 0.740 0.695 0.717 [4.305]	7 7 7 8		38	Berries	0.28, 0.22 (0.25)	0.17, 0.16 (0.17)	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Fennville, MI, USA [b] 2003 Blueberry/Rubel	0.717 0.706 0.729 0.706 0.706 0.729 [4.293]	-- 6 7 7 8 6	Fruiting	50	Berries	0.038, 0.017 (0.028)	0.12, 0.10 (0.11)	IR-4 PR No. 06129 Thompson, D.C. 2006a
	0.706 0.706 0.729 0.717 0.740 0.717 [4.316]	-- 8 6 8 6 7	Fruiting	29	Berries	0.064, 0.074 (0.069)	0.11, 0.11 (0.11)	
Fennville, MI, USA 2003 Blueberry/Rubel	0.740 0.729 0.740 0.706 0.729 0.706 [4.349]	-- 6 7 7 7 7	Fruiting	51	Berries	0.038, 0.065 (0.052)	0.078, 0.056 (0.067)	
	0.706 0.717 0.706 0.706 0.706 0.706 [4.249]	-- 7 7 7 7 7	Fruiting	30	Berries	0.17, 0.13 (0.15)	0.081, 0.099 (0.090)	
Burlington, WA, USA 2003 Blueberry/Blue crop	0.740 0.751 0.773 0.740 0.729 0.740 [4.473]	-- 7 7 7 7 7	Green fruit	50	Berries	0.36, 0.42 (0.39)	0.022, <0.02 (0.021)	
	0.740 0.729 0.717 0.729 0.729 0.729 [4.372]	-- 7 7 7 7 7	Fruiting	29	Berries	1.5, 1.2 (1.4)	0.025, 0.026 (0.026)	
Aurora, OR, USA 2003 Blueberry/Blue crop	0.751 0.762 0.762 0.751 0.729 0.762 [4.518]	-- 7 7 7 7 7	Green fruit, 5-10% blossoms remain	50	Berries	0.50, 0.47 (0.49)	0.052, 0.061 (0.057)	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
	0.740 0.729 0.751 0.740 0.751 0.729 [4.439]	-- 7 7 8 6 7	Green fruit	29	Berries	0.70, 0.64 (0.67)	0.084, 0.078 (0.081)	
St Andrews , PEI, Canada 2003 Blueberry/Wild low bush	0.751 0.751 0.751 0.762 0.751 0.751 [4.518]	-- 6 7 6 8 4	Fruiting	47	Berries	1.1, 0.86 (0.98)	0.064, 0.070 (0.067)	IR-4 PR No. 06129 Thompson, D.C. 2006a
	0.729 0.751 0.762 0.762 0.762 0.762 [4.529]	-- 8 4 4 6 8	Fruiting	29	Berries	1.6, 1.7 (1.7)	0.076, 0.084 (0.080)	
Hermanville, PEI, Canada 2003 Blueberry/ Wild low bush	0.717 0.751 0.740 0.740 0.695 0.729 [4.372]	-- 6 7 6 8 4	Fruiting	27	Berries	0.39 [0.42] 0.43 [0.41] (0.41)	0.066, 0.074 (0.070)	
	0.740 0.762 0.740 0.762 0.762 0.729 [4.495]	-- 8 4 4 6 8	Fruiting	29	Berries	1.6, 2.0 (1.8)	0.11, 0.094 (0.10)	
St-Paul d'Abbotsford, QC, Canada 2003 Blueberry/Northland	0.729 0.807 0.740 0.751 0.773 0.729 [4.529]	-- 3 4 6 7 6	Fruiting	22	Berries	0.19	0.1	
	0.729 0.807 0.740 0.751 0.773 0.729 [4.529]	-- 7 6 7 7 7	Fruiting	23	Berries	0.07	0.096	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Truro, NS, Canada 2004 Blueberry/Wild low bush	1.132 1.166 1.132 1.143 1.143 1.110 [6.827]	-- 9 7 9 6 6	Fruiting	28	Berries	3.0, 2.7 (2.9)	0.28, 0.24 (0.26)	

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Results in square brackets represent the re-analysis of the same analytical sample

n/a = not analysed

[a] Replicate trials. HR taken

[b] Replicate trials. HR taken.

Bulb onions

Nine residue trials were conducted in the USA in 2005 and 2006.

Six foliar applications were made using an SC formulation at application rates in the range of 0.555–0.631 kg ai/ha.

Samples of bulb onion were collected 6-8 days after the last treatment.

Samples were immediately frozen and maintained in frozen storage for periods of up to 418 days prior to extraction and analysis.

Residues of fluazinam in bulb onion were determined using the analytical method outlined above. Procedural recovery samples were analysed with the residue trial samples. Fortification levels for fluazinam of 0.01-1 mg/kg were made with recoveries in the range of 84 –117%.

Table 96 Residues in bulb onion from supervised trials in USA involving 6 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	Reference
GAP USA	MID: 0.583 MTD: 3.51 [a]	7-10	-	7	-	-	-
Freeville, NY, USA 2005 Onion/ Millennium	0.581 0.631 0.585 0.555 0.573 0.584 [3.509]	-- 7 8 7 7 7	9 true leaves	6	Bulb	<0.01, <0.01 (<0.01)	IR-4 PR No. 07092 Carpenter, D.H. 2008a 07092.05-NY04
Arlington, WI, USA 2005 Onion/ Frontier	0.590 0.578 0.602 0.576 0.591 0.594 [3.531]	-- 6 8 7 6 6	Vegetative, bulb filling	6	Bulb	0.045, 0.035 (0.04)	IR-4 PR No. 07092 Carpenter, D.H. 2008a 07092.05-WI05

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	Reference
Weslaco, TX, USA 2006 Onion/ El Toro	0.578 0.583 0.581 0.583 0.582 0.581 [3.487]	-- 7 7 7 8 6	Bulbs formed	7	Bulb	<0.01, <0.01 (<0.01)	IR-4 PR No. 07092 Carpenter, D.H. 2008a 07092.05-TX*15
Fort Collins, CO, USA 2005 Onion/Vantage	0.604 0.584 0.618 0.581 0.561 0.606 [3.551]	-- 6 7 6 7 7	Bulbing	7	Bulb	0.095, 0.101 [b] (0.098)	IR-4 PR No. 07092 Carpenter, D.H. 2008a 07092.05-CO09
Mesilla, NM, USA 2005 Onion/ Cimerron	0.586 0.590 0.586 0.577 0.586 0.586 [3.512]	-- 7 7 7 7 7	10-12 true leaves	6	Bulb	<0.01, <0.01 (<0.01)	IR-4 PR No. 07092 Carpenter, D.H. 2008a 07092.05-NM10
Holtville, CA, USA 2006 Onion/Ebano	0.591 0.587 0.574 0.583 0.586 0.586 [3.512]	- 8 5 7 8 6	Bulbs	1 8 14 21	Bulb	0.096, 0.074 (0.085) 0.031, 0.033 (0.032) <0.01, <0.01 (0.01) <0.01, <0.01 <0.01)	IR-4 PR No. 07092 Carpenter, D.H. 2008a 07092.05-CA48
Salinas, CA, USA 2006 Onion/Olympic F1	0.584 0.596 0.575 0.593 0.567 0.593 [3.508]	-- 7 8 7 7 7	Mature bulbs, 85-100% of tops down	7	Bulb	0.013, <0.01 (0.012)	IR-4 PR No. 07092 Carpenter, D.H. 2008a 07092.05-CA49
Aurora, OR, USA 2005 Onion/Gunnison	0.590 0.586 0.569 0.572 0.618 0.596 [3.531]	-- 6 8 7 6 6	Vegetative	7	Bulb	0.015, 0.016 (0.016)	IR-4 PR No. 07092 Carpenter, D.H. 2008a 07092.05-OR04
Moxee, WA, USA 2005 Onion/Olympic F1	0.593 0.585 0.585 0.593 0.594 0.589 [3.539]	-- 7 7 7 8 6	Vegetative	7	Bulb	0.013, 0.021 (0.017)	IR-4 PR No. 07092 Carpenter, D.H. 2008a 07092.05-WA*06

[a] The GAP authorised is restricted to a maximum of 6 applications

[b] Highest individual sample result

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Brassica vegetables

Broccoli

Thirteen residue trials were conducted in Canada and the USA in 2003 and 2004.

In each trial a single drench application at transplanting was made using an SC formulation at application rates 0.025 kg ai/hL (equivalent to 0.025 kg ai/1000 plants).

Samples of mature broccoli were collected 50-113 days after the last treatment.

Samples were immediately frozen and maintained in frozen storage for periods of up to 182 days prior to extraction and analysis.

Residues of fluazinam in broccoli were determined using the analytical methods 1 and 9. Procedural recovery samples were analysed with the residue trial samples. Fortification levels for fluazinam of 0.01–0.1 mg/kg were made with recoveries in the range of 57–110%.

The trials cannot be relied on as a result of the samples being subjected to significant temperature variations during the time period from sampling to analysis. Storage data generated under the same conditions confirmed the instability of residues.

Table 97 Residues in Broccoli from supervised trials in Canada and the USA involving one soil drench application of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/hL)	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP USA	0.025 kg/hl (i.e. 0.025 kg ai/1000 plants)	-	Soil drench at or after transplanting	50	-	-	-	-
Freeville, NY, USA 2004 Broccoli/Everest	0.025 (100 mL/plant)	--	3-leaf stage	61	Broccoli heads	<0.01, <0.01 (<0.01)	n/a	IR-4 PR No. 08795 Thompson, D.C. 2006b
Weslaco, TX, USA 2004 Broccoli/Buccaneer	0.025 (100 mL/plant)	--	second true leaves	83	Broccoli heads	<0.01, <0.01 (<0.01)	n/a	
Parlier, CA, USA 2004 Broccoli/Green Magic	0.025 (100 mL/plant)	--	2-3 true leaves	78	Broccoli heads	<0.01, <0.01 (<0.01)	n/a	
Parlier, CA, USA 2004 Broccoli/Green Magic	0.025 (100 mL/plant)	--	2-3 true leaves	113	Broccoli heads	<0.01, <0.01 (<0.01)	n/a	
Holtville CA, USA 2004 Broccoli/Marathon	0.025 (100 mL/plant)	--	Transplant	87	Broccoli heads	<0.01, <0.01 (<0.01)	n/a	

Location, Country Year, Crop/Variety	Rate (kg ai/hL)	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Salinas CA, USA 2004 Broccoli/Heritage	0.025 (100 mL/plant)	--	2-3 true leaves	67	Broccoli heads	<0.01, <0.01 (<0.01)	n/a	
Salinas CA, USA 2004 Broccoli/Marathon	0.025 (100 mL/plant)	--	3 true leaves	78	Broccoli heads	<0.01, <0.01 (<0.01)	n/a	IR-4 PR No. 08795 Thompson, D.C. 2006b
Aurora, OR, USA 2004 Broccoli/Waltham	0.025 (100 mL/plant)	--	Vegetative	55	Broccoli heads	<0.01, <0.01 (<0.01)	n/a	
Harrow, ON, Canada 2003 Broccoli/Paragon	0.025 (100 mL/plant)	--	-	50	Broccoli heads	<0.01, <0.01 (<0.01)	n/a	
Harrow, ON, Canada 2003 Broccoli/Paragon	0.025 (100 mL/plant)	--	-	56	Broccoli heads	<0.01, <0.01 (<0.01)	n/a	
St Remi, QC, Canada 2003 Broccoli/Patron	0.025 (100 mL/plant)	--	-	82	Broccoli heads	<0.01, <0.01 (<0.01)	n/a	
St Remi, QC, Canada 2003 Broccoli/Decathalon	0.025 (100 mL/plant)	--	-	70	Broccoli heads	<0.01, <0.01 (<0.01)	n/a	
Agassiz, BC, Canada 2003 Broccoli/Arcadia	0.025 (100 mL/plant)	--	-	67	Broccoli heads	<0.01, <0.01 (<0.01)	n/a	

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Results in square brackets represent the re-analysis of the same analytical sample

n/a = not analysed

Cabbage

Twenty residue trials were conducted in Canada and the USA between 2003 and 2012.

In each trial a single drench application at transplanting was made using an SC formulation at application rates of 0.025 kg ai/hL with 100 ml of the solution being applied per plant (i.e. 0.025 kg ai/1000 plants). In eight of the trials six additional foliar applications were made using an SC formulation at application rates in the range of 0.52–0.61 kg ai/ha.

Samples of cabbage heads were collected 0-104 days after the last treatment.

Samples were immediately frozen and maintained in frozen storage for periods of up to 526 days for fluazinam and 463 days for AMGT prior to extraction and analysis.

Residues of fluazinam in cabbage were determined using the analytical methods 7 and 8. Residues of AGMT in cabbage for some trials were determined using the analytical method 7 outlined above. Procedural recovery samples were analysed with the residue trial samples. Fortification levels of 0.01-10 mg/kg for fluazinam and AMGT were made with recoveries in the range of 50–113% and 70–99% for fluazinam and AMGT, respectively.

A number of the trials cannot be relied on as a result of the samples being subjected to significant temperature variations during the time period from sampling to analysis. Storage data generated under the same conditions confirmed the instability of residues. These trials are marked (‡). For all other trials the crops were were maintained at a temperature of ≤ -18 °C throughout the study and can be relied on.

Table 98 Residues in Cabbage from supervised trials in Canada and the USA involving one soil drench and 6 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP USA	Soil drench: 0.025 kg/hl (i.e. 0.025 kg ai/1000 plants) Foliar: MID: 0.561 MTD: 3.36	7	-	7	-	-	-	-
Freeville NY, USA 2003 Cabbage/Amtrak	0.025 kg ai/hL	--	3-4 leaf transplants	94	Cabbage heads	<0.01, <0.01 (<0.01)	n/a	IR-4 PR No. 08796 [a] Thompson, D.C. 2006c
Salisbury, MD, USA 2003 Cabbage/CXB93256	0.025 kg ai/hL	--	Transplants, second true leaves	104	Cabbage heads	<0.01, <0.01 (<0.01)	n/a	
Holt MI, USA 2003 Cabbage/Blue Lagoon	0.025 kg ai/hL	--	Seedling	77	Cabbage heads	<0.01, <0.01 (<0.01)	n/a	
Weslaco, TX, USA 2003 Cabbage/Blue Ventage	0.025 kg ai/hL	--	Vegetative	88	Cabbage heads	<0.01, <0.01 (<0.01)	n/a	
Citra, FL, USA 2003 Cabbage/Bravo	0.025 kg ai/hL	--	Transplant	70	Cabbage heads	<0.01, <0.01 (<0.01)	n/a	
Salinas, CA, USA 2003 Cabbage/Red Express	0.025 kg ai/hL	--	Vegetative transplants, 2-3 true leaves	90	Cabbage heads	<0.01, <0.01 (<0.01)	n/a	
La Salle, CO, USA 2003 Cabbage/Charmont	0.025 kg ai/hL	--	Vegetative, transplant	83	Cabbage heads	<0.01, <0.01 (<0.01)	n/a	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Harrow, ON, Canada 2003 Cabbage/Survivor	0.025 kg ai/hL	--	Seedling, just transplanted	63	Cabbage heads	<0.01, <0.01 (<0.01)	n/a	
Agassiz, BC, Canada 2003 Cabbage/Grenadier	0.025 kg ai/hL	--	Seedlings	60	Cabbage heads	<0.01, <0.01 (<0.01)	n/a	
St-Michel, QC, Canada 2003 Cabbage/Bronco	0.025 kg ai/hL	--	Transplant, 3-4 leaves	84	Cabbage heads	<0.01, <0.01 (<0.01)	n/a	
Weslaco, TX, USA 2012 Cabbage/Gonzales	Drench: 0.025 kg ai/hL Foliar: 0.566 0.557 0.571 0.575 0.573 0.562 [3.403]	-- 28 7 7 7 6 6	Heads 7.6-15.2 cm diameter	7	Cabbage heads	0.12, 0.13 (0.13)	<0.01, <0.01 (<0.01)	IR-4 PR No. 07093 Barney, W.P. 2014a
Freeville NY, USA 2012 Cabbage/Early Thunder	Drench: 0.025 kg ai/hL Foliar: 0.565 0.564 0.564 0.565 0.562 0.563 [3.382]	-- 35 7 7 7 6 8	Forming heads	6	Cabbage heads	1.4, 1.5 (1.5)	<0.01, <0.01 (<0.01)	
Las Cruces NM, USA 2012 Cabbage/Golden Acre	Drench: 0.025 kg ai/hL Foliar: 0.595 0.569 0.571 0.559 0.568 0.572 0.582 [4.017]	-- 14 8 8 8 8 8 8	Heads forming	7	Cabbage heads	0.14, 0.41 (0.28)	<0.01, <0.01 (<0.01)	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Salisbury, MD, USA 2012 Cabbage/Farao	Drench: 0.025 kg ai/hL Foliar: 0.562 0.549 0.553 0.554 0.553 0.553 [3.323]	-- 36 8 6 8 6 6	Marketable heads	7	Cabbage heads	0.49, 0.56 (0.53)	<0.01, <0.01 (<0.01)	
Arlington, WI, USA 2012 Cabbage/Kaitlin	Drench: 0.025 kg ai/hL Foliar: 0.565 0.580 0.563 0.569 0.555 0.564 [3.396]	-- 44 6 6 6 6 6	Vegetative	6	Cabbage heads	0.25, 0.21 (0.23)	<0.01, <0.01 (<0.01)	
Citra, FL, USA 2012 Cabbage/Benelli	Drench: 0.025 kg ai/hL Foliar: 0.566 0.568 0.567 0.573 0.562 0.569 [3.406]	-- 33 7 7 7 7 7	Vegetative	0	Cabbage heads	1.6, 2.8 (2.2)	<0.01, <0.01 (<0.01)	IR-4 PR No. 07093 Barney, W.P. 2014a
				3	Cabbage heads	2.3, 1.6 (2.0)	<0.01, <0.01 (<0.01)	
				7	Cabbage heads	1.7, 1.2 (1.5)	<0.01, <0.01 (<0.01)	
				9	Cabbage heads	0.16, 0.49 (0.33)	<0.01, <0.01 (<0.01)	
				14	Cabbage heads	1.4, 0.61 (1.0)	<0.01, <0.01 (<0.01)	
				21	Cabbage heads	0.61, 0.59 (0.60)	<0.01, <0.01 (<0.01)	
Charleston, SC, USA 2012 Cabbage/Blue Vantage	Drench: 0.025 kg ai/hL Foliar: 0.576 0.562 0.563 0.568 0.568 0.568 [3.406]	-- 36 8 6 6 8 7	Vegetative, head set	7	Cabbage heads	0.68, 0.65 (0.67)	0.011, <0.01 (<0.011)	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Clinton, NC, USA 2012 Cabbage/Bravo	Drench: 0.025 kg ai/hL Foliar: 0.584 0.552 0.609 0.559 0.547 0.552 [3.402]	-- 38 6 6 7 7 6	Vegetative	6	Cabbage heads	0.45, 0.32 (0.39)	<0.01, <0.01 (<0.01)	
Harrow, ON Canada 2005 Cabbage/Atlantis	0.025 kg ai/hL	--	Transplant	58	Cabbage heads	<0.01, <0.01 (<0.01)	n/a	AAFC03-066R Ballantine, J. 2006 Samples subjected to significant temperature variations during storage
Rougemont, QC Canada 2005 Cabbage/Bentley	0.025 kg ai/hL	--	Transplant	84	Cabbage heads	<0.01, <0.01 (<0.01)	n/a	

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Results in square brackets represent the re-analysis of the same analytical sample

n/a = not analysed

[a] Samples subjected to significant temperature variations during storage

Leafy vegetables

Mustard greens

Eleven residue trials were conducted in Canada and the USA in 2003.

In each trial a single drench application at transplanting was made using an SC formulation at application rates of 0.025 kg ai/hL with 100 ml of the solution being applied per plant (i.e. 0.025 kg ai/1000 plants).

Samples of mature mustard leaves were collected 22-78 days after the last treatment.

Samples were immediately frozen and maintained in frozen storage for periods of up to 682 days prior to extraction and analysis.

Residues of fluazinam in mustard leaves were determined using the analytical method 8. Procedural recovery samples were analysed with the residue trial samples. Fortification levels of 0.01–0.1 mg/kg for fluazinam were made with recoveries in the range of 64–104%.

The trials cannot be relied on as a result of the samples being subjected to significant temperature variations during the time period from sampling to analysis. Storage data generated under the same conditions confirmed the instability of residues.

Table 99 Residues in mustard greens from supervised trials in Canada and the USA involving one soil drench of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/hL) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP USA	MID: 0.025 MTD: 0.025 kg ai/hL	-	Soil drench at or after transplanting	20	-	-	-	-
Crossville, NY, USA 2003 Mustard greens/ Florida Broadleaf	0.025	--	-	31	Leaves	0.01, <0.01 (0.01)	n/a	IR-4 PR No. 08797 Thompson, D.C. 2006d
Salisbury, MD, USA 2003 Mustard greens/ Green Wave	0.025	--	Transplants, second true leaves	69	Leaves	<0.01, <0.01 (<0.01)	n/a	
Arlington , WI, USA 2003 Mustard greens/ Savanna	0.025	--	-	44	Leaves	<0.01, <0.01 (<0.01)	n/a	
Clinton, NC, USA 2003 Mustard greens/ Southern Giant Curled	0.025	--	Transplants	38	Leaves	0.01, 0.01 (0.01)	n/a	
Weslaco, TX, USA 2003 Mustard greens/ Florida Broadleaf	0.025	--	Vegetative	40	Leaves	<0.01, <0.01 (<0.01)	n/a	
Citra, FL, USA 2003 Mustard greens/ Green Wave	0.025	--	Transplant	37	Leaves	<0.01, <0.01 (<0.01)	n/a	
Salinas, CA, USA 2003 Mustard greens/Red Giant	0.025	--	Vegetative plants, 2 true leaves	49	Leaves	<0.01, <0.01 (<0.01)	n/a	
Parlier, CA, USA 2003 Mustard greens/ Florida Broadleaf	0.025	--	Vegetative, transplant ~2-3 true leaves	78	Leaves	<0.01, 0.01 (0.01)	n/a	
Harrow, ON, Canada 2003 Mustard greens/ Savanna	0.025	--	Seedling, just transplanted	22	Leaves	<0.01, <0.01 (<0.01)	n/a	

Location, Country Year, Crop/Variety	Rate (kg ai/hL) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Agassiz, BC, Canada 2003 Mustard greens/ Southern Giant Curled		--	Seedlings	38	Leaves	<0.01, <0.01 (<0.01)	n/a	IR-4 PR No. 08797 Thompson, D.C. 2006d
Sherrington, QC, Canada 2003 Mustard greens/ Green Wave	0.025	--	Transplant, 3-4 true leaves	25	Leaves	<0.01, <0.01 (<0.01)	n/a	

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Results in square brackets represent the re-analysis of the same analytical sample

n/a = not analysed

Lettuce

Fourteen residue trials were conducted in the USA in 2005 and 2006.

In each trial a single foliar application was made using an SC formulation at application rates of 1.0–1.1 kg ai/ha.

Samples of mature lettuces were collected 46-52 days after the last treatment.

Samples were immediately frozen and maintained in frozen storage for periods of up to 229 days prior to extraction and analysis.

Residues of fluazinam in lettuce leaves were determined using the analytical method 8 outlined above. Procedural recovery samples were analysed with the residue trial samples. Fortification levels of 0.01-1 mg/kg for fluazinam were made with recoveries in the range of 82–119%.

Table 100 Residues in lettuce from supervised trials in the USA involving one foliar application of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP USA	MID: 0.87 MTD: 0.87	--	-	30	-	-	-	-
Salisbury, MD, USA 2006 Head Lettuce/ Crispino MTO	1.105 [1.105]	--	2-3 true leaves	47	Lettuce heads	<0.01, <0.01 (<0.01)	n/a	IR-4 PR No. 06892 Carpenter, D.H. 2008b
Citra, FL, USA 2005 Head Lettuce/ Esmeralda	1.146 [1.146]	--	Seedling	49	Lettuce heads	<0.01, <0.01 (<0.01)	n/a	
Salinas CA, USA 2005 Head Lettuce/ Corona	1.132 [1.132]	--	4-5 true leaves	52	Lettuce heads	<0.01, <0.01 (<0.01)	n/a	
Salinas CA, USA 2005 Head Lettuce/ Hallmark W	1.131 [1.131]	--	Post thinning 3-4 true leaves	48	Lettuce heads	<0.01, <0.01 (<0.01)	n/a	IR-4 PR No. 06892 Carpenter, D.H.

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Parlier CA, USA 2005 Head Lettuce/Great Lakes 659	1.139 [1.139]	--	Vegetative	49	Lettuce heads	<0.01, <0.01 (<0.01)	n/a	2008b
Holtville, CA, USA 2005 Head Lettuce/ Coyote	1.067 [1.067]	--	~2 leaf	50	Lettuce heads	0.022, [0.012], <0.01 [<0.01] (0.013)	n/a	
Los Cruces, NM, USA 2005 Head Lettuce/ Icon	1.121 [1.121]	--	4-6 true leaves	46	Lettuce heads	<0.01, <0.01 (<0.01)	n/a	
Tifton, GA, USA 2005 Leaf Lettuce/ Red Grant Rapids	1.12 [1.12]	--	Vegetative	20	Lettuce leaves	0.022, 0.038 (0.03)	n/a	
Citra, FL, USA 2005 Leaf Lettuce/ Two Star	1.146 [1.146]	--	Seedling	25	Lettuce leaves	0.02, 0.02 (0.02)	n/a	
Salinas, CA, USA 2005 Leaf Lettuce/ SPX-0254	1.092 [1.092]	--	4-5 true leaves	30	Lettuce leaves	<0.01, <0.01 (<0.01)	n/a	
Salinas, CA, USA 2005 Leaf Lettuce/ Antigua	1.132 [1.132]	--	3-4 true leaves	27	Lettuce leaves	0.01, 0.02 (0.02)	n/a	
Parlier, CA, USA 2005 Leaf Lettuce/ Salad Bowl	1.177 [1.177]	--	Vegetative	25	Lettuce leaves	0.15, 0.16 (0.16)	n/a	
Holtville, CA, USA 2005 Leaf Lettuce/ Tehama	1.118 [1.118]	--	2-3 leaf	26	Lettuce leaves	1.45, 1.69 (1.57)	n/a	
Los Cruces, NM, USA 2005 Leaf Lettuce/ Red Sail	1.117 [1.117]	--	4-6 true leaves	32	Lettuce leaves	0.02, 0.02 (0.02)	n/a	

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Results in square brackets represent the re-analysis of the same analytical sample

n/a = not analysed

Cantaloupe

Eleven residue trials were conducted in Canada and the USA in 2007.

Six foliar applications were made using an SC formulation at application rates in the range of 0.839–0.958 kg ai/ha.

Samples of melon were collected 27-32 days after the last treatment. One decline trial was conducted and samples were collected from 6 to 34 days after the last application.

Trials 07097.07-TX*21 and 07097.07-TX*22, trials 07097.07-AZ*07 and 07097.07-AZ*08 and trials 07097.07-ON13 and 07097.07-ON14 were conducted at the same trial site at the same time. Hence they are regarded as replicate trials and not independent trials.

Samples were immediately frozen and maintained in frozen storage for periods of up to 1180 days prior to extraction and analysis.

Residues of fluazinam and AMGT in melon were determined using analytical method 10. Procedural recovery samples were analysed with the residue trial samples. Fortification levels for fluazinam and AMGT of 0.01–1 mg/kg were made with recoveries in the range of 84–101% and 79–102% for fluazinam and AMGT respectively.

Table 101 Residues in cantaloupe melon from supervised trials in Canada and the USA involving 6 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP USA	MID: 0.876 MTD: 5.26	7-10	-	30	-	-		-
Tifton, GA, USA 2007 Cantaloupe melon/Hale's Best Jumbo	0.874 0.882 0.874 0.879 0.877 0.869 [5.255]	-- 7 6 7 7 6	Fruiting	27	Fruit	0.025, 0.014 (0.02)	<0.01, <0.01 (<0.01)	IR-4 PR No. 07097 Thompson, D.C. 2011a 07097.07-GA*07
Weslaco, TX, USA 2007 Cantaloupe melon/Cruiser [a]	0.879 0.877 0.879 0.878 0.871 0.871 [5.253]	-- 6 7 7 7 7	Fruiting	28	Fruit	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No. 07097 Thompson, D.C. 2011a 07097.07-TX*21
Weslaco, TX, USA 2007 Cantaloupe melon/Primo [a]	0.875 0.878 0.878 0.878 0.873 0.873 [5.255]	-- 6 7 7 7 7	Fruiting	28	Fruit	<0.01, 0.012 (0.011)	<0.01, <0.01 (<0.01)	IR-4 PR No. 07097 Thompson, D.C. 2011a 07097.07-TX*22
Maricopa, AZ, USA 2007 Cantaloupe melon/Hale's Best Jumbo [a]	0.897 0.887 0.897 0.874 0.892 0.878 [5.324]	-- 8 7 6 7 7	Vegetative	32	Fruit	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No. 07097 Thompson, D.C. 2011a 07097.07-AZ*07
Maricopa, AZ, USA 2007 Cantaloupe melon/Top Mark [a]	0.886 0.893 0.887 0.917 0.877 0.881 [5.34]	-- 7 7 6 7 7	Vegetative	32	Fruit	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No. 07097 Thompson, D.C. 2011a 07097.07-AZ*08

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Salisbury, MD, USA 2007 Cantaloupe melon/Athena	0.868 0.874 0.868 0.871 0.864 0.871 [5.215]	-- 8 7 7 6 9	Fruits netting	27	Fruit	0.019, 0.077 (0.048)	<0.01, <0.01 (<0.01)	IR-4 PR No. 07097 Thompson, D.C. 2011a 07097.07-MD08
Las Cruces, NM, USA 2007 Cantaloupe melon/PMR-45	0.873 0.883 0.898 0.878 0.879 0.871 [5.281]	-- 7 6 7 7 7	Fruiting	6 12 19 28 34	Fruit	0.04, 0.068 (0.054) 0.023, 0.034(0.029) 0.012, 0.017(0.015) <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)	IR-4 PR No. 07097 Thompson, D.C. 2011a 07097.07-NM06
Holtville, CA, USA 2007 Cantaloupe melon/Golden express	0.882 0.883 0.882 0.880 0.878 0.885 [5.290]	-- 8 6 8 7 7	Bud, bloom, fruit	27	Fruit	0.026, 0.022 (0.024)	<0.01, <0.01 (<0.01)	IR-4 PR No. 07097 Thompson, D.C. 2011a 07097.07-CA35
L'Acadie, QC, Canada 2007 Cantaloupe melon/Athena	0.842 0.882 0.845 0.902 0.870 0.839 [5.179]	-- 7 7 7 7 7	BBCH 63; 3rd flower open on main stem	31	Fruit	0.015, 0.012 (0.014)	<0.01, <0.01 (<0.01)	IR-4 PR No. 07097 Thompson, D.C. 2011a 07097.07-QC06
Harrow, ON, Canada 2007 Cantaloupe melon/Athena‡	0.858 0.861 0.898 0.854 0.918 0.958 [5.346]	-- 6 6 7 8 7	Fruiting, grapefruit-sized fruit	27	Fruit	<0.01, 0.032 (0.021)	<0.01, <0.01 (<0.01)	IR-4 PR No. 07097 Thompson, D.C. 2011a 07097.07-ON13
Harrow, ON, Canada 2007 Cantaloupe melon/Primo [a]	0.862 0.865 0.873 0.859 0.905 0.859 [5.222]	-- 6 6 7 8 7	Fruiting, grapefruit-sized fruit	29	Fruit	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No. 07097 Thompson, D.C. 2011a 07097.07-ON14

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

[a] For replicate trials the HR from the two non-independent trials has been taken.

Cucumber

Six residue trials were conducted in the USA in 2012.

Five to seven applications were made using an SC formulation at application rates in the range of 0.438–1.509 kg ai/ha. The first application in all trials was a soil drench treatment with all subsequent applications being foliar applications.

Samples of cucumber were collected 6-7 days after the last treatment.

Samples were immediately frozen and maintained in frozen storage for periods of up to 480 days prior to extraction and analysis.

Residues of fluazinam and AMGT in cucumber were determined using analytical method 10. Procedural recovery samples were analysed with the residue trial samples. Fortification levels for fluazinam and AMGT of 0.01-1 mg/kg were made with recoveries in the range of 76–96% and 74–105% for fluazinam and AMGT respectively.

Table 102 Residues in cucumber from supervised trials in USA involving 5-7 applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP USA	0.876 × 4 [a]	7-10	-	7	-	-		-
Weslaco, TX, USA 2012 Cucumber/ Diamant	0.880 [b] 0.870 0.880 0.869 0.877 [4.375]	-- 11 7 6 7	Blooming and fruiting	7	Fruit	0.022, 0.032 (0.027)	<0.01, <0.01 (<0.01)	IR-4 PR No. 09238 Barney, W.P. 2014a 09238.12-TX03
Willard, OH, USA 2012 Cucumber/ Dasher II	0.438 [b] 0.897 0.860 0.916 0.876 1.509 0.877 [6.371]	-- 7 7 7 7 7 5	Fruiting	7	Fruit	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No. 09238 Barney, W.P. 2014a 09238.12-OH*02
Tifton, GA, USA 2012 Cucumber/ National pickling	0.888 [b] 0.886 0.867 0.878 0.871 [4.389]	-- 20 8 7 7	Fruiting	6	Fruit	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No. 09238 Barney, W.P. 2014a 09238.12-GA01
Citra, FL, USA 2012 Cucumber/ Dasher II	0.887 [b] 0.881 0.877 0.888 0.909 [4.441]	-- 30 7 7 7	Fruiting	7	Fruit	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No. 09238 Barney, W.P. 2014a 09238.12-FL08
Arlington, WI, USA 2012 Cucumber/ Fanfare	0.878 [b] 0.890 0.886 0.878 0.881 [4.412]	-- 27 7 7 6	Fruiting	7	Fruit	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No. 09238 Barney, W.P. 2014a 09238.12-WI03

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Salisbury, MD, USA 2012 Cucumber/ Minature white	0.885 [b] 0.872 0.778 0.869 0.872 0.868 [5.144]	-- 20 7 7 7	Mature fruits	7	Fruit	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No. 09238 Barney, W.P. 2014a 09238.12-MD05

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

[a] Only 4 applications at 0.876 kg ai/ha are permitted. The first application at 0.876 kg ai/ha may be made as soil drench at transplantation or when the plants have the first true leaves. The critical GAP is therefore four foliar applications.

[b] Soil drench treatments

Summer squash

Six residue trials were conducted in the USA in 2012.

Five applications were made using an SC formulation at application rates in the range of 0.540–0.916 kg ai/ha. The first application in all trials was a soil drench treatment with all subsequent applications being foliar applications.

Samples of summer squash were collected 6-7 days after the last treatment.

Samples were immediately frozen and maintained in frozen storage for periods from 400 days up to 472 days prior to extraction and analysis.

Residues of fluazinam and AMGT in summer squash were determined using analytical method 10. Procedural recovery samples were analysed with the residue trial samples. Fortification levels for fluazinam and AMGT of 0.01 mg/kg were made with recoveries in the range of 85 –96% and 89– 175% for fluazinam and AMGT respectively.

Table 103 Residues in summer squashes from supervised trials in USA involving 5 applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP USA	4.38 × 4 [a]	7-10	-	7	-	-		-
Willard, OH, USA 2012 Summer squash/Envy	0.540 [b] 0.881 0.842 0.916 0.880 [4.059]	-- 7 7 7 7	Fruiting	6	Fruit	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No. 08916 Barney, W.P. 2014b 08916.12-OH*01 Storage period = 400 days

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Citra, FL, USA 2012 Summer squash/Gentry	0.889 [b] 0.882 0.907 0.888 0.867 [4.432]	-- 14 7 7 7	Fruiting	7	Fruit	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No. 08916 Barney, W.P. 2014b 08916.12-FL07 Storage period = 472 days [c]
Freeville, NY, USA 2012 Summer squash/Multipik	0.882 [b] 0.876 0.888 0.881 0.887 [4.413]	-- 16 7 7 7	Blooming / Fruiting	6	Fruit	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No. 08916 Barney, W.P. 2014b 08916.12-NY03 Storage period = 400 days
Clinton, NC, USA 2012 Summer squash/Enterprise	0.871 [b] 0.883 0.880 0.865 0.865 [4.365]	-- 18 8 8 6	Fruiting, flowers	7	Fruit	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No. 08916 Barney, W.P. 2014b 08916.12-NC02 Storage period = 455 days [c]
Charleston, SC, USA 2012 Summer squash/Zucchini	0.878 [b] 0.884 0.881 0.879 0.877 [4.399]	-- 16 7 7 7	Blooming / Fruiting	6	Fruit	0.012, 0.011 (0.012)	<0.01, <0.01 (<0.01)	IR-4 PR No. 08916 Barney, W.P. 2014b 08916.12-SC*02 Storage period = 433 days
Davis, CA, USA 2012 Summer squash/Black Beauty	0.886 [b] 0.855 0.870 0.910 0.876 [4.397]	-- 17 7 7 7	Fruiting	7	Fruit	0.016, <0.01 (0.013)	<0.01, <0.01 (<0.01)	IR-4 PR No. 08916 Barney, W.P. 2014b 08916.12-CA19 Storage period = 411 days

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

[a] Only 4 applications at 0.876 kg ai/ha are permitted. The first application at 0.876 kg ai/ha may be made as soil drench at transplantation or when the plants have the first true leaves. The critical GAP is therefore 4 foliar uses.

[b] Soil drench treatments

[c] trials not supported by storage period demonstrate

Peppers (Bell and non-bell)

Thirteen residue trials were conducted in Canada and the USA in 2007.

Six applications were made using an SC formulation at application rates in the range of 0.462–0.963 kg ai/ha. The first two applications in all trials were soil drench treatment with all subsequent applications being foliar applications.

Trials 09556.07-ON11 and 09556.07-ON12 were conducted at the same trial site at the same time and are therefore regarded as replicate trials rather than independent trials. A number of other trials were conducted at the same trial site at the same time but as different varieties of pepper were used and the morphology is regarded as sufficiently different (bell pepper versus non-bell pepper) then the trials were regarded as independent.

Samples of pepper were collected 28-32 days after the last treatment. One decline trial was conducted with samples collected 7–35 days after the last treatment.

Samples were immediately frozen and maintained in frozen storage for periods of up to 1249 days prior to extraction and analysis.

Residues of fluazinam and AMGT in pepper were determined using analytical method 10. Procedural recovery samples were analysed with the residue trial samples. Fortification levels for fluazinam and AMGT of 0.01 mg/kg were made with recoveries in the range of 85–96% and 89–175% for fluazinam and AMGT respectively.

Table 104 Residues in pepper from supervised trials in Canada and the USA involving 6 applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP USA	MID: 0.876 MTD: 5.26 [a]	7-14	-	30	-	-		-
Citra, FL, USA 2007 Bell pepper/Aristotle	0.880 [b] 0.892 [b] 0.876 0.892 0.862 0.906 [5.195]	-- 7 7 7 6 8	Vegetative	31	Fruit	<0.01, <0.01 (<u><0.01</u>)	<0.01, <0.01 (<0.01)	IR-4 PR No 09556 Thompson, D.C. 2011b 09556.07-FL10
Parlier, CA, USA 2007 Bell pepper/Baron	0.880 [b] 0.878 [b] 0.885 0.850 0.873 0.887 [5.252]	-- 6 7 53 7 7	Fruiting	28	Fruit	0.022, 0.016 (<u>0.019</u>)	<0.01, <0.01 (<0.01)	IR-4 PR No 09556 Thompson, D.C. 2011b 09556.07-CA33
Irvine, CA, USA 2007 Bell pepper/Taurus	0.805 [b] 0.910 [b] 0.854 0.855 0.873 0.865 [5.163]	-- 7 7 15 7 7	Fruiting	32	Fruit	<0.01, <0.01 (<u><0.01</u>)	<0.01, <0.01 (<0.01)	IR-4 PR No 09556 Thompson, D.C. 2011b 09556.07-CA34

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Bridgeton, NJ, USA 2007 Bell pepper/Revolution	0.462 [b] 0.480 [b] 0.893 0.882 0.868 0.885 [4.470]	-- 8 8 8 7 7	Fruiting	7 13 20 28 35	Fruit	1.2, 0.06 (0.63) 0.11, 0.084 (0.097) 0.054, 0.038 (0.046) <0.01, <0.01 (<u><0.01</u>) <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)	IR-4 PR No 09556 Thompson, D.C. 2011b 09556.07-NJ16
Weslaco, TX, USA 2007 Bell pepper/Capistrano	0.881 [b] 0.880 [b] 0.874 0.878 0.873 0.883 [5.269]	-- 6 6 20 7 7	Bloom	28	Fruit	0.019, <0.01 (<u>0.015</u>)	<0.01, <0.01 (<0.01)	IR-4 PR No 09556 Thompson, D.C. 2011b 09556.07-TX*20
Clinton, NC, USA 2007 Bell pepper/Crusader	0.864 [b] 0.865 [b] 0.850 0.853 0.874 0.862 [5.168]	-- 7 7 21 7 6	Fruiting	29	Fruit	<0.01, 0.019 (<u>0.015</u>)	<0.01, <0.01 (<0.01)	IR-4 PR No 09556 Thompson, D.C. 2011b 09556.07-NC16
L'Acadie, Canada, 2007 Bell pepper/ Socrates	0.752 [b] 0.864 [b] 0.835 0.842 0.842 0.840 [4.974]	-- 7 7 7 7 7	BBCH 79	31	Fruit	0.038, 0.021 (<u>0.03</u>)	<0.01, <0.01 (<0.01)	IR-4 PR No 09556 Thompson, D.C. 2011b 09556.07-QC05
Harrow, ON, Canada, 2007 Bell pepper/ Boynton [c]	0.950 [b] 0.906 [b] 0.865 0.916 0.963 0.833 [5.433]	-- 7 8 13 7 6	Plum-sized peppers	30	Fruit	<0.01, 0.011 (<u>0.011</u>)	<0.01, <0.01 (<0.01)	IR-4 PR No 09556 Thompson, D.C. 2011b 09556.07-ON11
Harrow, ON, Canada, 2007 Bell pepper/ Staysgreen [c]	0.921 [b] 0.949 [b] 0.902 0.899 0.911 0.865 [5.448]	-- 7 8 13 7 6	Plum-sized peppers	30	Fruit	0.01, <0.01 (0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No 09556 Thompson, D.C. 2011b 09556.07-ON12

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Arlington, WI 2007 Non-bell pepper/Hungarian wax	0.863 [b] 0.861 [b] 0.841 0.834 0.847 0.852 [5.098]	-- 7 7 28 8 6	Bud, bloom, fruiting	31	Fruit	<0.01, 0.022 (0.016)	<0.01, <0.01 (<0.01)	IR-4 PR No 09556 Thompson, D.C. 2011b 09556.07-WI31
Citra, FL,USA 2007 Non-bell pepper/Mesilla	0.887 [b] 0.879 [b] 0.883 0.854 0.862 0.900 [5.265]	-- 7 7 7 6 8	Bloom	31	Fruit	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No 09556 Thompson, D.C. 2011b 09556.07-FL11
Weslaco, TX, USA 2007 Non-bell pepper/Tam Veracruz	0.874 [b] 0.881 [b] 0.884 0.871 0.880 0.894 [5.285]	-- 7 7 7 7 8	Fruiting	30	Fruit	0.036, 0.053, (0.054)	<0.01, <0.01 (<0.01)	IR-4 PR No 09556 Thompson, D.C. 2011b 09556.07-TX19
Las Cruces, NM, USA 2007 Non-bell pepper/Joe E. Parker	0.893 [b] 0.877 [b] 0.876 0.882 0.892 0.874 [5.294]	-- 9 6 55 7 7	Fruiting	28	Fruit	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IR-4 PR No 09556 Thompson, D.C. 2011b 09556.07-NM04

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

[a] The label states that the first application may be made at a rate of 0.876 kg ai/ha as a banded soil drench at transplant. The interval for the foliar uses is 7–14 days.

[b] Soil drench treatments

[c] For replicate trials the HR from the two non-independent trials has been taken.

Legume vegetables

Snap beans (succulent bean with pod)

Eleven residue trials were conducted in Canada and the USA in 2003 and 2004.

In ten of the trials at each site there were two treated plots; in one plot a single foliar application was made whereas in the second plot two foliar applications were made. In all cases applications were made using an SC formulation with applications in the range of 0.473–0.555 kg ai/ha. Snap beans were collected 10–24 days after the last treatment.

Trials 07602.03-QC12 and 07602.03-QC13 were conducted at the same location at the same time and are therefore are regarded as replicate trials and not independent trials.

Samples were immediately frozen and maintained in frozen storage for periods of up to 377 days prior to extraction and analysis.

Residues of fluazinam were determined using analytical method 1. Procedural recovery samples were analysed with the residue trial samples. Fortification levels for fluazinam of 0.02 mg/kg were made with recoveries in the range of 71–122%.

The trials cannot be relied on as a result of the samples being subjected to significant temperature variations during the time period from sampling to analysis. Storage data generated under the same conditions confirmed the instability of residues.

Table 105 Residues in snap beans from supervised trials in Canada and the USA involving 1-2 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	Reference
GAP USA	MID: 0.497 MTD: 1.02	7-10	-	14	-	-	-
Freeville, NY, USA	0.53	-	15% bloom	24	Bean with pod	<0.02, <0.02 (<0.02)	IR-4 PR No. 07602
2003							Starner, V.R. 2006a
Snap Bean/ Hystyle							07602.03-NY18
Salisbury, MD, , USA	0.51	-	First open bloom	17	Bean with pod	<0.02, <0.02 (<0.02)	IR-4 PR No. 07602
2003	0.506 0.513	- 3	50% bloom	14	Bean with pod	<0.02, <0.02 (<0.02)	Starner, V.R. 2006a
Snap Bean/ Slenderette	[1.019]						07602.03-MD17
Citra, FL, USA	0.513	-	First bloom	18	Bean with pod	<0.02, <0.02 (<0.02)	IR-4 PR No. 07602
	0.507	-	Mid bloom	10	Bean with pod	0.025, 0.032 (0.029)	
2003	0.521	4	(100% flowers)				Starner, V.R. 2006a
						0.046, 0.072 [b] (0.059)	
Snap Bean/ Leon	[1.028]			14			07602.03-FL56
						<0.02, <0.02 (<0.02)	
				20			
Lansing, MI, USA	0.498	-	90% bloom	14	Bean with pod	<0.02, <0.02 (<0.02)	IR-4 PR No. 07602
	0.521	-	90% bloom	12	Bean with pod	<0.02, <0.02 (<0.02)	
2003	0.510	4					Starner, V.R. 2006a
Snap Bean/ Hercules	[1.031]						07602.03-MI39
Holt, MI, USA	0.555	-	70% bloom	14	Bean with pod	<0.02, 0.029 (0.025)	IR-4 PR No. 07602
2003	0.525	-	100% bloom	13	Bean with pod	<0.02, 0.02 (0.02)	Starner, V.R. 2006a
	0.504	3					
Snap Bean/ Hercules	[1.029]						07602.03-MI40
Holtville, CA, USA	0.506	-	5% bloom	21	Bean with pod	<0.02, 0.02 (0.02)	IR-4 PR No. 07602
2004	0.503	-	32% bloom	11	Bean with pod	0.064, 0.060 (0.062)	Starner, V.R. 2006a
	0.510	6					
Snap Bean/ Ambra						0.050, 0.109 [b] (0.08)	07602.03-CA127
	[1.013]			15			
						<0.02, 0.02 (0.02)	
				20			
Kimberly, ID, USA	0.503	-	6% bloom	28	Bean with pod	<0.02, 0.02 (0.02)	IR-4 PR No. 07602
	0.5	-	42% bloom	22	Bean with pod	<0.02, 0.02 (0.02)	
2003	0.499	6					Starner, V.R. 2006a
Snap Bean/ Idelite	[0.999]						07602.03-ID08
Prosser, WA, USA	0.504	-	55% bloom	15	Bean with pod	<0.02 / <0.02 (<0.02)	IR-4 PR No. 07602
2003	0.507	-	40% bloom	15	Bean with pod	<0.02 / <0.02 (<0.02)	Starner, V.R. 2006a
	0.526	3					
Snap Bean/ Igloo	[1.032]						07602.03-WA20
New Glasgow, PE, Canada	0.51	-	3.3% bloom	20	Bean with pod	<0.02 / <0.02 (<0.02)	IR-4 PR No. 07602

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	Reference
2003 Snap Beans/ Goldrush	0.501 0.5 [1.001]	- 5	Mid bloom	15	Bean with pod	<0.02, 0.022§ (0.021)	Starner, V.R. 2006a 07602.03-PE04
St. Paul d'Abbotsford, QC, Canada 2003 Snap Beans/ Valdae [a]	0.529	-	30% bloom	22	Bean with pod	<0.02 / <0.02 (<0.02)	IR-4 PR No. 07602 Starner, V.R. 2006a 07602.03-QC12
	0.522 0.487 [1.011]	-2	60% bloom	21	Bean with pod	<0.02 / <0.02 (<0.02)	
St. Paul d'Abbotsford, QC, Canada 2003 Snap Beans/ Strike [a]	0.529	-	10% bloom	22	Bean with pod	<0.02 / <0.02 (<0.02)	IR-4 PR No. 07602 Starner, V.R. 2006a 07602.03-QC13
	0.516 0.473 [0.989]	- 3	25% bloom	21	Bean with pod	<0.02 / <0.02 (<0.02)	

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

[a] For replicate trials the HR from the two non-independent trials has been taken.

[b] Highest individual sample result

Lima beans (succulent bean without pod)

Seven residue trials were conducted in the USA in 2003.

Two foliar applications were made using an SC formulation at application rates in the range of 0.494–0.515 kg ai/ha.

Samples of beans were collected 28–71 days after the last application.

Samples were immediately frozen and maintained in frozen storage for periods of up to 254 days prior to extraction and analysis.

Residues of fluazinam were determined using analytical method 1. Procedural recovery samples were analysed with the residue trial samples. Fortification levels for fluazinam of 0.02 -1 mg/kg were made with recoveries in the range of 68–107%.

The trials cannot be relied on as a result of the samples being subjected to significant temperature variations during the time period from sampling to analysis. Storage data generated under the same conditions confirmed the instability of residues.

Table 106 Residues in lima beans from supervised trials in USA involving 2 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	Reference
GAP USA	MID: 0.497 MTD: 1.02	7-10	-	30	-	-	-
Brighton, NJ, USA 2003 Lima bean/ Bridgeton	0.498 0.507 [1.004]	- 5	43% bloom	41	Bean without pod	<0.02, <0.02 (<0.02)	IR-4 PR No. 08798 Starner V.R. 2006b 08798.03-NJ34
Salisbury, MD, USA 2003 Lima bean/ Burpee's improved bush	0.509 0.509 [1.018]	- 3	45-50% bloom	52	Bean without pod	<0.02, <0.02 (<0.02)	IR-4 PR No. 08798 Starner V.R. 2006b 08798.03-MD18

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	Reference
Tifton, GA, USA 2003 Lima bean/ Cangreen	0.506 0.512 [1.018]	- 4	Mid bloom (55% flowers)	28	Bean without pod	<0.02, <0.02 (<0.02)	IR-4 PR No. 08798 Starner V.R. 2006b 08798.03-GA*19
Parlier, CA, USA 2003 Lima bean/ Fordhook 242	0.508 0.513 [1.021]	- 4	40% bloom	71	Bean without pod	<0.02, <0.02 (<0.02)	IR-4 PR No. 08798 Starner V.R. 2006b 08798.03-CA129
Holtville, CA, USA 2003 Lima bean/ Fordhook, 242	0.508 0.515 [1.022]	- 4	37.5% bloom	70	Bean without pod	<0.02, <0.02 (<0.02)	IR-4 PR No. 08798 Starner V.R. 2006b 08798.03-CA130
Kimberly, ID, USA 2003 Lima bean/ Henderson	0.494 0.509 [1.003]	- 6	36% bloom	43	Bean without pod	<0.02, <0.02 (<0.02)	IR-4 PR No. 08798 Starner V.R. 2006b 08798.03-ID09
Aberdeen, ID, USA 2003 Lima bean/ Henderson	0.498 0.494 [0.992]	- 7	50% bloom	36	Bean without pod	<0.02, <0.02 (<0.02)	IR-4 PR No. 08798 Starner V.R. 2006b 08798.03-ID10

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Soya bean (dry)

Sixteen residue trials were conducted in the USA and one trial in Canada in 2010.

Two foliar applications were made using an SC formulation at application rates in the range of 0.549–0.717 kg ai/ha.

The last applications were made from full flowering (R2) to Pod formation (R3). Samples of the seed were collected 65–95 days after the last application.

Samples of soya bean seed were immediately frozen and maintained in frozen storage for periods of up to 99 days prior to extraction and analysis.

Residues of fluazinam and AMGT were determined using analytical method 10. Procedural recovery samples were analysed with the residue trial samples. Fortification levels for fluazinam of 0.01–0.1 mg/kg were made with recoveries in the range of 88–108%. For AMGT fortification levels of 0.01 mg/kg–0.1 mg/kg were made with recoveries in the range of 89.5–120.

Table 107 Residues in soya bean seeds from supervised trials in Canada and the USA involving 2 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP USA	MID: 0.583 MTD: 1.17	10-14	Early pod formation (R3)	-	-	-		-

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Seven Springs, NC, USA 2010 Soya bean/ Asgrow AG5605	0.561 0.561 [1.122]	- 9	Full flowering (R2)	66 76 87 95	Seed	<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, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-01
Suffolk, VA, USA 2010 Soya bean/ Pioneer 95Y20	0.594 0.717 [1.311]	- 11	Full flowering-Pod formation (R2- R3)	90	Seed	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-02
Cheneyville, LA, USA 2010 Soya bean, Terral 55R11	0.583 0.594 [1.177]	- 11	Pod formation (late R3)	90	seed	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-03
Proctor, AR, USA 2010 Soya bean, Armor 47G7RR	0.561 0.561 [1.122]	- 10	Beginning bloom (V7 R1)	82	Seed	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-04
Northwood, ND, USA 2010 Soya bean/ Pioneer 90Y41	0.561 0.561 [1.122]	- 10	Pod formation (R3)	72	Seed	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-05
Fisher, MN, USA 2010 Soya bean/ Asgrow AG00901	0.561 0.561 [1.122]	- 9	Pod formation (R3)	72	Seed	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-06

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Geneva, MN, USA 2010 Soya bean/ Pioneer 91Y70	0.561 0.561 [1.122]	- 10	Full flowering (R2)	80	Seed	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-07
Wyoming, IL, USA 2010 Soya bean/ AG 3130	0.583 0.561 [1.144]	- 10	Full flowering-Pod formation (R2- R3)	70	Seed	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-08
Fitchburg, WI, USA 2010 Soya bean/ S21-N6	0.549 0.561 [1.11]	- 10	Full flowering (R2)	70	Seed	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-09
Lesterville, SD, USA 2010 Soya bean/ Lantharn CS-0991236	0.561 0.561 [1.122]	- 16	Pod formation (R3)	65	Seed	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-10
Richland, IA, USA 2010 Soya bean/ Pioneer 92Y80	0.561 0.549 [1.11]	- 9	Full flowering-Pod formation (R2- R3)	94	Seed	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-11
Bagley, IA, USA 2010 Soya bean/ 93Y13-N203	0.549 0.561 [1.11]	- 10	Full flowering (R2)	79	Seed	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-12

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Shelbyville, IN, USA 2010 Soya bean/ D4523081	0.561 0.583 [1.144]	- 10	Full flowering-Pod formation (R2- R3)	70	Seed	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-13
Marysville, OH, USA 2010 Soya bean/SG-329RR	0.561 0.561 [1.122]	- 10	Full flowering-Pod formation (R2- R3)	36 46 56 66	Seed	<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, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-14
Leonard, MO, USA 2010 Soya bean/ Asgrow 3803	0.549 0.561 [1.11]	- 11	Full flowering (R2)	74	Seed	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-15
Cambridge, ON, Canada 2010 Soya bean/ Absoulte RR	0.572 0.549 [1.121]	- 9	Pod formation (R3)	67	Seed	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-16

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Pulses

Dried beans

Thirteen residue trials were conducted in the USA in 2003.

In each trial two applications were made using an SC formulation at application rates of 0.48–0.54 kg ai/ha.

Samples of dried beans were collected 31–57 days after the last treatment.

Samples were immediately frozen and maintained in frozen storage for periods of up to 245 days for fluazinam prior to extraction and analysis.

Residues of fluazinam were determined using analytical method 10. Procedural recovery samples were analysed with the residue trial samples. Fortification levels of 0.01-1 mg/kg for fluazinam and were made with recoveries in the range of 71–108%.

Table 108 Residues in Dried beans from supervised trials in the USA involving 2 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP USA	MID: 0.497 MTD: 1.02	7-10	-	30	-	-	-	-
Freeville NY, USA 2003 Dry bean/Cabernet	0.495 0.525 [1.020]	-- 13	1-3 inch beans	57	Beans	<0.01, <0.01 (<0.01)	n/a	IR-4 PR No. 06369 Thompson, D.C. 2006e
Holt, MI, USA 2003 Dry bean/ Redhawk kidney bean	0.537 0.539 [1.067]	-- 14	Fruiting	50	Beans	<0.01, <0.01 (<0.01)	n/a	
Arlington WI, USA 2003 Dry bean/ Redhawk kidney bean	0.508 0.509 [1.017]	-- 14	Bloom-fruiting	50	Beans	<0.01, <0.01 (<0.01)	n/a	
Brookings, SD, USA 2003 Dry bean/Vista Navy bean	0.504 0.512 [1.016]	-- 12	Full bloom	43	Beans	<0.01, <0.01 (<0.01)	n/a	
Brookings, SD, USA 2003 Dry bean/Schooner Navy bean	0.504 0.502 [1.006]	-- 12	Full bloom	47	Beans	<0.01, <0.01 (<0.01)	n/a	
Fargo, ND, USA 2003 Dry bean/Norstar Navy bean	0.507 0.504 [1.011]	-- 13	Early pod	39	Beans	<0.01, <0.01 (<0.01)	n/a	
Fargo, ND, USA 2003 Dry bean/Navigator Navy bean	0.515 0.515 [1.030]	-- 14	Early pod	35	Beans	<0.01, <0.01 (<0.01)	n/a	
Velva, ND, USA 2003 Dry bean/Bill-Z Pinto bean	0.484 0.492 [0.976]	-- 14	Late bloom	31	Beans	<0.01, <0.01 (<0.01)	n/a	
Yuma, CO, USA 2003 Dry bean/590 Pinto bean	0.520 0.507 [1.027]	-- 13	Full bloom	39	Beans	<0.01, <0.01 (<0.01)	n/a	IR-4 PR No. 06369 Thompson, D.C. 2006e

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Fort Collins CO, USA 2003 Dry bean/Olanthe Pinto bean	0.497 0.490 [0.987]	-- 15	Full bloom	36	Beans	<0.01, <0.01 (<0.01)	n/a	
Holtville CA, USA 2003 Dry bean/Apache	0.512 0.516 [1.028]	-- 15	Late bloom	41	Beans	<0.01, <0.01 (<0.01)	n/a	
Kimberly ID, USA 2003 Dry bean/Pinto bean	0.502 0.498 [1.000]	-- 13	Bloom	36	Beans	<0.01, <0.01 (<0.01)	n/a	
Prosser WA, USA 2003 Dry bean/Othello Pinto bean	0.504 0.498 [1.002]	-- 13	Bloom	40	Beans	<0.01, <0.01 (<0.01)	n/a	

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Results in square brackets represent the re-analysis of the same analytical sample

n/a = not analysed

Root and tuber vegetables

Carrot

Thirteen residue trials were conducted in Canada and the USA in 2005 and 2006.

Four to five foliar applications were made using an SC formulation at application rates in the range of 0.552–0.821 kg ai/ha. At each trial site there were two plots; in the first plot sprinkler irrigation occurred within 2 hours after the last treatment while in the second plot sprinkler irrigation occurred at least 24 hours after the last treatment. The trials conducted on each plot are regarded as replicate trials and as such the highest residue from each replicate plot has been taken to support the GAP.

Samples of carrot were taken 6–8 days after the last application. One decline trial was performed in which samples were taken from 1–20 days after the last application.

Samples of carrot were immediately frozen and maintained in frozen storage for periods of up to 449 days prior to extraction and analysis.

Residues of fluazinam were determined using analytical method 1. Procedural recovery samples were analysed with the residue trial samples. Fortification levels for fluazinam were made at 0.02 and 1 mg/kg with recoveries in the range of 70–119% except in one case where a recovery at 54% was obtained.

Table 109 Residues in carrots from supervised trials in Canada and the USA involving 4-5 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	Reference
GAP USA	MID: 0.583 MTD: 2.33†	7 -14	-	7	-	-	-
Willard, OH, USA 2005 Carrot/ Nevis	0.580 0.583 0.576 0.595 0.595 [2.929]	-- 7 7 7 7	-	7	Carrot root	<0.02, <0.02 (<u><0.02</u>)	IR-4 PR No. 07094 Barney, W.P. 2007 07094.05-OH*07
	0.583 0.582 0.573 0.594 0.587 [2.919]	-- 7 7 7 7	-	7	Carrot root	<0.02, <0.02 (<0.02)	
Weslaco, TX, USA 2006 Carrot/ Rex 248	0.583 0.580 0.591 0.589 [2.342]	-- 7 7 7	Vegetative, Roots formed	8	Carrot root	0.07, 0.19 (<u>0.13</u>)	IR-4 PR No. 07094 Barney, W.P. 2007 07094.05-TX*14
	0.581 0.582 0.583 0.585 [2.331]	-- 7 7 7	Vegetative, Roots formed	8	Carrot root	0.05, 0.07 (0.06)	
Salinas, CA, USA 2005 Carrot/ Nelson [c]	0.589 0.576 0.576 0.580 [2.32]	-- 7 7 7	Mature roots	7	Carrot root	0.10, 0.10 (<u>0.10</u>)	IR-4 PR No. 07094 Barney, W.P. 2007 07094.05-CA*45
	0.589 0.584 0.587 0.591 [2.351]	-- 7 7 7	Mature roots	7	Carrot root	0.09, 0.09 (0.09)	
Salinas, CA, USA 2005 Carrot/ Bolero [c]	0.592 0.595 0.578 0.576 [2.342]	-- 7 7 7	Large Roots, 1.9 -3.2 cm diameter	6	Carrot root	0.09, 0.11 (0.10)	IR-4 PR No. 07094 Barney, W.P. 2007 07094.05-CA*46
	0.584 0.587 0.596 0.599 [2.366]	-- 7 7 7	Large Roots, 1.9 -3.2 cm diameter	7	Carrot root	0.08, 0.09 (0.09)	
Moxee, WA, USA 2005 Carrot/ Siroco F1	0.583 0.590 0.589 0.590 [2.351]	-- 7 7 7	Vegetative	7	Carrot root	0.09, 0.08 (<u>0.09</u>)	IR-4 PR No. 07094 Barney, W.P. 2007

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	Reference
	0.600 0.590 0.589 0.591 [2.369]	-- 7 7 7	Vegetative	7	Carrot root	0.05, 0.04 (0.05)	07094.05-WA*05
Citra, FL, USA 2005 Carrot/ Indiana	0.585 0.595 0.591 0.593 [2.364]	-- 8 8 6	Vegetative, mature	6	Carrot root	0.34, 0.39 (0.37)	IR-4 PR No. 07094 Barney, W.P. 2007
	0.578 0.582 0.584 0.594 [2.338]	-- 8 8 6	Vegetative, mature	6	Carrot root	0.28, 0.24 (0.26)	07094.05-FL17
Riverside, CA, USA 2006 Carot/ SXC3292	0.552 0.582 0.574 0.582 [2.289]	- 8 6 9	Vegetative	1 8 13 20	Carrot root	0.62, 0.72 (0.67) 0.56, 0.46 (0.51) 0.43, 0.28 (0.34) 0.40, 0.28 (0.34)	IR-4 PR No. 07094 Barney, W.P. 2007
	0.564 0.552 0.592 0.580 [2.287]	- 8 6 9	Vegetative	1 8 13 20	Carrot root	0.57, 0.48 (0.53) 0.25, 0.16 (0.21) 0.43, 0.46 (0.45) 0.41, 0.46 (0.33)	07094.05-CA44
Parlier, CA, USA 2005 Carrot/ Davvers 126	0.574 0.589 0.589 0.592 [2.343]	-- 7 7 7	Near Maturity	7	Carrot root	0.23, 0.22 (0.23)	IR-4 PR No. 07094 Barney, W.P. 2007
	0.578 0.582 0.573 0.585 [2.318]	-- 7 7 7	Near maturity	7	Carrot root	0.02, 0.02 (0.02)	07094.05-CA47
Kentville, NS, Canada 2005 Carrot/ Sweetness II	0.843 0.785 0.801 0.813 [3.24]	 -- 8 7 6	8 true leaves	6	Carrot root	0.06, 0.04 (0.05)	IR-4 PR No. 07094 Barney, W.P. 2007
	0.810 0.794 0.802 0.805 [3.211]	-- 8 7 6	8 true leaves	6	Carrot root	0.05, 0.06 (0.06)	07094.05-NS01
Delhi, ON, Canada 2005 Carrot/ Sugarsnax 54 [b]	0.592 0.584 0.584 0.602 [2.362]	-- 7 8 6	Roots about 60% of size	7	Carrot roots	0.12, 0.12 (0.12)	IR-4 PR No. 07094 Barney, W.P. 2007

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	Reference
	0.567 0.581 0.591 0.608 [2.347]	-- 7 8 6	Roots about 60% of size	7	Carrot roots	0.04, 0.03 (0.04)	07094.05-ON16
Delhi, ON, Canada 2005 Carrot/ Sugarsnax 54 [b]	0.814 0.609 0.581 0.572 [2.576]	-- 7 7 6	7 -9 leaves	8	Carrot root	0.14, 0.12 (0.13)	IR-4 PR No. 07094 Barney, W.P. 2007
	0.821 0.591 0.591 0.581 [2.584]	-- 7 7 6	7–9 leaves	8	Carrot root	0.11, 0.10 (0.11)	07094.05-ON17
St Cypnien de Napierville, OC, Canada 2005 Carrot/ Appache	0.843 0.812 0.766 0.851 [3.272]	-- 8 7 6	Carrots, 25-30 cm	7	Carrot root	0.06, 0.05 (0.06)	IR-4 PR No. 07094 Barney, W.P. 2007
	0.834 0.854 0.827 0.881 [3.396]	-- 8 7 6	Carrots, 25-30 cm	7	Carrot root	0.19, 0.17 (0.18)	07094.05-QC01
Ste-Brigide d'iberville, QC, Canada 2005 Carrot/ Cello Bunch	0.858 0.790 0.843 0.804 [3.295]	-- 6 8 7	Roots about 15-20 cm	7	Carrot root	0.12, 0.11 (0.12)	IR-4 PR No. 07094 Barney, W.P. 2007
	0.829 0.828 0.818 0.807 [3.282]	-- 6 8 7	Roots about 15-20 cm	7	Carrot root	0.41, 0.45 (0.43)	07094.05-QC02

† A total of 4 applications can be made

[b] Replicate trials

[c] Replicate trials

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Potato

Eleven residue trials were conducted in Canada and the USA in 1992 to 1994.

Two to eleven foliar applications were made using an SC formulation at application rates in the range of 0.202 and 1.043 kg ai/ha. For the majority of trials there were two–three plots in which different application regimes were investigated.

Samples of potato were taken from 8-40 days after the last application.

Samples of potato were immediately frozen and maintained in frozen storage for periods of up to 476 days prior to extraction and analysis.

Residues of fluazinam were determined using analytical method 1. Procedural recovery samples were analysed with the residue trial samples. Fortification levels for fluazinam were made between 0.01 and 1 mg/kg with recoveries in the range of 70–122 %.

Table 110 Residues in potatoes from supervised trials in Canada and the USA involving foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	Reference
GAP USA	MID: 0.292 MTD: 2.04	7 -10	-	14	-	-	-
Exeter, ME, USA 1992 Potato/ Friti-Ly 945	0.202 × 9 [1.818]	7-10	Plants starting to die back	14	tuber	<0.01, <0.01 (<u>≤0.01</u>)	5349-92-0253-CR-001 Fitzgerald, T.J. 1994
	0.504 × 4 [2.016]	21-25	Plants starting to die back	14	Tuber	<0.01, <0.01 (<0.01)	
Ephrata, WA, USA 1992 Potato/ Russet Burbank	0.202 × 10 [2.02]	7	Potatoes 46-51 cm tall, no flowers, vines still green, vigorous tubers	8	Tuber	<0.01, <0.01 (<u>≤0.01</u>)	5349-92-0253-CR-001 Fitzgerald, T.J. 1994
	0.504 × 4 [2.016]	22-28	Potatoes 46-51 cm tall, no flowers, vines still green, vigorous tubers	14	Tuber	<0.01, <0.01 (<0.01)	
Madison, OH, USA 1992 Potato/ Katahdin	0.247 0.247 0.504 0.504 [1.502]	-- 1 41 29	Not stated	40	Tuber	<0.01, <0.01 (<0.01)	5197-92-0047-CR-001 Fitzgerald, T.J. and Kenyon, R.G. 1994
	0.504 0.504 0.504 [1.512]	-- 28 28	Not stated	32	Tuber	<0.01, <0.01 (<0.01)	
	0.202 × 10 [2.02]	6-8	Not stated	14	Tuber	<0.01, <0.01 (<0.01)	
Madison, OH, USA 1993 Potato/ Katahdin	0.538 2.590 2.601 [5.728]	-- 28 27	Not stated	14	Tuber	<0.01, <0.01 (<0.01)	5706-93-0105-FR-001 Fitzgerald, T.J. and McFall, D.D. 1996c
	0.213 0.348 0.213 0.213 0.213 1.043 1.043 1.009 1.043 [5.336]	-- 7 7 7 8 13 13 7 7	Not stated	14	Tuber	<0.01, <0.01 (<0.01)	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	Reference
Arkansaw, WI, USA 1993 Potato/ Russet Burbank	0.527 0.527 0.516 0.527 [2.096]	- 28 28 29	Plants in early senescence	14	Tuber	<0.01, <0.01 (<0.01)	5880-93-0342-CR- 001 McFall, D.D. 1996b
	0.213 × 11 [2.343]	8-10	Plants in early senescence	14	Tuber	<0.01, <0.01 (<0.01)	
Minidoka, ID, USA 1993 Potato/ Russet Burbank	0.527 0.504 0.516 [1.574]	- 28 27	Vines setting, plants vigorous	34	Tuber	<0.01, <0.01 (<0.01)	5880-93-0342-CR- 001 McFall, D.D. 1996b
	0.191 0.202 0.213 0.213 0.202 0.213 0.202 0.202 [1.637]	8-11	Not stated	13	Tuber	<0.01, <0.01 (<0.01)	
Eaton, CO, USA 1993 Potato/Snowdon	0.572 0.561 0.561 [1.693]	- 28 29	Tuber enlargement stage	14	Tuber	<0.01, <0.01 (<0.01)	5880-93-0342-CR- 001 McFall, D.D. 1996b
	0.224 × 8 [1.794]	7-10	Tuber enlargement stage	14	Tuber	<0.01, <0.01 (<0.01)	
Northwood, ND, USA 1994 Potato/ Irish Norchip	0.213 × 8 [1.704]	7-9	Nearing maturity	14	Tuber	<0.01, <0.01 (<0.01)	5880-93-0342-CR- 001 McFall, D.D. 1996b
Moses Lake, WA, USA 1994 Potato/Russet Burbank	0.213 0.213 0.213 0.213 0.213 0.213 0.213 0.202 [1.693]	7-10	45.7 cm tall, vines laying down between rows	14	Tuber	<0.01, <0.01 (<0.01)	5880-93-0342-CR- 001 McFall, D.D. 1996b
Portervile, CA, USA 1994	0.516 0.516 [1.032]	- 28	Not stated	14	Tuber	<0.01, <0.01 (<0.01)	5880-93-0342-CR- 001 McFall, D.D. 1996b

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	Reference
Potato/ Red LaSola	0.202 × 5 [1.009]	7	Not stated	14	Tuber	<0.01, <0.01 (<0.01)	
Portage La Prairie, MB, Canada 1994 Potato/ Russet Burbank	0.213 × 8 [1.704]	8-10	Plants maturing, no signs of early or late blight	18	Tuber	<0.01, <0.01 (<0.01)	5880-93-0342-CR-001 McFall, D.D. 1996b
Sommerset, NS, Canada 1994 Potato/ Kennebec	0.202 0.202 0.202 0.202 0.202 0.202 0.213 [1.48]		Not stated	14	Tuber	<0.01, <0.01 (<0.01)	5880-93-0342-CR-001 McFall, D.D. 1996b

‡Replicate trials

§Replicate trials

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Ginseng

Five residue trials were conducted in Canada and the USA in 2003 and 2007.

Four foliar applications were made using an SC formulation at application rates in the range of 0.092 and 1.3 kg ai/ha. In one trial two different application regimes were investigated.

Samples of ginseng were taken from 28-31 days after the last application.

Samples of ginseng were immediately frozen and maintained in frozen storage for periods of up to 332 days prior to extraction and analysis.

Residues of fluazinam were determined using analytical method 1. Procedural recovery samples were analysed with the residue trial samples. Fortification levels for fluazinam were made between 0.01 and 1 mg/kg with recoveries in the range of 64–110%.

The trials cannot be relied on as a result of the samples being subjected to significant temperature variations during the time period from sampling to analysis. Storage data generated under the same conditions confirmed the instability of residues.

Table 111 Residues in Ginseng from supervised trials in Canada and the USA involving foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	Reference
GAP USA	MID: 0.874 MTD: 3.51	7 -14	-	30	-	-	-
East Lansing, MI, USA 2003 Ginseng/ American Ginseng	0.926 0.877 0.897 0.905 [3.604]	-- 9 14 14	Fruiting	29	Root	1.2, 1.4 (1.3)	IR-4 PR No. 08791 Corley, J. 2006 08791.03-MI37

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	Reference
Holt, MI, USA 2003 Ginseng/ American Ginseng	0.874 0.890 0.872 0.874 [3.511]	-- 9 14 14	Fruiting	29	Root	0.73, 0.94 (0.84)	IR-4 PR No. 08791 Corley, J. 2006 08791.03-MI38
	1.768 1.934 1.823 1.881 [7.405]	-- 9 14 14	Fruiting	29	Root	2.2, 2.1 (2.2)	
Athens, WI, USA 2003 Ginseng/ American Ginseng	0.892 1.049 0.847 0.936 [3.725]	-- 14 14 14	Fruiting	31	Root	0.58, 0.96 (0.77)	IR-4 PR No. 08791 Corley, J. 2006 08791.03-WI17
Marathon, WI, USA 2003 Ginseng/ American Ginseng	0.936 0.892 1.035 0.937 [3.8]	-- 12 14 14	Fruiting	30	Root	0.46, 0.28 (0.37)	IR-4 PR No. 08791 Corley, J. 2006 08791.03-WI25
Summerland, BC, Canada 2007 Ginseng/ American Ginseng	0.905 0.920 0.903 1.013 [3.741]	-- 13 14 13	Fruiting	28	Root	0.071, 0.094, 0.130, 0.072 (0.092)	AAFC07-042R Ballantine, J. 2010b AAFC07-042R-386

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets. In the case of the Canadian trial four independent samples were taken from the trial site.

Oilseeds

Nine residue trials were conducted in the USA between 1991 and 1994.

In each trial two to three applications were made using an SC formulation at application rates in the range of 0.37– 1.12 kg ai/ha. In some trials applications were made either as a broadcast foliar spray or as a banded foliar application.

Peanut vines were inverted (dug out) and the crop was allowed to dry in the field for 7-10 days before samples were harvested by combine harvester and collected. Samples of whole peanuts were collected 17-59 days after the last treatment, and separated into nutmeat and hulls.

Samples were immediately frozen and maintained in frozen storage for periods of up to 176 days for nutmeat and 164 days for hulls prior to extraction and analysis.

Residues of fluazinam in peanut nutmeat and hulls were determined using analytical method 1. Procedural recovery samples were analysed with the residue trial samples. Fortification levels of 0.01–1 mg/kg for fluazinam were made for nutmeat and hulls with recoveries in the range of 62–119% and 78–120% for nutmeat and hulls, respectively.

Table 112 Residues in Peanuts from supervised trials in the USA involving 2–3 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP USA	MID: 0.874 MTD: 2.34	21-28	-	30	-	-	-	-
Waller County TX, USA 1993 Peanut/Florunner	0.773 0.758 0.759 [2.365] Broadcast	-- 28 28	-	29	Peanut Nutmeat	<0.01, <0.01 (<0.01)	n/a	5879-93-0335-CR-001 Hayes, P.C. Jr. and Kenyon, R.G. 1994
	0.619 0.614 0.616 [2.399] Banded	-- 28 28	-	29	Peanut Nutmeat	<0.01, <0.01 (<0.01)	n/a	
Skippers, VA, USA 1993 Peanut/NC-V11	0.746 0.686 0.763 [2.276] Broadcast	-- 29 31	-	30	Peanut Nutmeat	<0.01, <0.01 (<0.01)	n/a	
	0.771 0.766 0.752 [2.377] Banded	-- 29 31	-	30	Peanut Nutmeat	<0.01, <0.01 (<0.01)	n/a	
Shorterville, AL, USA 1993 Peanut/Florunner	0.752 0.761 0.752 [2.343] Broadcast	-- 27 29	Pod fill	58	Peanut Nutmeat	<0.01, <0.01 (<0.01)	n/a	
	0.372 0.377 0.432 [1.244] Banded	-- 27 29	Pod fill	58	Peanut Nutmeat	<0.01, <0.01 (<0.01)	n/a	
Eakly, OK, USA 1991 Peanut/Okrun	1.121 1.121 [2.242]	-- 29	R7 beginning maturity	59	Peanut Nutmeat	<0.01, <0.01 (<0.01)	n/a	2105-91-0307-CR-001 Kenyon, R.G. 1992b
	1.121 1.121 1.121 [3.363]	-- 29 29	R7 beginning maturity	31	Peanut Nutmeat	<0.01, <0.01 (<0.01)	n/a	
Lucama, NC, USA 1991	1.121 1.054 [2.175]	-- 30	Pegging	49	Peanut Nutmeat	<0.01, <0.01 (<0.01)	n/a	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Peanut/Florigiant	1.080 1.110 1.087 [3.277]	-- 30 32	Nut filling	17	Peanut Nutmeat	<0.01, <0.01 (<u>≤0.01</u>)	n/a	
Pinehurst, GA USA 1991 Peanut/Florunner	1.121 1.121 [2.242]	-- 31	Pre-harvest	46	Peanut Nutmeat	<0.01, <0.01 (<0.01)	n/a	2105-91-0307-CR-001 Kenyon, R.G. 1992b
	1.121 1.121 1.121 [3.363]	-- 31 29	Pre-harvest	17	Peanut Nutmeat	<0.01, <0.01 (<u>≤0.01</u>)	n/a	
Lucama, NC USA 1994 Peanut/NC-V11	0.762 0.762 0.773 [2.298] Broadcast	-- 32 31	Excellent growth, rows lapped, 33-43 cm tall	32	Peanut Nutmeat	<0.01, <0.01 (<0.01)	n/a	6107-95-0013-CR-001 McFall, D.D. 1995
	0.773 0.785 0.785 [2.343] Banded	-- 32 31	Excellent growth, rows lapped, 33-46 cm tall	32	Peanut Nutmeat	<0.01, <0.01 (<u>≤0.01</u>)	n/a	6107-95-0013-CR-001 McFall, D.D. 1995
Eakly, OK USA 1994 Peanut/Florunner	0.796 0.830 0.785 [2.42] Broadcast	-- 29 28	Damage from gopher is approx. 15-25% in the plots	33	Peanut Nutmeat	<0.01, <0.01 (<0.01)	n/a	
	0.796 0.886 0.796 [2.477] Banded	-- 29 28	Damage from gopher is approx. 15-25% in the plots	33	Peanut Nutmeat	<0.01, <0.01 (<u>≤0.01</u>)	n/a	
Montezuma, GA, USA 1994 Peanut/GK-7	0.874 0.818 0.796 [2.489] Broadcast	-- 28 25	not noted	41	Peanut Nutmeat	<0.01, <0.01 (<0.01)	n/a	
	0.863 0.796 0.796 [2.455] Banded	-- 28 25	not noted	41	Peanut Nutmeat	<0.01, <0.01 (<u>≤0.01</u>)	n/a	

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Results in square brackets represent the re-analysis of the same analytical sample

n/a = not analysed

Tea

Seven residue trials, in non-GLP studies, consisting of the analytical phase only, were conducted in Japan in 1986, 1992, 1993 and 1997. The studies were conducted following the national requirements applicable at the time.

For the trials conducted in 1986, each trial was divided into three plots; in the first plot an application rate at 1 kg ai/ha × 1 with a PHI of 7 days was investigated, in the second plot an application rate of 1 kg ai/ha × 1 with a 14 days was investigated and in the third plot an application rate of 1 kg ai/ha × 2 with a PHI of 21 days was investigated.

In the trials conducted in 1992, 1993 and 1997 the application rate was one foliar application of fluazinam at a rate of 0.025 kg ai/hL with samples collected 14 days after the application. In two of the trials three separate plots were treated and in addition to the application regime stated being investigated in one plot, in a second plot one application of 0.025 kg ai/hL was made with samples collected 7 days after treatment and in a third plot two applications of 0.025 kg ai/hL were made with samples collected 21 days after treatment.

Four independent representative samples were taken from each trial site. Two of the samples were analysed in one laboratory with the other two samples analysed in a different laboratory.

Samples were immediately frozen and maintained in frozen storage for periods of up to 6 months for the samples from the trials conducted in 1986 and up to 5 months for the samples from the 1992, 1993 and 1997 trials.

Residues of fluazinam, MAPA and HYPA were determined using analytical methods 1 and 12. CAPA was also determined in the trials from 1986 using analytical method 1. Residues were <0.02 mg/kg at each time point.

Procedural recovery samples were analysed with the residue trial samples. Fortification levels for fluazinam of 0.02–20 mg/kg were made with recoveries in the range of 68–107%.

Table 113 Residues in tea from supervised trials in Japan

Location, Country Year, Crop/Variety	Rate (kg ai/hL) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	MAPA (mg/kg)	HYPA (mg/kg)	Reference
GAP Japan	MID: 0.025 MTD:0.025	-	-	14	-	-			-
Kanagawa, Japan	0.025 [0.025]	-	-	7	Dried leaves	27.1, 26.2 (26.6)	0.33, 0.32 (0.32)	0.20, 0.19 (0.20)	Kato, S. 1987
1986	0.025 [0.025]	-	-	14	Dried leaves	2.87, 2.85 (2.86)	0.05, 0.04 (0.04)	<0.01, <0.01 (<0.01)	Kanagawa[a]
Tea/Yabukita	0.025 0.025 [0.05]	- 7	-	21	Dried leaves	0.60, 0.60 (0.60)	0.03, 0.02 (0.02)	<0.01, <0.01 (<0.01)	
Kanagawa, Japan	0.025 [0.025]	-	-	7	Dried leaves	22.8, 20.9 (21.8)	0.11, 0.09 (0.10)	0.25, 0.23, (0.24)	Hagi, I, 1996
1986	0.025 [0.025]	-	-	14	Dried leaves	3.39, 3.06 (3.23)	0.09, 0.04 (0.06)	0.04, 0.03 (0.04)	Kanagawa[a]
Tea/Yabukita	0.025 0.025 [0.05]	- 7	-	21	Dried leaves	0.80, 0.71 (0.76)	0.03, 0.02 (0.02)	0.01, 0.01 (0.01)	mean residue for (a) = 3.05 mg/kg
Aichi, Japan	0.025 [0.025]	-	-	7	Dried leaves	48.1, 48.1 (48.1)	0.82, 0.81 (0.82)	0.42, 0.41 (0.42)	Kato, S. 1987
1986	0.025 [0.025]	-	-	14	Dried leaves	8.20, 7.76 (7.98)	0.25, 0.23 (0.24	0.09, 0.09 (0.09)	Aichi[b]
Tea/Yabukita	0.025 0.025 [0.05]	- 7	-	21	Dried leaves	2.41, 2.37 (2.39)	0.08, 0.08 (0.08)	0.02, 0.02 (0.02)	
Aichi, Japan	0.025 [0.025]	-	-	7	Dried leaves	45.6, 44.3 (45)	0.90, 0.85 (0.88)	0.41, 0.39 (0.40)	Hagi, I, 1996
1986	0.025 [0.025]	-	-	14	Dried leaves	10.41, 9.46 (9.95)	0.24, 0.21 (0.22)	0.09, 0.07 (0.08)	Aichi[b]
Tea/Yabukita	0.025 0.025 [0.05]	- 7	-	21	Dried leaves	2.47, 2.34 (2.41)	0.17, 0.08 (0.12)	0.02, 0.01 (0.02)	mean residue for (b) = 8.97 mg/kg
Kanagawa, Japan	0.025	-	-	7	Dried Leaves	32.2, 30.8 (31.5)	0.29, 0.28 (0.28)	0.14, 0.14 (0.14)	Komatsu, K. and Yabusaki. T. 1993

Location, Country Year, Crop/Variety	Rate (kg ai/hL) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	MAPA (mg/kg)	HYPA (mg/kg)	Reference
1992 Tea/Yabukita	0.025	-	-	14	Dried Leaves	2.78, 2.59 (2.68)	0.08, 0.07 (0.08)	0.04, 0.02 (0.03)	Kanagawa[c]
	0.025 0.025 [0.05]	- 7	-	21	Dried Leaves	0.50, 0.48 (0.49)	0.02, 0.02 (0.02)	0.02, 0.02 (0.02)	
Kochi, Japan 1993 Tea/Yabukita	0.025	-	-	7	Dried Leaves	31.1 , 30.1 (30.6)	0.36 , 0.35 (0.36)	0.36, 0.34 (0.35)	Komatsu, K. and Yabusaki. T. 1993 Kochi[d]
	0.025	-	-	14	Dried Leaves	0.52 , 0.48 (0.50)	0.02, 0.02 (0.02)	0.01, 0.01 (0.01)	
	0.025 0.025 [0.05]	- 7	-	21	Dried Leaves	0.17, 0.16 (0.16)	0.01, 0.01 (0.01)	<0.01, <0.01 (<0.01)	
Kanagawa, Japan 1992 Tea/Yabukita	0.025	-	-	7	Dried Leaves	22.7, 20.1 (21.4)	0.11 , 0.09 (0.10)	0.10, 0.09 (0.10)	Ohyama, J. 1993 Kanagawa[c] mean residue for (c) = 2.39 mg/kg
	0.025	-	-	14	Dried Leaves	2.11 , 2.10 (2.10)	0.03, 0.03 (0.03)	0.01 , 0.01 (0.01)	
	0.025 0.025 [0.05]	- 7	-	21	Dried Leaves	0.37 , 0.36 (0.36)	0.01 , 0.01 (0.01)	<0.01, <0.01 (<0.01)	
Kochi, Japan 1993 Tea/Yabukita	0.025	-	-	7	Dried Leaves	18.9 , 18.3 (18.6)	0.33 , 0.30 (0.32)	0.18, 0.18 (0.18)	Ohyama, J. 1993 Kochi[d] mean residue for (d) = 0.4 mg/kg
	0.025	-	-	14	Dried Leaves	0.30 , 0.29 (0.30)	0.02 , 0.02 (0.02)	<0.01, <0.01 (<0.01)	
	0.025 0.025	- 7	-	21	Dried Leaves	0.12, 0.11 (0.12)	0.02 , 0.02 (0.02)	<0.01, <0.01 (<0.01)	
Mie, Japan 1997 Tea/Yakbukita	0.025	-	3 foliar stage	14	Dried Leaves	0.72 / 0.66 (0.69)	0.03 / 0.03 (0.03)	0.02 / 0.01 (0.02)	Komatsu, K. and Yabusaki. T. 1997 Mie[e]
Kyoto, Japan 1997 Tea/Kyoken No. 129	0.025	-	-	14	Dried Leaves	2.74 / 2.73 (2.74)	0.04 / 0.04 (0.04)	0.01 / 0.01 (0.01)	Komatsu, K. and Yabusaki. T. 1997 Kyoto[f]
Fukuoka, Japan 1997 Tea/Yakbukita	0.025	-	2-2.5 leaves stage	14	Dried Leaves	0.77 / 0.74 (0.76)	0.05 / 0.04 (0.04)	<0.01 / <0.01 (<0.01)	Komatsu, K. and Yabusaki. T. 1997 Fukuoka[g]
Mie, Japan 1997 Tea/Yakbukita	0.025	-	3 foliar stage	14	Dried Leaves	0.59, 0.58 (0.58)	0.04 / 0.04 (0.04)	0.02 / 0.01 (0.02)	Kondo, K. 1997 Mie[e] mean residue for (e) = 0.64 mg/kg
Kyoto, Japan 1997 Tea/Kyoken No. 129	0.025	-	-	14	Dried Leaves	2.54 , 2.32 (2.43)	0.04 / 0.04 (0.04)	0.02 / 0.01 (0.02)	Kondo, K. 1997 Kyoto[f] mean residue for (f) = 2.59 mg/kg

Location, Country Year, Crop/Variety	Rate (kg ai/hL) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	MAPA (mg/kg)	HYPA (mg/kg)	Reference
Fukuoka, Japan 1997 Tea/Yakbukita	0.025	-	2-2.5 leaves stage	14	Dried Leaves	0.58 , 0.56 (0.57)	0.05, 0.03 (0.04)	0.01, 0.01 (0.01)	Kondo, K. 1997 Fukuoka[g] mean residue for (g) = 0.667 mg/kg

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

[a, b, c, d, e, f, g] Trials with the same letter represent the same trial but analysis was conducted on independent samples from the trial sites in two different labs

Animal feeds

Soya bean

The trials submitted for soya bean included residues data for forage and hay. Fifteen residue trials were conducted in the USA and one trial in Canada in 2010.

Two foliar applications were made using an SC formulation at application rates in the range of 0.549–0.717 kg ai/ha.

The last applications were made from full flowering (R2) to Pod formation (R3). Samples of the seed were collected 65–95 days after the last application.

Samples of soya bean seed were immediately frozen and maintained in frozen storage for periods of up to 99 days prior to extraction and analysis.

Residues of fluazinam and AMGT were determined using analytical method 3. Procedural recovery samples were analysed with the residue trial samples. Fortification levels for fluazinam of 0.01–0.1 mg/kg were made with recoveries in the range of 88–108%. For AMGT fortification levels of 0.01 mg/kg–0.1 mg/kg were made with recoveries in the range of 89.5–120 %.

Table 114 Residues in soya bean seeds from supervised trials in Canada and the USA involving 2 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP USA	MID: 0.583 MTD: 1.17	10-14	Early pod formation (R3)	-	-	-		-
Seven Springs, NC, USA 2010 Soya bean/ Asgrow AG5605	0.561 0.561 [1.122]	- 9	Full flowering (R2)	10 20 31 40	Forage	12.439, 10.749 (11.592) 1.803, 1.646 (1.725) 0.599, 0.604 (0.602) 0.861, 0.965 (0.913)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-01

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
				12 24 34 43	Hay	12.669, 17.603 (15.136) 3.759, 5.432 (4.596) 1.578, 2.633 (2.106) 1.240, 0.659 (0.950)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	
Suffolk, VA, USA 2010 Soya bean/ Pioneer 95Y20	0.594 0.717 [1.311]	- 11	Full flowering-Pod formation (R2- R3)	30 32	Forage Hay	2.385, 3.725 (3.055) 3.676, 3.679 (3.678)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-02
Cheneyville, LA, USA 2010 Soya bean, Terral 55R11	0.583 0.594 [1.177]	- 11	Pod formation (late R3)	30 35	Forage Hay	0.753, 1.221 (0.987) 2.020 , 1.636 (1.828)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-03
Proctor, AR, USA 2010 Soya bean, Armor 47G7RR	0.561 0.561 [1.122]	- 10	Beginning bloom (V7 R1)	30 31	Forage Hay	1.080 , 0.990 (1.035) 1.553, 3.107 (2.330)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-04
Northwood, ND, USA 2010 Soya bean/ Pioneer 90Y41	0.561 0.561 [1.122]	- 10	Pod formation (R3)	30 72	Forage Hay	0.637, 0.376 (0.507) 1.241, 0.747 (0.994)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-05
Fisher, MN, USA 2010 Soya bean/ Asgrow AG00901	0.561 0.561 [1.122]	- 9	Pod formation (R3)	30 72	Forage Hay	1.048 , 0.740 (0.894) 0.423 , 0.276 (0.350)	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-06
Geneva, MN, USA	0.561 0.561	- 10	Full flowering (R2)	30	Forage	0.025 , 0.043 (0.034)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
2010 Soya bean/ Pioneer 91Y70	[1.122]			34	Hay	0.019 , 0.027 (0.023)	<0.01, <0.01 (<0.01)	Wiedmann, J.L. 2011 IB-2010-JLW-006-07
Wyoming, IL, USA 2010 Soya bean/ AG 3130	0.583 0.561 [1.144]	- 10	Full flowering-Pod formation (R2- R3)	31	Forage	0.460, 0.233 (0.347)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01
				34	Hay	0.444 , 0.976 (0.710)	<0.01, <0.01 (<0.01)	Wiedmann, J.L. 2011 IB-2010-JLW-006-08
Fitchburg, WI, USA 2010 Soya bean/ S21-N6	0.549 0.561 [1.11]	- 10	Full flowering (R2)	31	Forage	0.460, 0.233 (0.347)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01
				34	Hay	0.444, 0.976 (0.710)	<0.01, <0.01 (<0.01)	Wiedmann, J.L. 2011 IB-2010-JLW-006-09
Lesterville, SD, USA 2010 Soya bean/ Lantharn CS-0991236	0.561 0.561 [1.122]	- 16	Pod formation (R3)	29	Forage	0.447, 0.234 (0.341)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01
				44	Hay	0.546 /, 0.436 (0.491)	<0.01, <0.01 (<0.01)	Wiedmann, J.L. 2011 IB-2010-JLW-006-10
Richland, IA, USA 2010 Soya bean/ Pioneer 92Y80	0.561 0.549 [1.11]	- 9	Full flowering-Pod formation (R2- R3)	29	Forage	0.062 , 0.042 (0.052)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01
				32	Hay	0.468 , 0.021 (0.245)	<0.01, <0.01 (<0.01)	Wiedmann, J.L. 2011 IB-2010-JLW-006-11
Bagley, IA, USA 2010 Soya bean/ 93Y13-N203	0.549 0.561 [1.11]	- 10	Full flowering (R2)	30	Forage	0.113, 0.263 (0.188)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01
				34	Hay	0.271 , 1.171 (0.721)	<0.01, <0.01 (<0.01)	Wiedmann, J.L. 2011 IB-2010-JLW-006-12
Shelbyville, IN, USA 2010 Soya bean/ D4523081	0.561 0.583 [1.144]	- 10	Full flowering-Pod formation (R2- R3)	30	Forage	0.090 , 0.021 (0.056)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01
				31	Hay	0.025 , <0.01 (0.018)	<0.01, <0.01 (<0.01)	Wiedmann, J.L. 2011 IB-2010-JLW-006-13

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
Marysville, OH, USA 2010 Soya bean/SG-329RR	0.561 0.561 [1.122]	- 10	Full flowering-Pod formation (R2- R3)	10	Forage	0.718, 1.574 (1.146)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-14
				20		0.188, 0.325 (0.257)	<0.01, <0.01 (<0.01)	
				30		0.146 / 0.040 (0.093)	<0.01, <0.01 (<0.01)	
				40		0.139 / 0.030 (0.085)	<0.01, <0.01 (<0.01)	
				13	Hay	1.204, 1.485 (1.345)	<0.01, <0.01 (<0.01)	
				23		0.687, 0.526 (0.607)	<0.01, <0.01 (<0.01)	
				33		0.120, 0.214 (0.167)	<0.01, <0.01 (<0.01)	
				43		0.090, 0.070 (0.080)	<0.01, <0.01 (<0.01)	
Leonard, MO, USA 2010 Soya bean/ Asgrow 3803	0.549 0.561 [1.11]	- 11	Full flowering (R2)	30	Forage	0.881 , 0.915 (0.898)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-15
				34	Hay	1.449 , 1.526 (1.488)	<0.01, <0.01 (<0.01)	
Cambridge, ON, Canada 2010 Soya bean/ Absolute RR	0.572 0.549	- 9	Pod formation (R3)	35	Forage	0.734 , 0.716 (0.725)	<0.01, <0.01 (<0.01)	IB-2010-JLW-006-00-01 Wiedmann, J.L. 2011 IB-2010-JLW-006-16
				45	Hay	4.116, 2.604 (3.362)	<0.01, <0.01 (<0.01)	
				67				

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Peanut

Six residue trials were conducted in the USA between 1991 and 1994 which included animal feed items.

In each trial two to three applications were made using an SC formulation at application rates in the range of 0.37– 1.12 kg ai/ha. In some trials applications were made either as a broadcast foliar spray or as a banded foliar application.

Peanut vines were inverted (dug out) and the crop was allowed to dry in the field for 7-10 days before samples were harvested by combine harvester and collected. Samples of whole peanuts were collected 17-59 days after the last treatment, and separated into nutmeat and hulls. Samples of peanut hay were allowed to dry in the field for a further 1- 8 days after harvest before collection 33-58 days after the last treatment.

Samples were immediately frozen and maintained in frozen storage for periods of up to 164 days for hulls and 153 days for hay prior to extraction and analysis.

Residues of fluazinam in peanut hulls and hay were determined using analytical method 1. Procedural recovery samples were analysed with the residue trial samples. Fortification levels of 0.01-1 mg/kg for fluazinam were made for hulls with recoveries in the range of 78–120%. For hay fortification levels of 0.01-15 mg/kg for fluazinam were made with recoveries in the range of 62–125%.

Table 115 Residues in Peanuts from supervised trials in the USA involving 2-3 foliar applications of fluazinam

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
GAP USA	MID: 0.874 MTD: 2.34	21-28	-	30	-	-	-	-
Waller County TX, USA 1993 Peanut/Florunner	0.773 0.758 0.759 [2.365] Broadcast	-- 28 28	-	29	Peanut Hulls	0.10, 0.09 (0.10)	n/a	5879-93-0335-CR-001 Hayes, P.C. Jr. and Kenyon, R.G. 1994
					Peanut Hay	0.29, 0.30 (0.30)	n/a	
	0.619 0.614 0.616 [2.399] Banded	-- 28 28	-	29	Peanut Hulls	0.17, 0.18 (0,18)	n/a	
					Peanut Hay	0.39, 0.14 (0.42)	n/a	
Skippers, VA, USA 1993 Peanut/NC-V11	0.746 0.686 0.763 [2.276] Broadcast	-- 29 31	-	30	Peanut Hulls	0.04, 0.03 (0.04)	n/a	
					Peanut Hay	1.54, 1.46 (1.50)	n/a	
	0.771 0.766 0.752 [2.377] Banded	-- 29 31	-	30	Peanut Hulls	0.05, 0.05 (0.05)	n/a	
					Peanut Hay	1.77, 2.01 (1.89)	n/a	
Shorterville, AL, USA 1993 Peanut/Florunner	0.752 0.761 0.752 [2.343] Broadcast	-- 27 29	Pod fill	58	Peanut Hulls	0.02, 0.02 (0.02)	n/a	
					Peanut Hay	0.20, 0.22 (0.21)	n/a	
	0.372 0.377 0.432 [1.244] Banded	-- 27 29	Pod fill	58	Peanut Hulls	0.01, 0.01 (0.01)	n/a	
					Peanut Hay	0.07, 0.23 (0.16)	n/a	
Lucama, NC USA 1994 Peanut/NC-V11	0.762 0.762 0.773 [2.298] Broadcast	-- 32 31	Excellent growth, rows lapped, 33-43 cm tall	32	Peanut Hulls	0.12, 0.13 (0.13)	n/a	6107-95-0013-CR-001 McFall, D.D. 1995
				33	Peanut Hay	7.08, 7.55 (7.32)	n/a	
	0.773 0.785 0.785 [2.343] Banded	-- 32 31	Excellent growth, rows lapped, 33-46 cm tall	32	Peanut Hulls	0.25, 0.22 (0.24)	n/a	6107-95-0013-CR-001 McFall, D.D. 1995
				33	Peanut Hay	9.69, 10. 7 (10.2)	n/a	
Eakly, OK USA	0.796 0.830	-- 29	Damage from	33	Peanut Hulls	0.14, 0.13 (0.13)	n/a	

Location, Country Year, Crop/Variety	Rate (kg ai/ha) [Total]	Interval (days)	Growth stage at last application	DALA (days)	Crop part	Fluazinam (mg/kg)	AMGT (mg/kg)	Reference
1994 Peanut/Florunner	0.785 [2.42] Broadcast	28	gopher is approx. 15-25% in the plots	38	Peanut Hay	1.35, 0.98 (1.17)	n/a	
	0.796 0.886 0.796 [2.477] Banded	-- 29 28	Damage from gopher is approx. 15-25% in the plots	33	Peanut Hulls	0.17, 0.15 (0.16)	n/a	
				38	Peanut Hay	2.33, 2.28 (2.31)	n/a	
Montezuma, GA, USA 1994 Peanut/GK-7	0.874 0.818 0.796 [2.489] Broadcast	-- 28 25	not noted	41	Peanut Hulls	0.18, 0.18 (0.18)	n/a	
				41	Peanut Hay	0.4`, 0.26 (0.34)	n/a	
	0.863 0.796 0.796 [2.455] Banded	-- 28 25	not noted	41	Peanut Hulls	0.18, 0.21 (0.20)	n/a	
				41	Peanut Hay	0.63, 1.13 (0.88)	n/a	

MID Maximum individual dose

MTD Maximum total dose

Duplicate results represent two independent representative treated samples taken at the trial site with the mean residue level given in brackets

Results in square brackets represent the re-analysis of the same analytical sample

n/a = not analysed

FATE OF RESIDUES IN STORAGE AND PROCESSING

In stored products

Fluazinam is not intended for use in stored products.

In Processing

The meeting received information on high temperature hydrolysis of fluazinam and the fate of fluazinam residues during processing of apples, grapes, soya beans, potatoes and peanuts.

High-temperature hydrolysis

The degradation of ^{14}C-fluazinam was studies under hydrolytic conditions at high temperatures in sterile aqueous buffers at pH 4, 5 and 6 representing pasteurization, baking/brewing/boiling and sterilization.

Fluazinam was labelled in the phenyl or pyridyl ring. Solutions were prepared in duplicate at a nominal concentration of 0.1 mg/L for each test system.

Control samples were analysed immediately at time zero (these samples were not heated). Two additional samples at pH 4 ± 0.1 were placed in an oven and maintained at 90 °C for 20 minutes, another two samples at pH 5 were placed in an oven and maintained at 100 °C for 60 minutes, and two more samples at pH 6 were placed in an autoclave and maintained at sterilizing conditions (120 °C) for 20 minutes.

Radioactive recoveries were determined by LSC. Samples were analysed directly by HPLC. Fluazinam and significant degradation products were identified by LC-MS and by HPLC co-chromatography with certified standards.

The radioactive recovery of all samples was in the range 91.9–100.8% of applied radioactivity (AR). A summary of the results are provided in Tables 116 and 117.

Table 116 Hydrolysis recovery under the conditions for processing simulation, 14C-phenyl labelled fluazinam

Component	Mean % Applied radioactivity (AR)

	pH 4, 90 °C, 20 mins, pasteurization		pH5, 100 °C, 60 mins, Baking/Brewing/Boiling		pH6, 120 °C, 20 mins, Sterilization	
	Heated	Control	Heated	Control	Heated	Control
Fluazinam	89.3	93.68	33.84	95.82	-	98.52
DCPA	-	-	-	-	36.25	-
G-504	-	-	2.13	-	11.17	-
CAPA	0.71	-	55.78	-	44.56	-
Minor degradates (each less than 3.5% of AR)	2.52	-	3.88	2.13	5.63	2.04
Total recovery	92.52	95.75	95.63	97.94	97.61	100.56

Table 117 Hydrolysis recovery under the conditions for processing simulation, 14C-pyridinyl labelled fluazinam

Component	Mean % Applied radioactivity (AR)					
	pH 4, 90 °C, 20 mins, pasteurization		pH5, 100 °C, 60 mins, Baking/Brewing/Boiling		pH6, 120 °C, 20 mins, Sterilization	
	Heated	Control	Heated	Control	Heated	Control
Fluazinam	93.20	95.66	39.37	99.16	-	96.96
DCPA	-	-	-	-	37.15	-
G-504	-	-	1.56	-	11.01	-
CAPA	1.09	-	50.89	-	42.94	0.14
Minor degradates (each less than 3.5% of AR)	1.40	1.29	2.81	1.03	3.53	1.40
Total recovery	95.68	96.95	94.64	100.19	94.72	98.50

Fluazinam was stable under ambient conditions at all pH values tested. It was also stable under conditions simulating pasteurisation with only a few minor degradates being detected (each less than 2% AR).

Fluazinam, however, degraded rapidly under conditions simulating baking/brewing/boiling forming a single major metabolite, CAPA, plus several other minor components (including small amounts of G-504 in the phenyl label).

Fluazinam degraded completely under the conditions simulating sterilisation, forming three major components, CAPA, DCPA and G-504, plus several other minor components. The minor components detected all accounted for ≤ 3.5% AR individually.

In conclusion, fluazinam was stable under conditions representing pasteurization. However, significant degradation was observed under conditions representing baking/brewing/boiling and sterilization (see below):

In processing-effect on the residue level

The meeting received information on the effects of processing on the magnitude of fluazinam and AMGT residue levels for apple, grape, soya beans, potato and peanuts.

Apple

A processing study with apples was conducted 1993 in the USA. Apples were subjected to the following processing procedures:

Raw juice, wet and dry pomace

Apples were grounded in a Hammer-mill and the mash was loaded into cloth stacks on a hydraulic press and pressed for five minutes. Juice was collected and the cloths were opened to collect wet pomace. Dry pomace was obtained by drying wet pomace in a dryer at 77–88 °C over 1–4 hours until the moisture content was <10%.

Pasteurised apple juice

Raw juice was heated to 49 °C and clarified using pectinase. The cleared juice was filtrated using diatomaceous earth and pasteurised at 88 °C.

Samples were stored frozen for up to 231 days prior to analysis for fluazinam and up to 802 days for AMGT. Fluazinam and AMGT were determined in the RAC and processed fractions using analytical method 1. The procedural recoveries were all acceptable. The results are summarised in tables 118 and 119 for fluazinam and AMGT respectively.

Table 118 Summary of the processing data for apples for fluazinam

Commodity	Fluazinam residue (mg/kg)	Processing factor
Apple	0.03	-
Raw apple juice	<0.01	<0.33
Pasteurised apple juice	<0.01	<0.33
Wet apple pomace	0.07	2.33
Dry apple pomace	0.09	3.0

Table 119 Summary of the processing data for apples for AMGT

Commodity	AMGT residue (mg/kg)	Processing factor
Apple	<0.01	-
Raw apple juice	<0.01	1
Pasteurised apple juice	<.0.01	1
Wet apple pomace	<0.01	1
Dry apple pomace	0.01	1

Grapes

Study 1

Grapes were subjected to the following processing procedures:

Juice, wet and dry pomace

Grapes were manually stemmed and crushed. Pectinase was added to the crushed grapes and heated to 60 °C for 2 hours. The crushed grapes were pressed to obtain juice and wet pomace. Wet pomace was dried at 60-63 °C in a forced air dryer to obtain dry pomace. The juice was heated to 85-88 °C and clarified at -1 to 0 °C for 4 to 6 weeks. The juice was filtered using diatomaceous earth, heated to 91-93 °C and canned.

Raisins

Grapes for sun drying were spread on a tray covered with aluminium foil and placed in a sunny area. Grapes were dried for 14-25 days to a moisture content of 12-14%. Dried grapes were separated from the stems. Dried grapes were washed and rehydrated to 18-20% moisture to obtain raisins.

Samples were stored frozen for up to 181 days prior to analysis. Fluazinam was determined in the RAC and processed fractions using analytical method 1. The procedural recoveries were all acceptable. The results are summarised in Table 120.

Table 120 Summary of the processing data for grapes from study 1

Commodity	Fluazinam residue (mg/kg)	Processing factor
Grape	1.37	-
Wet pomace	9.43	6.9
Dry pomace	17.5	12.8
Juice	<0.01	<0.01
Raisin waste	4.24	3.1
Raisins	0.34	0.25

Study 2

Grapes from two trials were subjected to the following processing procedures:

Juice, wet and dry pomace

Grapes were stemmed and crushed. The crushed grapes were pressed to obtain juice and wet pomace. Wet pomace was blended with the stems from the de-stemming process. Wet pomace was dried at 79-93 °C in a forced air dryer to obtain dry pomace. The juice was filtered and bottled.

Raisins

Grapes were placed on paper trays and sun dried for approx. 27 days. The raisins were sieved to remove loose dirt, and field debris. Stems were removed from the field dried raisins. Raisins were batch spray washed with cold water for 10-15 seconds and allowed to dry to obtain finished raisins.

Samples were stored frozen for up to 273 days prior to analysis. Fluazinam and AMGT were determined in the RAC and processed fractions using analytical method 1. The procedural recoveries were all acceptable. The results are summarised in Tables 121 and 122 for fluazinam and AMGT respectively.

Table 121 Summary of the processing data for grapes for fluazinam from study 2

Commodity	Fluazinam residue–trial 1 (mg/kg)	Processing factor–trial 1	Fluazinam residue–trial 2 (mg/kg)	Processing factor–trial 2
Grape	0.08	-	0.08	-
Raisins	0.02	0.25	-	-
Raisins waste	0.36	4.5	-	-
Juice	-	-	0.02	0.25
Wet pomace	-	-	0.41	5.13
Dry pomace	-	-	0.50	6.25

Table 122 Summary of the processing data for grapes for AMGT from study 2

Commodity	AMGT residue–trial 1 (mg/kg)	Processing factor–trial 1	AMGT residue–trial 2 (mg/kg)	Processing factor–trial 2
Grape	0.27	-	0.08	-
Raisins	0.32	1.19	-	-
Raisins waste	0.43	1.59	-	-
Juice	-	-	0.02	0.25
Wet pomace	-	-	0.21	2.62
Dry pomace	-	-	0.33	4.13

Study 3

Four processing studies using grapes from two trials were subjected to the processing procedures outlined below. In two of the cases the residue levels in grapes prior to processing was not determined.

White wine and must

Grapes were pressed with a manual hydraulic press. Potassium metabisulphite and pectolytic enzymes were added to the juice and decanted after 12 hours. Yeast was added to start alcoholic fermentation. White crystalline sugar was added to the must to increase the alcoholic content by 2%. When alcoholic fermentation was achieved, potassium metabisulphite was added and the wine clarified using dry gelatine for 15 days at 5-10 °C. After clarification, the wine was filtered, potassium metabisulphite added and bottled.

Red wine and must

Grapes were crushed, potassium metabisulphite and yeast were added to start alcoholic fermentation. The solid parts were pressed using a manual hydraulic press. Malolacetic fermentation was started by inoculation with lactic bacteria. When the malolacetic fermentation was complete, potassium metabisuphite was added and the wine clarified using dry gelatine for at least 15 days. After clarification, potassium metabisulphite and metatartaric acid were added, the wine was filtered and bottled.

Samples were stored frozen for up to 201 days prior to analysis. Fluazinam and AMGT were determined in the RAC and processed fractions using analytical method 1. The procedural recoveries were all acceptable. The results are summarised in Tables 123 and 124 for fluazinam and AMGT respectively.

Table 123 Summary of the processing data for grapes for fluazinam from study 3

White wine

Commodity	Fluazinam residue–trial 1 (mg/kg)	Processing factor–trial 1	Fluazinam residue–trial 2 (mg/kg)	Processing factor–trial 2
Grape	0.22	-	-	-
Must	0.09	0.41	0.44	-
Wine	<0.01	<0.05	<0.01	-

Red wine

Commodity	Fluazinam residue–trial 1 (mg/kg)	Processing factor–trial 1	Fluazinam residue–trial 2 (mg/kg)	Processing factor–trial 2
Grape	0.61	-	-	-
Must	0.04	0.07	0.27	-
Wine	<0.01	<0.02	<0.01	-

Table 124 Summary of the processing data for grapes for AMGT from study 3

White wine

Commodity	AMGT residue–trial 1 (mg/kg)	Processing factor–trial 1	AMGT residue–trial 2 (mg/kg)	Processing factor–trial 2
Grape	0.21	-	-	-
Must	0.13	0.62	0.23	-
Wine	0.18	0.86	0.35	-

Red wine

Commodity	AMGT residue–trial 1 (mg/kg)	Processing factor–trial 1	AMGT residue–trial 2 (mg/kg)	Processing factor–trial 2
Grape	0.17	-	-	-
Must	0.12	0.71	0.25	-
Wine	0.03	0.18	0.06	-

Study 4

Grapes from four trials were processed into wine as follows:

Wine processing

Grapes were pressed, sulphited and allowed to settle for 2-16 hours. Sugar and yeast were added to start alcoholic fermentation. After alcoholic fermentation, wine was decanted and malolacetic fermentation started by adding *Inobacter*. After malolacetic fermentation, wine was decanted, sulphited and bottled.

Samples were stored frozen for up to 228 days prior to analysis. Fluazinam and AMGT were determined in the RAC and processed fractions using analytical method 1. The procedural recoveries were all acceptable. The results are summarised in Tables 125 and 126 for fluazinam and AMGT respectively.

Table 125 Summary of the processing data for grapes for fluazinam from study 4

Trial	Sample	Commodity	Fluazinam residue (mg/kg)	Processing factor	Mean processing factor for each trial
1	1	Grape	0.01	<1	0.39
		Wine	<0.01		
	2	Grape	0.02	<0.5	
		Wine	<0.01		
	3	Grape	0.22	<0.05	
		Wine	<0.01		
	4	Grape	1.10	<0.01	
		Wine	<0.01		
2	1	Grape	<0.01	<1	0.55
		Wine	<0.01		
	2	Grape	0.01	<1	
		Wine	<0.01		
	3	Grape	0.06	<0.17	
		Wine	<0.01		
	4	Grape	0.41	<0.024	

Trial	Sample	Commodity	Fluazinam residue (mg/kg)	Processing factor	Mean processing factor for each trial
		Wine	<0.01		
3	1	Grape	<0.01	<1	0.38
		Wine	<0.01		
	2	Grape	0.10	<0.1	
		Wine	<0.01		
	3	Grape	0.34	<0.029	
		Wine	<0.01		
	4	Grape	2.41	-	
		Wine	-		
4	1	Grape	<0.01	<1	0.39
		Wine	<0.01		
	2	Grape	0.07	<0.14	
		Wine	<0.01		
	3	Grape	0.40	<0.025	
		Wine	<0.01		
	4	Grape	1.21	-	
		Wine	-		

Table 126 Summary of the processing data for grapes for AMGT from study 4

Trial	Sample	Commodity	AMGT residue (mg/kg)	Processing factor
1	1	Grape	0.06	<0.17
		Wine	<0.01	
	2	Grape	0.08	0.25
		Wine	0.02	
	3	Grape	0.11	0.18
		Wine	0.02	
	4	Grape	0.11	0.18
		Wine	0.02	
2	1	Grape	0.04	1
		Wine	0.04	
	2	Grape	0.11	0.82
		Wine	0.09	
	3	Grape	0.14	0.93
		Wine	0.13	
	4	Grape	0.13	0.92
		Wine	0.12	
3	1	Grape	0.05	1
		Wine	0.05	
	2	Grape	0.13	1.46
		Wine	0.19	
	3	Grape	0.22	1.16
		Wine	0.19	
	4	Grape	0.25	-
		Wine	-	
4	1	Grape	0.03	0.33
		Wine	0.01	
	2	Grape	0.10	0.008
		Wine	0.08	
	3	Grape	0.26	0.69
		Wine	0.18	
	4	Grape	0.17	-
		Wine	-	

Study 5

Red wine

Grapes from two trials were processed into wine as follows:

Grapes were crushed and stemmed. Potassium metabisulphite and yeast were added to start alcoholic fermentation. White crystallised sugar was added to increase the alcohol content to 11.5%. Alcoholic fermentation was considered complete when the density of the must fell below the value of 1000 (using a mustimeter). The solid parts were pressed using a manual hydraulic press. Malolacetic fermentation was started by inoculation with lactic bacteria. When the malolacetic fermentation was complete, potassium metabisuphite was added and the wine clarified using dry gelatine for at least 15 days. After clarification, the wine was filtered, potassium metabisulphite was added and the wine was bottled.

Samples were stored frozen for up to 246 days prior to analysis. Fluazinam and AMGT were determined in the RAC and processed fractions using analytical method 1. The procedural recoveries were all acceptable. The results are summarised in Tables 127 and 128 for fluazinam and AMGT respectively.

Table 127 Summary of the processing data for grapes for fluazinam from study 5

Trial	Commodity	Fluazinam residue (mg/kg)	Processing factor
1	Grape	0.03	<0.33
	Wine	<0.01	
2	Grape	0.02	<0.5
	Wine	<0.01	

Table 128 Summary of the processing data for grapes for AMGT from study 5

Trial	Commodity	AMGT residue (mg/kg)	Processing factor
1	Grape	-	-
	Wine	0.03	
2	Grape	-	-
	Wine	0.05	

Soya bean

Soya bean samples were processed as outlined below:

Grain dust (aspirated grain fraction)

Whole soya beans were used for aspirated grain fraction generation. After moisture determination, soya beans were dried in an oven at 43-57 °C until the moisture content was between 10 and 13%. To generate aspirated grain fraction, samples were placed in a dust generation room containing a holding bin, two bucket conveyors and a screw conveyor. The samples travelled the system for 120 minutes and aspiration removed the light impurities (grain dust).

Hulls

After moisture determination, soya beans were dried in an oven at 54-71°C until the moisture content was below 13.5%. Samples for processing were cleaned by aspiration and screening. Light impurities were separated from the sample using an aspirator. After aspiration, the sample was screened to separate large and small foreign particles from the whole soya bean sample. Cleaned whole soya beans were fed into an a roller mill to crack the hull and liberate the kernel. After hulling, the material was passed through an aspirator to separate hull and kernel material.

Meal

Moisture adjusted kernel material (13.5%) was heated to 71-79 °C and processed into flakes. Flakes were expanded in a continuous processor, where they were turned into collets by direct steam injection and compression. Collets exited the processor at 93-121°C. After expansion, the collets were dried in the oven at 66-82 °C for 30-40 minutes. Dried collets were placed in stainless steel batch extractors and submerged in 49-60 °C warm hexane. After 30 minutes, the miscella (crude oil and hexane) was drained and fresh hexane was added to repeat the cycle two more times. Extracted collets were de-solvented by heating to 99-104 °C to give the final soya bean meal.

Refined oil

Miscella were passed through a laboratory vacuum evaporator unit to separate the crude oil and hexane. Crude oil was heated to 91-96 °C to remove hexane and then filtered. The percentage of free fatty acid (FFA) content was determined for the crude oil. Based on the FFA content, a weighed amount of crude oil and sodium hydroxide was placed in a water bath at 20-24 °C and mixed for 90 minutes at high RPM, and then for 20 minutes at low RPM at 63-67 °C. The neutralised oil was then centrifuged. Refined oil was decanted and filtered. The resulting fractions were alkali refined oil and soapstock. Soapstock was discarded.

Samples were stored frozen for up to 104 days prior to analysis. Fluazinam and AMGT were determined in the RAC and processed fractions using analytical method 3. The procedural recoveries were all acceptable. The results are summarised in Table 129.

Table 129 Summary of the processing data for soya bean

Commodity	Fluazinam residue (mg/kg)	Processing factor
Soya bean	0.048	-
Grain dust	<0.01	<0.21
Hulls	0.231	4.81
Meal	<0.01	<0.21
Oil	0.046	0.96

Potato

Potato samples from two trials were processed as outlined below:

Potato chips

Potatoes were washed for 5-10 minutes, culls removed and peeled using an continuous abrasive peeler. Potatoes were inspected and trimmed by hand to remove rot, green or otherwise damaged potatoes. Potatoes were cut into thin slices (1.6 mm). Slices were placed into warm water to remove free starch and heated in a deep fat fryer in hot oil at 163-177 °C for 60-90 seconds. Oil was drained in draining tray and potato chips salted by hand.

Potato flake and granules, wet and dry peel

Potatoes were tub washed and steam peeled and scrubbed to remove loosened potato peel. Potatoes were inspected and trimmed by hand to remove rot, green or otherwise damaged potatoes. Wet peel was collected and dried.

For potato flakes, peeled potatoes were cut into 1.3 cm slabs. Slabs were spray washed with cold water to remove free starch and pre-cooked at 71-74 °C for 20 minutes in a steam kettle and cooled. The pre-cooked slabs were steam cooked at 99-100 °C for 45 minutes, mashed and mixed in a Hobart Mixer with pre-weighed food-additives.

The wet mash was dried. The resulting thin potato sheet was hand broken to large flakes and processed in a Hammer-mill to potato flakes.

For potato granules, the pre-cooked slabs were steam cooked at 99-100 °C for 45 minutes, mashed and mixed in a Hobart Mixer with pre-weighed food-additives. The mash was packaged into plastic bags and frozen for later dehydration. The potato mash bags were thawed to give the potato granules.

French fries

Potatoes were tub washed and steam peeled. Potatoes were scrubbed to remove loosened peel. Potatoes were inspected and trimmed by hand to remove rot, green or otherwise damaged potatoes. The peeled potatoes were cut into 0.64x0.64 cm strips using a French Fry Cutter. French fry strips were blanched at 71-74 °C for 10 minutes and again blanched at 88-91°C for 3 minutes. The French fry strips were placed in an air dryer at 71°C for 18 minutes. The French fries were then fried at 177-191°C for 60 seconds, drained and air cooled.

Samples were stored frozen for up to 406 days prior to analysis. Fluazinam was determined in the RAC and processed fractions using analytical method 1. The procedural recoveries were all acceptable. The results are summarised in Table 130.

Table 130 Summary of the processing data for potato

Commodity	Fluazinam residue–trial 1 (mg/kg)	Processing factor	Fluazinam residue–trial 2 (mg/kg)	Processing factor
Potato tubers	<0.01	-	<0.01	-
Potato chips	<0.01	-	<0.01	-
Wet potato peels	<0.01	-	<0.01	-
Dry potato peels	<0.01	-	<0.01	-
Potato flakes	<0.01	-	<0.01	-
French fries	<0.01	-	<0.01	-
Potato granules	<0.01	-	<0.01	-

Peanut

Potato samples from one trial were processed as outlined below:

Hulling and separation

Peanuts were cleaned by removing rocks and soil from the sample. The cleaned samples was fed though a peanut sheller to liberate the kernels /nutmeat from the hulls. After shelling, hull material was separated from kernels using an aspiration unit. After hull and kernel separation, the moisture content of the kernels was determined and if necessary, samples dried in an air oven at 61-71°C to a final moisture content of 7-10%.

Peanut oil

The moisture content of the kernels was adjusted to 12%. The kernels were heated to 94-104 °C and then fed though an expeller to mechanically remove a majority of the oil and this gave rise to the press cake and crude oil. The press cake was flaked and residual oil extracted with warm hexane in a batch extractor for 30 minutes. The solvent was drained and the extraction repeated two times without heating. After draining, warm air was forced through the press cake to remove the hexane. The resulting fractions from the solvent extraction step were meal and miscella (crude oil and hexane).

The miscella was separated using an evaporator at 75-85 °C. The free fatty acid content was determined in the crude oil and based on this, NaOH was added to the crude oil. The solution was mixed for 30 minutes at 20-24 °C and for 12 minutes at 63-67 °C. The neutralised oil was allowed to settle at 60-65 °C for one hour. The oil solution was refrigerated for a minimum of 12 hours, decanted and filtered and collected as refined oil. The fraction settling to the bottom was collected as soapstock.

Samples were stored frozen for up to 93 days prior to analysis. Fluazinam was determined in the RAC and processed fractions using analytical method 1. The procedural recoveries were all acceptable. The results are summarised in Table 131.

Table 131 Summary of the processing data for peanut

Commodity	Fluazinam residue (mg/kg)	Processing factor
Peanut	<0.01	-
Hulls	0.36	36
Presscake	<0.01	<1
Crude oil	0.03	3
Refined oil	0.01	1
Soapstock	0.05	5

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Lactating cow

Three groups, each comprising three or six lactating females received fluazinam orally once daily at nominal dose levels equivalent to 2.5, 7.5 and 25 mg/kg dry matter feed for 28 days using gelatine capsules and a balling gun. A similarly constituted control group received placebos (empty capsules) concurrently with the treated group. Three animals were maintained in the high dose group and maintained for up to seven days after the cessation of treatment in order to provide data on the decline of any incurred residues.

Animals were inspected visually at least twice daily for clinical abnormalities from receipt until the scheduled termination for each animal. If daily observations revealed any health-related issues, these animals were re-examined by a licensed veterinarian. Animals were otherwise noted as normal and active. Individual body weights were obtained upon receipt and weekly during the

dosing (Study days 1, 8, 15, 22, and 28). The consumption of individual feed rations was recorded daily from acclimation to termination. Milk yields were recorded daily from acclimation to termination.

Milk was collected twice daily (am and pm) via milking machines from acclimation to termination. Separate milking machines were used for the study groups. Milk from study day 13 and 28 was separated into cream and skim milk from one control, three low dose and three high dose group animals that were not designated for use in the depuration phase.

After 28 days the animals (parts from those in the depuration study) were sacrificed within 24 hours of the last dose. samples of liver, kidney, muscle and fat were collected for analysis. Each tissue was weighed and cubed, except fat, which was allowed to freeze prior to cubing and separated into analytical and retention sample. The analytical sample was homogenised in the presence of dry ice.

All samples were then stored at -24 to -14 °C prior to analysis. The maximum lengths of storage were:

Milk: 183 days

Muscle: 157 days

Fat: 203 days

Liver: 248 days

Kidney: 255 days

Animals from the depuration phase were terminated on study day 30, 32 and 36 and samples were collected as above.

Samples of whole milk, cream, skimmed milk and tissues (muscle, fat, liver, and kidney) were analysed to determine the residues of fluazinam, AMPA and DAPA. Residues were determined using method IB-2007-JLW-004-00-01. The method used for milk included a hydrolysis step to extract any sulfamate conjugates that may be present. For kidney and liver samples two sets of analysis were undertaken; extraction with acetonitrile: water, and extraction with acetonitrile: water followed by a hydrolysis step with HCl.

Procedural recoveries were analysed with the samples. The fortification levels used were 0.01 mg/kg and 0.1 mg/kg. Only the mean procedural recoveries were reported. The mean recoveries were > 70% except for:

Analyte	Matrix	Method	Mean procedural recovery (%)
Fluazinam	Liver	Non hydrolysis	57
AMPA	Liver	Non hydrolysis	59
	kidney	Non hydrolysis	60
		Hydrolysis	41
DAPA	Liver	Non hydrolysis	31
		Hydrolysis	38
	Kidney	Non hydrolysis	62
		Hydrolysis	12

The mean weekly intakes of fluazinam for the different dose groups are given in Table 132.

Table 132 Summary of fluazinam dose administration to lactating cows

Group	Target dose [mg/kg dry feed]	Dosing week				Average dose [mg/kg dry feed]	Average dose [mg/kg bw]
		1	2	3	4		
		[mg/kg dry feed]					
Low	2.5	2.92	2.91	2.90	2.90	2.91	0.67
Mid	7.5	8.73	8.72	8.72	8.72	8.72	2.10
High	25	28.74	28.64	29.00	28.99	28.84	6.40

The average dose of fluazinam administered over the four week study period was 2.91, 8.72 and 28.84 mg/kg dry weight for the low, mid and high dose group, respectively. When related to the body weight, the achieved fluazinam intakes were 0.67, 2.10 and 6.40 mg/kg bw per day for the low, mid and high dose group, respectively.

All animals were observed to be healthy and normal throughout the study. No treatment related effects were observed. Body weights were considered normal throughout the study for animals of this species and age. Milk production appeared to be consistent throughout the study and did not appear to be affected by treatment with the test substance.

Following termination, tissues were observed for gross lesions. In one animal (cow #1, control group), pale discoloration of the liver and abscesses in the right front leg and left rear upper leg as a result of a stanchion injury from test day 14 was observed. Gross observations of all other animals revealed no findings.

Total residues of fluazinam, AMPA and DAPA in milk are shown in Table 133 to Table 137.

Since residues in milk of the mid dose group were below the limit of quantification (0.01 mg/kg), the milk samples from the low dose group were not analysed.

Table 133 Residues of fluazinam in milk

Dose level	Animal number	Day of treatment														
		0	1	2	3	4	5	6	7	10	13	16	19	22	25	28
		[mg/kg]														
Mid dose group	6	<0.01	-	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	<0.01
	7	<0.01	-	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	<0.01
	8	<0.01	-	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	<0.01
	Mean	<0.01	-	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	<0.01
High dose group	9	<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
	10	<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
	11	<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
	12	<0.01	<0.01	<0.01	<0.01	-	-	-	-	-	-	-	-	-	<0.01	<0.01
	13	<0.01	<0.01	<0.01	<0.01	-	-	-	-	-	-	-	-	-	<0.01	<0.01
	14	<0.01	<0.01	<0.01	<0.01	-	-	-	-	-	-	-	-	-	<0.01	<0.01
	Mean	<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

Table 134 Residues of AMPA in milk

Dose level	Animal number	Day of treatment														
		0	1	2	3	4	5	6	7	10	13	16	19	22	25	28
		[mg/kg]														
Mid dose group	6	<0.01	-	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	<0.01
	7	<0.01	-	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	<0.01
	8	<0.01	-	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	<0.01
	Mean	<0.01	-	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	<0.01
High dose group	9	<0.01	<0.01	<0.01	0.0112	0.0125	0.0158	0.0126	0.0133	0.0133	0.0114	0.0120	0.0121	0.0151	0.0159	0.0147
	10	<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
	11	<0.01	<0.01	<0.01	<0.01	0.0107	0.0115	<0.01	0.0110	<0.01	<0.01	<0.01	<0.01	0.0103	0.0137	<0.01
	12	<0.01	<0.01	<0.01	<0.01	-	-	-	-	-	-	-	-	-	<0.01	<0.01
	13	<0.01	<0.01	<0.01	<0.01	-	-	-	-	-	-	-	-	-	<0.01	<0.01
	14	<0.01	<0.01	<0.01	<0.01	-	-	-	-	-	-	-	-	-	<0.01	<0.01
	Mean	<0.01	<0.01	<0.01	<0.01	<0.01	0.0107	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.0101	<0.01	<0.01

Table 135 Residues of DAPA in milk

Dose level	Animal number	Day of treatment														
		0	1	2	3	4	5	6	7	10	13	16	19	22	25	28
		[mg/kg]														
Mid dose group	6	<0.01	-	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	<0.01
	7	<0.01	-	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	<0.01
	8	<0.01	-	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	<0.01
	Mean	<0.01	-	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	-	<0.01	<0.01
High dose group	9	<0.01	<0.01	0.0155	0.0208	0.0239	0.0266	0.0195	0.0210	0.0202	0.0150	0.0174	0.0179	0.0216	0.0229	0.0227
	10	<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
	11	<0.01	<0.01	0.0154	0.0205	0.0203	0.0186	0.0229	0.0283	0.0184	0.0112	0.0162	0.0141	0.0226	0.0331	0.0160
	12	<0.01	<0.01	<0.01	<0.01	-	-	-	-	-	-	-	-	-	0.0131	<0.01
	13	<0.01	<0.01	<0.01	0.0134	-	-	-	-	-	-	-	-	-	<0.01	<0.01
	14	<0.01	<0.01	<0.01	<0.01	-	-	-	-	-	-	-	-	-	0.0167	<0.01
	Mean	<0.01	<0.01	0.0104	0.0143	0.0164	0.0170	0.0157	0.0178	0.0146	0.0100	0.0129	0.0120	0.0163	0.0163	0.0119

Table 136 Residues of fluazinam, AMPA and DAPA in milk from the depuration phase

Dose level	Animal number	Study day								
		29	30	31	29	30	31	29	30	31
		IKF-1216 [mg/kg]			AMPA [mg/kg]			DAPA [mg/kg]		
High dose group	12	<0.01	-	-	<0.01	-	-	<0.01	-	-
	13	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
	14	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Table 137 Distribution of residues of fluazinam, AMPA and DAPA in skim milk and cream

Dose level	Animal number	Day	Fluazinam [mg/kg]	AMPA [mg/kg]	DAPA [mg/kg]
Whole milk					
High dose group	9	13	<0.01	0.0114	0.0150
	10	13	<0.01	<0.01	<0.01
	11	13	<0.01	<0.01	0.0112
	Mean		<0.01	<0.01	0.0101
Skim milk					
High dose group	9	13	<0.01	<0.01	<0.01
	10	13	<0.01	<0.01	<0.01
	11	13	<0.01	<0.01	<0.01
	Mean		<0.01	<0.01	<0.01
Cream					
High dose group	9	13	<0.01	0.0565	0.0860
	10	13	<0.01	0.0170	0.0338
	11	13	<0.01	0.0342	0.0765
	Mean			<0.01	0.0654
Whole milk					
High dose group	9	28	<0.01	0.0147	0.0227
	10	28	<0.01	<0.01	<0.01
	11	28	<0.01	<0.01	0.0160
	Mean		<0.01	<0.01	0.0145
Skim milk					
High dose group	9	28	<0.01	<0.01	<0.01
	10	28	<0.01	<0.01	<0.01
	11	28	<0.01	<0.01	<0.01
	Mean		<0.01	<0.01	<0.01
Cream					
High dose group	9	28	<0.01	0.0582	0.1200
	10	28	<0.01	0.0215	0.0324
	11	28	<0.01	0.0348	0.1000

Dose level	Animal number	Day	Fluazinam [mg/kg]	AMPA [mg/kg]	DAPA [mg/kg]
	Mean		<0.01	<0.01	0.0841

Total residues of fluazinam, AMPA and DAPA in tissues are shown in Tables 138 to 141.

Since mean residues in muscle of the high dose group were below the limit of quantification (0.01 mg/kg), the muscle samples from the mid and low dose group were not analysed.

Table 138 Distribution of residues of fluazinam, AMPA and DAPA in muscle

Dose level	Animal number	Matrix	Fluazinam [mg/kg]	AMPA [mg/kg]	DAPA [mg/kg]
High dose group	9	Loin muscle	<0.01	0.0101	0.0179
	10	Loin muscle	<0.01	<0.01	<0.01
	11	Loin muscle	<0.01	<0.01	<0.01
	9	Round muscle	<0.01	<0.01	<0.01
	10	Round muscle	<0.01	<0.01	<0.01
	11	Round muscle	<0.01	<0.01	<0.01
	Mean		<0.01	<0.01	<0.01
Depuration phase					
High dose group-depuration phase	12	Loin muscle	<0.01	<0.01	<0.01
	12	Round muscle	<0.01	<0.01	<0.01
	13	Loin muscle	<0.01	<0.01	<0.01
	13	Round muscle	<0.01	<0.01	<0.01
	14	Loin muscle	<0.01	<0.01	<0.01
	14	Round muscle	<0.01	<0.01	<0.01

Table 139 Distribution of residues of fluazinam, AMPA and DAPA in fat

Dose level	Animal number	Matrix	Fluazinam [mg/kg]	AMPA [mg/kg]	DAPA [mg/kg]
Low dose group	3	Abdominal fat	<0.01	<0.01	0.0113
		Perirenal fat	<0.01	<0.01	<0.01
		Subcutaneous fat	<0.01	<0.01	<0.01
	4	Abdominal fat	<0.01	<0.01	<0.01
		Perirenal fat	<0.01	<0.01	<0.01
		Subcutaneous fat	<0.01	<0.01	<0.01
	5	Abdominal fat	<0.01	0.0169	0.0219
		Perirenal fat	<0.01	0.0145	0.0197
		Subcutaneous fat	<0.01	<0.01	0.0107
	Mean	Abdominal fat	<0.01	0.0111	0.0132
	Mean	Perirenal fat	<0.01	<0.01	0.0111
	Mean	Subcutaneous fat	<0.01	<0.01	<0.01
Mid dose group	6	Abdominal fat	<0.01	0.0173	0.0253
		Perirenal fat	<0.01	0.0152	0.0195
		Subcutaneous fat	<0.01	<0.01	<0.01
	7	Abdominal fat	<0.01	<0.01	<0.01
		Perirenal fat	<0.01	0.0331	0.0432
		Subcutaneous fat	<0.01	0.0210	0.0273
	8	Abdominal fat	<0.01	0.0201	0.0223
		Perirenal fat	<0.01	0.0218	0.0237
		Subcutaneous fat	<0.01	0.0146	0.0152
	Mean	Abdominal fat	<0.01	0.0152	0.0179
	Mean	Perirenal fat	<0.01	0.0234	0.0288
	Mean	Subcutaneous fat	<0.01	0.0140	0.0174
High dose group	9	Abdominal fat	<0.01	0.1439	0.2437
		Perirenal fat	<0.01	0.1341	0.1853
		Subcutaneous fat	<0.01	0.1100	0.1757
	10	Abdominal fat	<0.01	0.0624	0.0468
		Perirenal fat	<0.01	0.0675	0.0473

Dose level	Animal number	Matrix	Fluazinam [mg/kg]	AMPA [mg/kg]	DAPA [mg/kg]
		Subcutaneous fat	<0.01	0.0476	0.0378
	11	Abdominal fat	<0.01	0.1182	0.2875
		Perirenal fat	<0.01	0.1035	0.2348
		Subcutaneous fat	<0.01	0.0618	0.1295
	Mean	Abdominal fat	<0.01	0.1082	0.1927
	Mean	Perirenal fat	<0.01	0.1017	0.1558
	Mean	Subcutaneous fat	<0.01	0.0731	0.1143
Depuration phase					
High dose group-depuration, study day 29	12	Abdominal fat	<0.01	0.0575	0.0891
		Perirenal fat	<0.01	0.0398	0.0697
		Subcutaneous fat	<0.01	0.0176	0.0277
		Mean	<0.01	0.0383	0.1865
High dose group-depuration, study day 31	13	Abdominal fat	<0.01	<0.01	<0.01
		Perirenal fat	<0.01	<0.01	<0.01
		Subcutaneous fat	<0.01	<0.01	<0.01
		Mean	<0.01	<0.01	<0.01
High dose group-depuration, study day 35	14	Abdominal fat	<0.01	0.0141	0.0293
		Perirenal fat	<0.01	<0.01	0.0107
		Subcutaneous fat	<0.01	0.0121	0.0293
		Mean	<0.01	0.0121	0.0693

Table 140 Distribution of residues of fluazinam, AMPA and DAPA in liver

Dose level	Animal number	Matrix	Fluazinam [mg/kg][a]	AMPA [mg/kg][a]	DAPA [mg/kg][b]
Low dose group	3	Liver	<0.01	<0.01	<0.01
	4	Liver	<0.01	<0.01	<0.01
	5	Liver	<0.01	<0.01	<0.01
	Mean		<0.01	<0.01	<0.01
Mid dose group	6	Liver	<0.01	<0.01	<0.01
	7	Liver	<0.01	<0.01	0.0222
	8	Liver	<0.01	<0.01	<0.01
	Mean		<0.01	<0.01	0.0136
High dose group	9	Liver	<0.01	0.0110	0.0220
	10	Liver	<0.01	<0.01	0.0130
	11	Liver	<0.01	0.0140	0.0310
	Mean		<0.01	<0.01	0.0220
Depuration phase					
High dose group-depuration, study day 29	12	Liver	<0.01	<0.01	<0.01
High dose group-depuration, study day 31	13	Liver	<0.01	<0.01	<0.01
High dose group-depuration, study day 35	14	Liver	<0.01	<0.01	<0.01

[a] Hydrolysis procedure

[b] Non-hydrolysis procedure

Table 141 Distribution of residues of fluazinam, AMPA and DAPA in kidney

Dose level	Animal number	Matrix	Fluazinam [mg/kg][a]	AMPA [mg/kg][a]	DAPA [mg/kg][b]
Low dose group	3	Kidney	<0.01	<0.01	<0.01
	4	Kidney	<0.01	<0.01	<0.01
	5	Kidney	<0.01	<0.01	<0.01
	Mean		<0.01	<0.01	<0.01

Dose level	Animal number	Matrix	Fluazinam [mg/kg][a]	AMPA [mg/kg][a]	DAPA [mg/kg][b]
Mid dose group	6	Kidney	<0.01	<0.01	<0.01
	7	Kidney	<0.01	<0.01	<0.01
	8	Kidney	<0.01	<0.01	<0.01
	Mean		<0.01	<0.01	<0.01
High dose group	9	Kidney	<0.01	<0.01	<0.01
	10	Kidney	<0.01	<0.01	<0.01
	11	Kidney	<0.01	<0.01	<0.01
	Mean		<0.01	<0.01	<0.01
Depuration phase					
High dose group-depuration, study day 29	12	Kidney	<0.01	<0.01	<0.01
High dose group-depuration, study day 31	13	Kidney	<0.01	<0.01	<0.01
High dose group-depuration, study day 35	14	Kidney	<0.01	<0.01	<0.01

[a] Hydrolysis procedure

[b] Non-hydrolysis procedure

Lactating cows were dosed for 28 days with fluazinam at feed levels of 2.91, 8.72 and 28.84 mg/kg dry weight for the low, mid and high dose group, respectively. When related to the body weight, the feeding rates were 0.67, 2.10 and 6.40 mg/kg bw/d for the low, mid and high dose group, respectively.

No residues of fluazinam were found in any milk sample (whole milk, skim milk and cream).

Mean residues of AMPA in milk were range from < 0.01 mg/kg to 0.011 mg/kg (day 5) for the high dose group. In the mid dose group residues of AMPA were < 0.01 mg/kg.

In skim milk, AMPA was < 0.01 mg/kg and in cream mean AMPA levels were 0.036 and 0.038 mg/kg for day 13 and 28, respectively.

Mean residues of DAPA in milk ranged from < 0.01 mg/kg) to 0.0178 mg/kg (day 7) for the high dose group. In the mid dose group residues were <0.01 mg/kg.

In skim milk, DAPA was <0.01 mg/kg and in cream, mean residues of DAPA levels were 0.065 and 0.084 mg/kg for day 13 and 28, respectively.

In the depuration phase of the high dose group, residues of fluazinam, AMPA and DAPA in milk were all <0.01 mg/kg.

In muscle, mean residues of fluazinam, AMPA and DAPA in muscle were <0.01 mg/kg in all samples.

In fat, residues of fluazinam were all <0.01 mg/kg. In the low dose group, mean residues of AMPA were highest in abdominal fat (0.011 mg/kg).

For the mid dose group, mean residues of AMPA were highest in the perirenal fat (0.023 mg/kg). In the high dose group, the mean AMPA levels were highest in abdominal fat (0.108 mg/kg).

In the low dose group, mean residues of DAPA were highest in abdominal fat (0.013 mg/kg). For the mid dose group, the mean residues of DAPA , was highest on perirenal phase and was 0.029 mg/kg. In the high dose group, DAPA levels were highest in abdominal fat (0.193 mg/kg).

Residues of fluazinam in liver were <0.01 mg/kg in all samples.

Mean residues of AMPA in liver were <0.01, <0.01 and 0.010 mg/kg for the low, mid and high dose group, respectively.

Mean residues of DAPA in liver were <0.01, 0.014 and 0.022 mg/kg for the low, mid and high dose group, respectively.

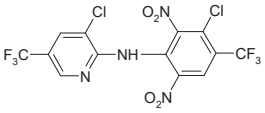
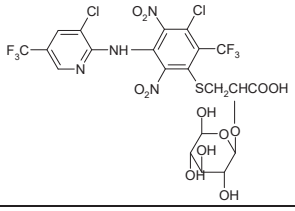
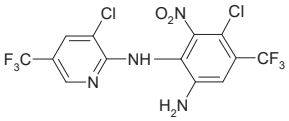
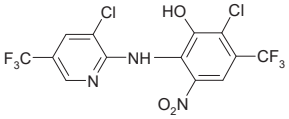
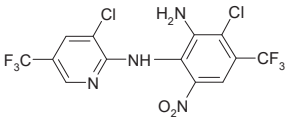
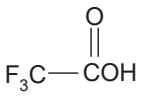
Residues of fluazinam, AMPA and DAPA in kidney were all <0.01 mg/kg in all samples.

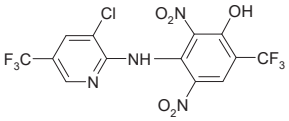
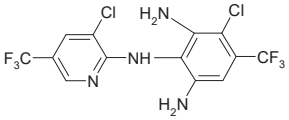
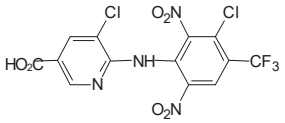
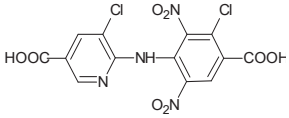
APPRAISAL

Fluazinam acts as a fungicide with activity against fungus from the class of *Oomycetes*, especially against *Phytophthora infestans*. It works protectively and needs to be applied before the disease attacks. At the Forty-eighth Session of the CCPR (2016), it was scheduled for evaluation as a new compound by the 2018 JMPR.

The Meeting received information on the identity, physical chemical properties, metabolism (plants, rotational crops and animals), environmental data, methods of analysis, freezer storage data, GAP information, supervised residue trials, fate of residues on processing and animal transfer studies.

In this document, the common names, chemical structures and chemical names of the compounds are as follows:

Chemical name (IUPAC)	Compound Name/Code	Structure	Occurrence in metabolism studies
3-Chloro-*N*-(3-chloro-5-trifluoromethyl-2-pyridyl)-*α,α,α*-trifluoro-2,6-dinitro-*p*-toluidine	Fluazinam, IKF-1216		Potatoes, peanut (foliage), grapes, apples, laying hen (liver, kidney, muscle, fat, egg yolk), RAT
3-[[4-amino-3-[[3-chloro-5-(trifluoromethyl)-2-pyridyl]amino]- *α,α,α*-trifluoro-6-nitro-*o*-tolyl]thio]-2-(β-D-glucopyranosyloxy) propionic acid	AMGT		Potatoes grapes, wine, apples
2-(6-amino-3-chloro-*α,α,α*-trifluoro-2-nitro-*p*-toluidino)-3-chloro-5-(trifluoromethyl) pyridine	AMPA		Potatoes, peanut (foliage), wine goat (liver, kidney, muscle, fat, milk), laying hen (liver, kidney, muscle, fat, egg yolk and white), RAT
2-chloro-6-[(3-chloro-5-(trifluoromethyl)-2-pyridyl)amino]- *α,α,α*-trifluoro-5-nitro-*m*-cresol	SDS-67230		Grapes, apples
2-(2-amino-3-chloro-*α,α,α*-trifluoro-6-nitro-*p*-toluidino)-3-chloro-5-(trifluoromethyl) pyridine	MAPA		Laying hen (liver, kidney, muscle, fat, egg yolk and white)
Trifluoroacetic acid	TFAA		Potatoes, peanut (foliage), apples, rotational crops:

Chemical name (IUPAC)	Compound Name/Code	Structure	Occurrence in metabolism studies
			lettuce (DAT 30) carrots (DAT 30) barley grain: DAT 120 DAT 365
5-[(3-chloro-5-(trifluoromethyl)-2-pyridyl)amino]- *α,α,α*-trifluoro-4,6-dinitro-*o*-cresol	HYPA		Laying hen (liver, kidney, muscle, fat, egg yolk and white); SOIL (major)
3-chloro-2-(2,6-diamino-3-chloro-*α,α,α*-trifluoromethyl-*p*-toluidino)-3-chloro-5-(trifluoromethyl) pyridine	DAPA		goat (liver, kidney, muscle, fat, bile, urine, milk) laying hen (liver, kidney, muscle, fat egg yolk and white), RAT
5-Chloro-6-(3-chloro-2,6-dinitro-4-trifluoromethylanilino) nicotinic acid	CAPA		Potato Hydrolysis
6-(4-Carboxy-3-chloro-2,6-dinitroanilino)-5-chloronicotinic acid	DCPA		Hydrolysis
4,9-dichloro-6-nitro-8-(trifluoromethyl)-pyrido-[1,2-α]benzimidazole-2-carboxylic acid	G-504		Hydrolysis

With respect to the physical and chemical properties that may impact on residues in crops, fluazinam is not regarded as volatile, it has a higher solubility in organic solvents compared to its solubility in water, the partition coefficient indicates its potential to sequester in fat, and aqueous photolysis and hydrolysis may play an important role in its degradation.

Plant metabolism

The Meeting noted that TFAA was identified in the plant metabolism studies (primary and rotational) formed as a result of ring cleavage and fragmentation. The plant metabolism studies were conducted with phenyl or pyridyl labelled fluazinam. The Meeting noted that it would not be possible to identify and quantify residues of TFAA that may have arisen from the pyridyl radiolabelled studies.

Potato

Study 1

Potatoes, grown outdoors, were treated with foliar applications of ^{14}C-fluazinam labelled in the phenyl or pyridyl ring. Two application regimes were investigated; in the low-dose regime potatoes received four applications of 0.6 kg ai/ha and in the high-dose regime potatoes received four applications at 1.8 kg ai/ha. The applications were performed 55, 76, 99 and 105 days after sowing.

Potato tubers were sampled 7 and 22 days after the last application, with the latter time period representing crop maturity. Potato tubers were separated into pulp and peel.

At 7/22 DALA the TRR for whole potato were: low dose-phenyl label (0.065/0.069 mg eq/kg), low dose-pyridyl label (0.055/0.072 mg eq/kg), high dose-phenyl label (0.11/0.11 mg eq/kg) and high dose-pyridyl label (0.105/0.10 mg eq/kg).

Initial solvent extractions were conducted with acetonitrile, acetonitrile: water (80: 20, v/v) and methanol: water (80: 20, v/v). Solvent extractable residues, in terms of whole potatoes, ranged from 30–51% TRR. Owing to the low radioactivity limited identification work was undertaken. In the peel samples (low dose, 22 DALA) all identified components, including fluazinam, were ≤ 0.004 mg eq/kg. A number of unidentified metabolites were found with the highest metabolite, unknown M3, occurring at a level of 0.011 mg eq/kg (0.002 mg eq/kg in terms of whole potato).

Study 2

Potatoes (variety *Kennebec*), grown outdoor, were treated four times, with a foliar spray, either with phenyl-labelled ^{14}C-fluazinam at a rate of 0.505 kg ai/ha or pyridyl-labelled ^{14}C-fluazinam at a rate of 0.43 kg ai/ha. Applications were made 40–41, 26–27, 15–16 and 6–7 days before harvest.

Total radioactive residues in potato were low: 0.0097 mg eq/kg (phenyl label) and 0.0236 mg eq/kg (pyridyl label).

Initial residues were extracted with acetonitrile. The extractable residue were 36% TRR for the phenyl label and 47% TRR for the pyridyl label. Owing to the low radioactivity, limited identification work was undertaken. TFAA was identified at a level of < 0.001 mg eq/kg, AMGT (< 0.001 mg eq/kg), AMPA (< 0.001 mg eq/kg) and fluazinam (0.001 mg /kg).

The PES, accounting for 48–51% TRR, was found to be almost entirely composed of starch.

Grapes

Field grown grapevines (variety *Pinot Noir*) were treated twice, with a foliar spray, with ^{14}C-phenyl-fluazinam or ^{14}C-pyridyl-fluazinam at the rate of 0.75 kg ai/ha. The first application was made at 80% of petal fall and the second at bunch closure (35 days after the first application). Samples were harvested 71 days after the last application. The TRR was 1.7 mg eq/kg from grapes treated with phenyl-label and 1.7 mg eg/kg in grapes treated with pyridyl-label.

Solvent extraction (acetonitrile: water, 90: 10, v/v) extracted 57% TRR (phenyl label) and 49% TRR (pyridyl label). In the extractable residue fluazinam (max 0.36 mg/kg, 21.3% TRR) was the major component. All other identified metabolites occurred at levels of < 4% TRR.

A large portion of the radioactivity present in the solids (PES) after the initial solvent extractions was found to be associated with natural products: 52% TRR for the phenyl label and 45% TRR for the pyridyl label.

The radioactivity in wine produced from the treated grapes was also investigated. The TRR residues in wine were: 0.73 mg eq/kg (vin de presse, both labels), 0.41 mg eq/kg (vin de goutte, phenyl label) and 0.54 mg eq/kg (vin de goutte, pyridyl label). The solvent (hexane and ethyl acetate) extractable residue ranged from 24–36% TRR. The aqueous phase accounted for 45% TRR for both the phenyl and pyridyl labels. The only two metabolites identified were AMPA (0.038 mg eq/kg, 5.2% TRR) and AMGT (0.076 mg eq/kg, 10% TRR). The ethanol, produced from the fermentation process, was found to contain radioactive residues (maximum 0.043 mg eq/kg, 5.9% TRR).

Apple

Apple trees (variety *Golden delicious*) grown outdoors were treated with a foliar spray with either phenyl or pyridyl-labelled fluazinam. A total of six applications of approximately 0.93 kg ai/ha per application were made. The first application was applied 161 days before harvest. The following five applications were made at intervals of 9, 22, 34, 34, and 30 days.

Samples were harvested 32 days after the last application.

The total radioactive residue levels in apples were 1.9 mg eq/kg and 2.8 mg eq/kg for the phenyl and pyridyl labels respectively. The apples were surface washed with acetonitrile which accounted for 36% TRR for the phenyl label and 46% TRR for the pyridyl label. Fluazinam (max 1.2 mg/kg, 42% TRR) and SDS-67230 (max 0.07 mg eq/kg, 2.5% TRR) were identified in the surface wash.

In terms of whole apple, including the surface wash, acetonitrile extracted 56% TRR for the phenyl label, and 64%TRR for the pyridyl label.

The whole apples were separated into pomace and juice.

For pomace the extraction with acetonitrile gave an extractability of 20–24% TRR (in terms of whole apple) for both labels. None of the identified metabolites (fluazinam, SDS-37230, AMGT and sugars) occurred at levels above 3% TRR in the solvent extract.

Enzymatic and acid hydrolysis of the PES of the pomace demonstrated a significant portion of the solids were associated with natural products: 26% TRR (phenyl label) and 30% TRR (pyridyl label).

In the juice, the metabolites identified (fluazinam, AMGT and sugars) were all at levels ≤ 5% TRR.

In summary, the main residue identified in apples was fluazinam, ranging from 37 to 45% of the TRR (0.69–1.2 mg/kg). The two metabolites of fluazinam that retained the basic structural form of the parent molecule, SDS-67230 and AMGT, were present at levels below 3% of the TRR (< 0.08 mg eq/kg). Radiolabelled sugars, formed by incorporation of radioactivity, accounted for 6-9% of the TRR (0.16–0.17 mg eq/kg), while structural polymeric compounds such as hemicellulose, pectin. Lignin and cellulose accounted for another 26–30% of the TRR (0.49–0.839 mg eq/kg). TFAA comprised < 1% of the TRR (0.003 mg eq/kg).

Peanut

Peanut plants, initially grown outdoors and then grown under protection, were treated four times with a foliar spray with either phenyl-labelled or pyridyl-labelled fluazinam at a rate of 0.56 kg ai/ha per application. The first application was made 56 days after planting and then at intervals of 21, 22 and 23 days.

Peanut nutmeat, shells and foliage were collected 90 days after the last application.

The TRR distributions for the phenyl/pyridyl labels were: foliage (25 mg eq/kg/32 mg eq/kg), shells (0.87 mg eq/kg/ 4.7 mg eq/kg) and nutmeats (0.85 mg eq/kg/ 1.2 mg eq/kg).

Initial solvent extraction was performed with acetonitrile: water (80: 20, v/v) for foliage and shells, and with hexane, acetonitrile and water for the nutmeats. The extractabilities for the phenyl/pyridyl labels were: foliage (37%/ 47% TRR), shells (55% / 44% TRR) and nutmeats (51%/ 54% TRR).

In nutmeats, neither fluazinam nor any metabolites containing the phenyl-pyridyl ring structure were present in detectable amounts (≥ 0.01 mg eq/kg). The major metabolites were TFAA (0.28 mg eq/kg, 38% TRR and fatty acids (0.23–0.58 mg eq/kg, 31–49% TRR).

Foliage contained detectable levels of fluazinam (1.8–2.3 mg/kg, 7.4–7.5% TRR) and the metabolite AMPA (0.24–0.4 mg eq/kg, 0.8–1.6% TRR). TFAA was also identified indicating that extensive metabolism of fluazinam had occurred.

In peanut shells, only fluazinam was identified.

The enzymatic, acid and base hydrolysis of the PES demonstrated that a significant portion of the radioactive residue was associated with natural products for the foliage and shells: foliage (49–53% TRR) and shells (40–52% TRR).

For nutmeats half of the radioactivity in the PES were found to be associated with natural products: 23–28% of the TRR.

In summary, the metabolism of fluazinam in peanuts was found to consist of extensive degradation and incorporation of the radioactivity into natural products

Summary of plant metabolism

In summary, the metabolism of fluazinam in primary crops of grapes, apples, potatoes and peanuts has been investigated. The metabolism of fluazinam proceeds through the reduction of one or both nitro groups to form AMPA and then replacement of the phenyl chlorine with a sulphur-containing side chain, followed by attachment of glucose to form AMGT. The metabolite SDS-67230 was also identified in apples and grapes.

Fluazinam is the main residue on plant parts such as foliage or fruit that are exposed to the spray application. However, fluazinam was not found in peanut nutmeats and only at low levels in potato tubers.

The appearance of radiolabelled natural products provides evidence that fluazinam is extensively metabolised. The presence of TFAA also supports the extensive metabolism of fluazinam and the incorporation into natural products. In potatoes, the fact that radioactivity from both phenyl ring- and pyridyl ring-labelled fluazinam appeared in starch indicated that both rings were broken down into fragments that could enter the carbon pool.

For the plant metabolites identified, only AMPA was observed in the rat metabolism studies.

Animal metabolism

The Meeting received animal metabolism studies with fluazinam in goats and hens. Evaluation of the metabolism studies in rats was carried out by the WHO core assessment group.

The tissues, milk and egg from the metabolism studies were stored at ≤ -18 °C for up to 6 months, negating the need to generate storage stability data. However, the Meeting noted that the storage stability data, generated using fortified samples, demonstrated that fluazinam, AMPA and DAPA were unstable in a number of animal matrices.

Within the livestock metabolism studies the metabolic profiles of various samples after different storage periods were compared. The metabolic profiles of ruminant liver, time zero compared to 4 months of storage, and milk, time zero compared to 7

month of storage, were comparable. For the hen liver and egg samples, metabolic profile changes were observed from the time zero compared to 4 months of storage. The changes were most prominent for three unidentified metabolites in egg yolk.

A comparison of the metabolic profiles for stored radiolabelled muscle (hen and goat) samples, for which the greatest instability was observed for the fortified samples, were not undertaken.

The meeting decided that owing to the instability observed in the fortified samples, in particular for muscle (goat), the lack of information on the stability of radiolabelled muscle samples (hen and goat) and the changes observed in the HPLC profiles for hen liver and egg, not to use the livestock metabolism studies to recommend residue definitions for animal commodities.

Environmental fate

The Meeting received information on the environmental fate and behaviour of fluazinam, including aerobic soil degradation, soil photolysis, aqueous photolysis and aqueous hydrolysis. Studies were also received on the behaviour of [^{14}C]-fluazinam in rotational crops.

Aerobic soil degradation

Soil degradation studies were conducted on two soil types at application rates ranging from 0.75–5 kg ai/ha. The primary degradates observed were MAPA (maximum 2.2% applied radioactivity (AR), 30 DAT), HYPA (maximum 14% AR, 48 DAT) and DAPA (1.9% AR, 14 DAT). The mineralisation of fluazinam into CO_2 accounted for up to a maximum 6% of the AR and soil bound residues accounted for up to 46% of the AR.

The DT_{50} values calculated for fluazinam ranged from 17–56 days for the sandy loam soil. A DT_{50} value of 212 days was calculated for the loamy sand soil.

For HYPA the DT_{50} value calculated for the sandy loam soil ranged from 166–257 days.

The Meeting considered that fluazinam was moderately–medium persistent in soil under aerobic conditions.

Soil photolysis

A photo-degradation study on a loamy sand soil was conducted with [^{14}C]-fluazinam at a dose rate of approximately 3 mg/kg. The samples were exposed to simulated sunlight for a 12 hour light/12 hour dark cycle for 30 days.

The DT_{50} values for the net photodegradation of fluazinam were 32 and 21 days for the phenyl and pyridyl labels respectively.

Fluazinam degraded moderately in light and represented an average of 35% of the AR after 30 days. After 30 days CO_2 accounted for an average of 2.4% of the AR and bound residues accounted for an average of 22% of the AR. The only metabolites identified were AMPA and HYPA, and after 30 days these metabolites accounted for an average of 4.7% and 6.2% of the AR respectively.

The Meeting considered that fluazinam was stable in soil when exposed to light.

Aqueous photolysis

The aqueous photolysis of fluazinam was investigated for [^{14}C]-fluazinam in sterile buffer at pH 5. The samples were exposed to simulated sunlight for a 12 hour light/12 hour dark cycle for 30 days. The only major analytes identified were G-504 (maximum 17% TRR, at day 10) and CO_2 (maximum 18% TRR after 30 days). The DT_{50} value for fluazinam was 2.5 days.

The Meeting concluded that photolysis may play an important role in the degradation of fluazinam.

Aqueous hydrolysis

Fluazinam was found to be hydrolytically stable at pH 4 for 5 days at 50 °C.

At pH 7 and 9 (stored for 29 days at 25 °C and 56 days at 50 °C) fluazinam was hydrolytically unstable.

At pH 7 and 25 °C fluazinam was hydrolysed to CAPA which was present at > 90% of the AR at the end of the incubation period. At pH 7 and 50 °C fluazinam was hydrolysed to CAPA and DCPA. At the end of the incubation period DCPA accounted for up to 71% of the AR and CAPA accounted for up to 29% of the AR.`

At pH 9 hydrolysis of fluazinam was comparable to that observed at pH 7.

The DT_{50} values calculated at pH 7 and 25 °C ranged from 2.7–4.5 days. At pH 9 and 25 °C the DT_{50} values ranged from 3.5–3.9 days.

The Meeting concluded that hydrolysis may play an important role in the degradation of fluazinam.

Confined rotational crop studies

A confined rotational crop study was undertaken with the application of either phenyl or pyridyl labelled fluazinam to the bare soil at an application rate of 2 × 1.12 kg ai/ha with an interval of 28 days between applications. Rotational crops of barley, carrots and lettuce were planted 30, 120 and 365 days after the last application.

The TRR in the mature crops tested were 0.04–0.30 mg eq/kg (lettuce), < 0.01–0.07 mg eq/kg (carrot roots), 0.034–0.35 mg eq/kg (carrot tops), 0.075–0.93 mg eq/kg (barley forage), 0.054–0.30 mg eq/kg (barely grain) and 0.093–1.2 mg eq/kg (barley straw).

The initial extraction was undertaken with methanol: acetone (1:1, v/v). The extractabilities were 51–95% TRR (mature lettuce), 69–92% TRR (mature carrot root), 45–91% TRR (mature carrot top), 8.8–78% TRR (barley grain), 68–96% TRR (barley forage) and 41–85% TRR (barley straw).

TFAA was found in the solvent extracts from all crops and all plant back intervals. The levels ranged from 0.004 mg eq/kg (35% TRR) for carrot roots for a PBI of 365 days to 0.88 mg eq/kg (94% TRR) for barely forage from a PBI of 120 days.

HPLC analysis of the solvent extracts resulted in several distinct regions being identified. The HPLC profiles indicated each region contained multiple components. In addition, the HPLC profiles of the extracts were different for the phenyl and pyridyl labels. These two pieces of information, along with the presence of TFAA in the rotational crops indicates cleavage of the two rings and extensive fragmentation.

Cellulase hydrolysis of the PES succeeded in releasing 11% TRR. Analyses of the aqueous fractions from enzyme hydrolysis indicated two regions of radioactivity. Subsequent mild acid and strong base hydrolysis succeeded in releasing most of the radioactivity from the PES. After base hydrolysis the resulting PES-fractions were all < 10% of the TRR and < 0.01 mg eq/kg, with the exception of phenyl-label barley straw where a TRR of 0.011 mg eq/kg was obtained.

Amylase hydrolysis of the grain PES demonstrated that up to 29% TRR was associated with starch.

In summary, in rotational crops no residues of fluazinam or related compounds based on the two-ring structure of fluazinam were found. Differences in the HPLC profiles from the phenyl and pyridyl labels indicate extensive metabolism of fluazinam. The only metabolite identified was TFAA. This occurred in significant amounts in lettuce (0.45 mg eq/kg, 120 day PBI), barley grains (0.18 mg eq/kg, 365 day PBI) and carrots (0.07 mg eq/kg, 30 day PBI). Enzymatic, base and acid hydrolysis did not release fluazinam or any other structurally related two ring structures. The incorporation into natural plant products, such as starch, was demonstrated.

Overall the metabolic pattern in rotational crops is more extensive than observed in primary crops.

The Meeting agreed that residues of TFAA could occur at significant levels in rotational crops.

The meeting noted that the rotational crop metabolism study was underdosed by a factor of 2.3 when considering the crops that can be rotated and the maximum application rates considered in this Meeting. In addition, since the position of the pyridyl –radiolabel does not address formation of the TFAA, no information on its presence in the raw agricultural commodities is available for the representative samples. For the phenyl-label, the meeting noted that TFAA was quantified using LSC-detection. It remains unclear if the lower radioactivity in TFAA compared to the full phenyl-label (1 vs 6 ^{14}C-atoms) was taken into account for the quantification of residues. The Meeting concluded that the data submitted are insufficient to estimate TFAA concentrations under field conditions.

Methods of analysis

Plant commodities

Residues were determined in crops using several different analytical methods. Following solvent extraction, using various solvents, and sample clean up, the majority of the methods employed GC-ECD to determine fluazinam. LC-MS/MS was also employed. An LOQ of 0.01 mg/kg was supported for fluazinam. The Meeting concluded that suitable methods are available for the determination of fluazinam in the crops under consideration.

An LC-MS/MS enforcement method was also validated for the determination of fluazinam in crops of high starch, high acid, high water, high protein and high oil content. Two ion transitions were validated and an LOQ of 0.01 mg/kg was supported for fluazinam in all five crop matrices. The method was successfully validated by an independent laboratory. The extraction efficiency of the method was not investigated. The method employed methanol: acetic acid (98: 2, v/v) as the extraction solvent compared to aqueous acetonitrile extractions employed in the plant metabolism studies.

The applicability of previous versions of FDA PAM methods for the determination of fluazinam in crops of high water content and high fat content was demonstrated.

Animal commodities

A method was investigated for the determination of fluazinam, AMPA and DAPA in animal matrices. This method was used in the ruminant feeding study.

In this method, milk and tissues were extracted with various solvents and then concentrated and partitioned in hexane. Following evaporation to near dryness and dissolving the residue in acetonitrile: water (1:1, v/v) final determination was achieved by GC-MS (DAPA/milk only) and LC-MS/MS (fluazinam, AMPA and DAPA).

For the determination of conjugates in liver and kidney, samples were also extracted with aqueous acetonitrile followed by an additional hydrolysis step (HCl at 37 °C for 1 hour).

Only three replicates were undertaken at each fortification level. However, the Meeting agreed the data were sufficient to conclude on the accuracy and repeatability of the method.

The Meeting agreed that the method had not been validated for all analyte/matrix combinations and in particular the recoveries were poor for fluazinam/ kidney, AMPA/kidney and DAPA/liver.

The extraction efficiency of this method was not investigated.

The applicability of the hydrolysis step was investigated with analytical standards of the free form of the analytes only, standards of the conjugates were not employed. The Meeting concluded that the validation data were not acceptable for the determination of AMPA in kidney and DAPA in liver and kidney. In addition, the Meeting concluded that as the validation data were not generated using standards of the conjugates, or using samples with incurred residues (e.g. if standards of the conjugates are unstable), then the efficiency of the hydrolysis step had not been investigated.

It was noted by the Meeting that validation data had been generated for only one ion transition.

The initial ILV of the method was unsuccessful. Owing to the poor reproducibility observed in the ILV, the extraction procedure (non-hydrolysis method) was modified and a second ILV undertaken. Overall the reproducibility of the modified extraction procedure was demonstrated for the determination of fluazinam, AMPA and DAPA in liver only.

The meeting concluded that the method employed in the ruminant feeding study was not suitable and therefore the results from the ruminant feeding study could not be relied on. With respect to enforcement, reproducibility has only been demonstrated for a modified extraction procedure for the determination of fluazinam, AMPA and DAPA in liver.

Stability of residues in stored analytical samples

Plant commodities

The freezer storage stability of fluazinam in homogenised plant samples fortified with fluazinam was investigated in a number of matrices. Fluazinam was found to be stable on storage in crops with high water content for at least 915 days, crops of a high acid content for at least 1144 days, crops of high starch content for at least 1096 days and crops of high oil content for at least 790 days.

Additional stability investigations were undertaken as part of a number of residue trials. In the majority of these studies both the stored samples and the residue trial samples were subjected to significant temperature variations throughout the study (maximum 0 to -40 °C). As a result of the instability of fluazinam observed in these crops (broccoli, mustard greens, snap beans, lima beans and ginseng) the Meeting concluded the trials could not be used to estimate maximum residue levels, STMRs or HR for fluazinam.

Data generated specifically on soya bean, alongside the residue trial samples, demonstrated that fluazinam was stable in soya bean under the storage conditions (≤ -10 °C for 153 days) employed in the residue trial.

Animal commodities

The stability of fluazinam, AMPA and DAPA in tissues and milk was investigated as part of the ruminant feeding study. Fluazinam, AMPA and DAPA were stable in milk, and fluazinam and AMPA were stable in fat, for the duration of the study. DAPA was not stable in fat, and fluazinam, AMPA and DAPA were not stable in liver and muscle. The Meeting concluded that as a result of the poor stability observed and the poor recoveries for the analytical method, the results of the ruminant feeding study could not be relied on.

Definition of the residue

Plant commodities

The nature of the residue was investigated in apple, grape, potato and peanut following foliar applications. The metabolic pathway is generally similar in all crops investigated but the extent of metabolism in the edible parts investigated differs.

In potatoes the TRR levels were low and significant residues were not identified.

In grapes, fluazinam was identified to be the main component of the residue accounting for up to 21% of the TRR. Fluazinam was also the main component of the residue identified in apple accounting for up to 45% of the TRR. In peanut the major compound identified was TFAA accounting for 38% of the TRR).

The nature of the residue in rotational crops was investigated in barley, carrots and lettuce. TFAA was found in significant amounts in lettuce (96% TRR), barley grains (59% of the TRR) and carrots (70% of the TRR). Concentrations were reported up to 0.45 mg eq/kg in lettuce. However, the Meeting concluded that the radio-label addressed only a portion of the total TFAA present.

The nature of the residue under simulated processing conditions was investigated. Under conditions representative of pasteurization (pH4, 90 °C, 20 minutes) fluazinam was found to be stable. However, under conditions representative of baking/brewing/boiling (pH5, 100 °C, 60 minutes) and sterilization (pH 6, 120 °C, 20 minutes) fluazinam was found to be unstable. Under conditions representative of baking/brewing/boiling fluazinam was 34–39% AR and CAPA was 51–56% AR. Under conditions representative of sterilization DCPA was 36–37% AR, G-504 was 11% AR and CAPA was 43–45% AR.

In summary, fluazinam and TFAA are the major compounds present in crops, and DCPA, G-504 and CAPA are the major degradates on processing.

TFAA can occur from several sources including other pesticides (e.g. flurtamone and saflufenacil) and as such would not be a suitable marker.

The Meeting considered that fluazinam was a suitable marker for the enforcement of MRLs for all crops.

Suitable analytical methods are available to determine fluazinam.

From a dietary risk perspective, as the WHO Core Assessment Group could not conclude on toxicological reference values for fluazinam, the Meeting was unable to consider a residue definition for dietary risk assessment.

In summary, based on the above, the Meeting recommended the following residue definitions for compliance with the MRL.

Definition of the residue for compliance with the MRL for plant commodities: *fluazinam*

The Meeting was unable to conclude on a residue definition for dietary risk assessment.

Results of supervised residue trials on crops

The Meeting received residue trials data for fluazinam on apple, grape, blueberries, bulb onion, cabbage, mustard greens, broccoli, melon, cucumber, summer squashes, peppers, lettuce, beans with pods, beans without pods, soya beans, carrot, potato, ginseng, peanuts and tea.

Due to the storage stability issues observed in the residue trials for broccoli, mustard greens, snap beans, lima beans and ginseng the Meeting concluded that maximum residue levels, STMRs and HRs could not be estimated for these crops.

TFAA was not included in the analysis of the samples from the residue trials considered in this Meeting.

Apples

The critical GAP in the USA is for ten foliar applications of 0.504 kg ai/ha with a re-treatment interval of 7 days and a PHI of 28 days. Trials conducted in Canada and the USA were provided.

Residues of fluazinam in apple approximating the GAP in rank order were (n = 13): 0.03, 0.03, 0.04, 0.12, 0.13, 0.14, 0.14, 0.14, 0.15, 0.16, 0.18, 1.4 and 1.5 mg/kg with the highest analytical result reported as 1.7 mg/kg.

For fluazinam the Meeting estimated a maximum residue level of 3 mg/kg for apples.

Grapes

GAP information was provided from Chile, Hungary and Italy. None of the trials matched the GAP for these countries. The Meeting concluded that a maximum residue level, a STMR and HR could not be estimated for grapes.

Subgroup of Bush berries

The critical GAP in the USA (Subgroup of Blueberries) is for a maximum of six foliar applications at a rate of 0.73 kg ai/ha. The re-treatment interval between applications is 7 days with a PHI of 30 days. Trials conducted in the USA were provided.

Residues of fluazinam in blueberries in rank order were (n = 9): 0.19, 0.25, 0.47,0.53, 0.67, 1.1, 1.4, 1.7 and 1.8 mg/kg with the highest analytical result reported as 2 mg/kg.

For fluazinam the Meeting estimated a maximum residue level of 4 mg/kg. The maximum residue level applies to the Subgroup of Bushberries.

Subgroup of Bulb Onion

The critical GAP in the USA (Subgroup Bulb Onion) is for 6 foliar applications at 0.583 kg ai/ha with a re-treatment interval of 7 days and a PHI of 7 days. Trials conducted in the USA matching GAP were provided.

Residues of fluazinam in bulb onion in rank order were (n = 9): < 0.01, < 0.01, < 0.01, 0.012, 0.016, 0.017, 0.032, 0.04 and 0.098 mg/kg with the highest analytical result reported as 0.10 mg/kg.

For fluazinam the Meeting estimated a maximum residue level of 0.15 mg/kg. The maximum residue level applies to the Subgroup of Bulb Onions.

Cabbage

The critical GAP in the USA is for a soil drench followed by foliar treatment. The soil drench treatment is 0.025 kg ai/hL with 100 mL of this solution being applied per plant (i.e. 0.025 kg ai/1000 plants) applied at or just after transplantation. The foliar use has a maximum individual application rate of 0.561 kg ai/ha with a total application rate of 3.36 kg ai/ha. The interval between applications is 7 days with a PHI of 7 days. Trials conducted in the USA were provided.

Residues of fluazinam in cabbage in rank order were (n = 8): 0.13, 0.23, 0.28, 0.39, 0.53, 0.67, 1.5 and 1.5 mg/kg with the highest analytical result reported as 1.7 mg/kg.

For fluazinam the Meeting estimated a maximum residue level of 3 mg/kg for cabbage.

Lettuce

The critical GAP in the USA is for one foliar application at 0.87 kg ai/ha with a PHI of 30 days. Six trials, conducted in the USA, can be regarded as supporting the GAP. These trials were all conducted on leaf lettuces. In addition, as the application rate in these trials were outside the 25% limit then the application rate and resulting residue levels needed to be scaled using the proportionality principle.

Residues of fluazinam in lettuce (unscaled) in rank order were (n = 6): < 0.01, 0.02, 0.02, 0.02, 0.16 and 1.6 mg/kg with the highest analytical result reported as 1.7 mg/kg.

Residues of fluazinam in lettuce were scaled using scaling factors ranging from 1.26–1.32.

Residues of fluazinam in lettuce (scaled) in rank order were (n = 6): < 0.01, 0.015, 0.015, 0.015, 0.12 and 1.2 mg/kg with the highest analytical result reported as 1.3 mg/kg.

For fluazinam the Meeting estimated a maximum residue level of 3 mg/kg for lettuce, leaf.

Subgroup of Fruiting Vegetables, Cucurbits–Melon, Pumpkins and Winter squashes

The critical GAP in the USA (Subgroup of Fruiting vegetables, Cucurbits–Melon, Pumpkins and Winter squashes), is for a maximum foliar application rate of 0.876 kg ai/ha with a total application of 5.26 kg ai/ha. The interval between applications is 7 days with a PHI of 30 days. Trials conducted in the USA matching this GAP were provided.

Residues of fluazinam in melon in rank order were (n = 8): < 0.01, < 0.01, 0.011, 0.014, 0.02, 0.021, 0.024 and 0.048 mg/kg.

For fluazinam the Meeting estimated a maximum residue level of 0.07 mg/kg. The maximum residue level applies to the subgroup of Fruiting Vegetables, Cucurbits–Melons, Pumpkins and Winter squashes.

Subgroup of Fruiting Vegetables, Cucurbits –Cucumbers and Summer squashes

The critical GAP is for the USA (Subgroup of Fruiting Vegetables, Cucurbits-Cucumber and Summer squashes), is for four foliar applications of 0.876 kg ai/ha, with an interval between applications of 7 days and a PHI of 7 days.

A total of six trials, conducted on cucumber, and six trials, conducted on summer squash, were provided. The trials were conducted in the USA. Two of the trials conducted on summer squash cannot be used as the storage interval from sampling to analysis is not supported. One trial in cucumber was regarded as an overdosed trial, but as the residue was < 0.01 mg/kg it is

regarded as supporting the GAP. The remaining trials do not reflect the GAP as 5 applications were made. The first application was a drench treatment and the Meeting agreed that the contribution of this treatment to the overall residue would be low and therefore the trials could be used to support the GAP.

Residues of fluazinam in cucumber and summer squash approximating the GAP in rank order were (n = 10): < 0.01 (7), 0.012, 0.013 and 0.027 mg/kg

For fluazinam the Meeting estimated a maximum residue level of 0.04 mg/kg. The maximum residue level applies to the Subgroup of Fruiting Vegetables, Cucurbits—Cucumbers and Summer squashes.

Subgroup of Peppers and Subgroup of Eggplant

The critical GAP in the USA (subgroup of Peppers and Subgroup of Eggplant), is for a maximum individual foliar application of 0.876 kg ai/ha with a total application rate of 5.26 kg ai/ha. The interval between applications is 7 days with a PHI of 30 days. The first application may be a soil drench treatment. Trials conducted in the USA were provided.

The trials do not reflect the GAP as the first two applications were a soil drench treatment. The Meeting concluded that the drench treatments early in the growing season are unlikely to impact on the final residue level and therefore the trials can be regarded as supporting the GAP. In five of the trials the interval between two of the applications exceeded the range of 7 days and was up to 55 days. As the residues from all trials were comparable the Meeting concluded all trials could be used to support the GAP.

Residues of fluazinam in peppers approximating the GAP in rank order were (n = 12): < 0.01 (5), 0.011, 0.015, 0.015, 0.016, 0.019, 0.03 and 0.054 mg/kg.

For fluazinam the Meeting estimated a maximum residue level of 0.07 mg/kg for peppers. The maximum residue level applies to the subgroup of peppers, except martynia, okra and roselle, and the Subgroup of eggplant.

Based on a drying factor of 10 the Meeting estimated a maximum residue level of 0.7 mg/kg for dried chili peppers.

Soya bean (dry)

The critical GAP in the USA is for a maximum individual dose of 0.583 kg ai/ha, a total maximum application of 1.17 kg ai/ha, 10 days between applications and a latest time of application at early pod formation. Trials conducted in the USA were provided.

Residues of fluazinam in soya bean in rank order were (n = 16): < 0.01 (16) mg/kg.

For fluazinam the Meeting estimated a maximum residue level of 0.01 mg/kg for soya bean.

Carrots

The critical GAP in the USA, is for maximum individual treatment rate of 0.583 kg ai/ha, with a maximum yearly application total of 2.33 kg ai/ha, a 7 day re-treatment interval and a PHI of 7 days. The GAP also specifies that no more than 4 applications can be made. Trials conducted in the USA were provided.

Within the trials submitted, several were regarded as replicate trials and hence the highest residue from the replicates has been selected.

Residues of fluazinam in carrot in rank order were (n = 8): < 0.02, 0.09, 0.1, 0.13, 0.13, 0.23, 0.37 and 0.51 mg/kg with the highest analytical result reported as 0.56 mg/kg.

For fluazinam the Meeting estimated a maximum residue level of 0.9 mg/kg for carrot.

Potato

The critical GAP in the USA, is for a total seasonal maximum application amount of 2.04 kg ai/ha with a maximum individual application rate of 0.292 kg ai/ha, 7–10 days between applications and a PHI of 14 days. Trials conducted in the USA were provided.

In a majority of the trials two replicate trials were undertaken to investigate different application regimes. In terms of the GAP the Meeting concluded that there were 8 trials that support the GAP. There was one further trial that represented an overdosed trial compared to the GAP. However, as the residue in the potato tuber was < 0.01 mg/kg it was regarded as supporting the GAP.

Residues of fluazinam in potato approximating the GAP in rank order were (n = 9): < 0.01 (9) mg/kg.

For fluazinam the Meeting estimated a maximum residue level of 0.01 mg/kg for potatoes.

Peanut

The critical GAP in the USA, is for a seasonal maximum application total of 2.34 kg ai/ha with a maximum individual application rate of 0.874 kg ai/ha, 21–28 days between applications and a PHI of 30 days. Trials conducted in the USA were provided.

Six trials support the GAP. A further three trials are regarded as overdosed trials compared to the GAP. However, as residues in the nutmeats were < 0.01 mg/kg then the trials were regarded as supporting the GAP.

Residues of fluazinam in peanut approximating the GAP in rank order were (n = 9): < 0.01 (9) mg/kg.

For fluazinam the Meeting estimated a maximum residue level of 0.01 mg/kg for peanut.

Tea, green, black (black, fermented and dried)

The critical GAP in Japan is for one foliar application at a rate of 0.025 kg ai/hL with a PHI of 14 days. The trials were conducted in Japan.

The samples were stored frozen for up to 6 months prior to analysis. Storage stability data supports a storage period of 5 months. However, no degradation was observed at 5 months of storage and therefore the Meeting concluded that the data were sufficient to cover the 6 months of storage.

Residues of fluazinam in tea in rank order were (n = 7): 0.4, 0.64, 0.67, 2.4, 2.6, 3.1 and 9.0 mg/kg with the highest analytical result reported as 10 mg/kg.

For fluazinam the Meeting estimated a maximum residue level of 15 mg/kg for tea, green, black (black, fermented and dried).

Animal feeds

Soya bean forage and hay, and Peanut hay

For soya bean and peanut the authorised label from the USA does not permit the feeding of animal feed items to livestock. Therefore the animal feed items from soya bean and peanut were not considered further.

Rotational crops

The Meeting noted that significant residues of TFAA could occur in rotational crops. However, rotational crop field trial data for TFAA were not provided to the Meeting.

Fate of residues during processing

High temperature hydrolysis

In the high temperature hydrolysis study, fluazinam was found to be stable under conditions representative of pasteurisation (pH 4, 90 °C, 20 minutes). However, under conditions representative of baking/brewing/boiling (pH 5, 100 °C, 60 minutes) and sterilisation (pH 6, 120 °C and 20 minutes) fluazinam was degraded to CAPA (maximum 56% AR) , G-504 (maximum 11% AR) and DCPA (maximum 37% AR).

Processing

The Meeting received information on the effects of processing on the magnitude of fluazinam residue levels for apple, grape, soya beans, potato and peanuts. The major degradates identified on hydrolysis (CAPA, G-504 and DCPA) were not investigated.

Data on residue levels of TFAA in processed commodities was not provided to the Meeting.

As residues in the raw agricultural commodities of potato tubers and peanut were < 0.01 mg/kg no processing factors could be derived. The processing factors (PF) determined for the other commodities, the best estimate PF, and the STMR-P and HR-P values estimated by the Meeting for fluazinam are outlined below:

Commodity	Individual processing factors for fluazinam	Best estimate PF for fluazinam	STMR-P for fluazinam (mg/kg)	HR-P for fluazinam (mg/kg)	Comment
Apple, Juice (raw)	0.33	-	0.0462	0.55	-
Apple, Juice (pasteurised)	0.33	-	0.0462	0.55	-
Apple, wet pomace	2.33	-	0.33	-	-
Apple, dry pomace	3	-	-	-	-
Grape, wet pomace	6.9, 5.13	6	-	-	Median PF
Grape, dry pomace	12.8, 6.25	9.53	-	-	-
Grape, juice	< 0.01, 0.25	0.25	0.15	1.78	Highest PF as 10 fold difference between two values
Raisins	0.25, 0.25	0.25	0.15	1.78	Mean PF
Grape, wine	0.39, 0.55, 0.38, 0.39,	0.39	0.23	2.77	Median PF

Commodity	Individual processing factors for fluazinam	Best estimate PF for fluazinam	STMR-P for fluazinam (mg/kg)	HR-P for fluazinam (mg/kg)	Comment
Grape, red wine	< 0.02, 0.33, 0.5	0.33	0.20	2.34	-
Grape, white wine	< 0.05	-			-

Residues in animal commodities

The Meeting received a lactating dairy cow feeding study which provided information on residue levels of fluazinam arising in tissues and milk when dairy cows were fed at rates of 2.5, 7.5 and 25 ppm. The Meeting concluded that as residues were not stable in all analyte/matrix combinations and the recovery data for the analytical method were poor, the feeding study could not be relied on.

RECOMMENDATIONS

Definition of the residue for compliance with the MRL for plant commodities: *fluazinam*

The Meeting was unable to conclude on a residue definition for dietary risk assessment for plant commodities.

DIETARY RISK ASSESSMENT

No Maximum residue levels are recommended, nor are levels estimated for use in long-term and acute dietary exposure assessments as the Meeting could not reach a conclusion on the residue definition for dietary risk assessment for plant commodities. In addition, the Meeting could not reach a conclusion on the residue levels of TFAA in the crops considered in this Meeting.

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