FLUBENDIAMIDE (242)

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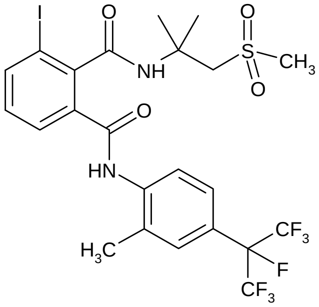
EXPLANATION

Flubendiamide belongs to a chemical family of benzenedicarboxamides or phthalic acid diamides with insecticidal activity through the activation of the ryanodine-sensitive intracellular calcium release channels, leading to the cessation of feeding immediately after ingestion of the compound. The compound was evaluated as a new compound by the 2010 JMPR for both residues and toxicological aspects. Information considered in this evaluation include metabolism of flubendiamide in farm animals and plants, methods of analysis, GAP information, supervised residue trials on pome fruit, stone fruits, cherries, grapes, broccoli, cabbage, tomato, peppers, sweet corn, cucumber, melon, watermelon, lettuce, green beans and peas, celery, corn, rice, tree nuts, soya bean, cotton and animal feed, processing and animal feeding studies.

IDENTITY

ISO Common name	Flubendiamide
Chemical name	
IUPAC:	3-iodo-N’-(2-mesyl-1,1-dimethylethyl)-N-{4-[1,2,2,2-tetrafluoro-1-trifluoromethyl)ethyl] -o-tolyl} phthalamide
CAS:	N^2-[1,1-dimethyl-2-(methylsulfonyl)ethyl]-3-iodo-N^1-[2-methyl-4-[1,2,2,2-tetrafluoro-1-(trifluoromethyl)ethyl]phenyl]-1,2-benzenedicarboxamide
CAS number:	272451-65-7
Synonyms:	
Structural formula:	
Molecular formula:	$C_{23}H_{22}F_7IN_2O_4S$
Molecular weight:	

PHYSICAL AND CHEMICAL PROPERTIES

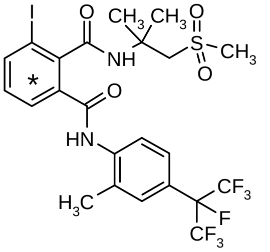
Pure active ingredient (purity > 98%)

Property	**Results**	**Reference**
Appearance	white crystalline powder	van der Baan-Treur, 2004a, b
Odour		
Melting point	217.5–220.7 °C	Bates, 2002 a
Relative density		
Vapour pressure *Knudsen Effusion technique*	< 10^{-4} Pa (at 200 °C)	Bates, 2002 c
Volatility (Henry's law constant)	2E-09 Pa m^3/mol, 20 °C	Schneider, 2004
Spectra for active substance	UV/VIS: Neutral: 204.4 nm (ε=39066) Acidic: 203.0 nm (ε=40500) Basic: 218.0 nm (ε=24967) No absorption for wavelengths > 290 nm IR: Absorbance corresponding to vibrations of C-H, N-H, aromatic C=C, C=O, and S=O. NMR : Coupling of protons was confirmed by H-H COSY spectrum, two amide protons observed at 8.428 and 9.782 ppm were assigned to alkyl amide proton and toluamide proton, respectively, according to NOESY MS : Molecular peak observed at m/z = 682, major fragment ion peaks at m/z= 663, 649, 531, 513, 487, 462, 444, 418, 408, 360, 274, 256, 206, 156, 103, 75, 69 and 55	Motoba, 2003 a
Solubility	Water (pH 4–10): 29.90 ± 2.87 µg/L at 20.0 °C and pH 5.98. Determined by column-elution method at two different flow rates. Solubility is obviously not pH-dependent in the range of 4–10 as it is indicated by the n-octanol/water partition coefficient	Motoba, 2003 c
	Organic solvents, at 19.8 °C (g/L) p-xylene: 0.488 n-heptane: 0.000835 methanol: 26.0 1,2-dichloroethane: 8.12 acetone: 102 ethyl acetate: 29.4	Bates, 2002d
Partition coefficient n-octanol/water *Shake* Flask Method	average pH = 5.91 - Log Po/w = 4.20 ± 0.01 at 24.9 ± 0.1 °C;, pH 4: Log Po/w = 4.13 ± 0.02 pH 7: Log Po/w = 4.14 ± 0.04 pH 9: Log Po/w = 4.11 ± 0.04	Motoba 2002 a; Motoba 2003 d
Hydrolysis	at pH 4, 7 and 9; sterile/dark conditions, at 25 °C: slightly hydrolysed	Yamashita, 2003
Photolysis	in sterile water (pH 6, 25 °C) Photolytic half-life 5.5 days (mean) DT_{50} at natural sunlight days (Tokyo, 35 °N latitude): 32.5 days (mean). Major degradates: NNI-des-iodo (A-1), NNI-3-OH (A-2) and NNI-3-OH-hydroxyperfluoro-propyl (A-10). Thereafter several degradates more polar than A-10 were generated. No unidentified degradate accounting ≥ 10% of applied radioactivity was found even after 168 hours irradiation.	Motoba, 2002 b
Quantum yield	Φ = 0.002408	Hellpointner, 2004

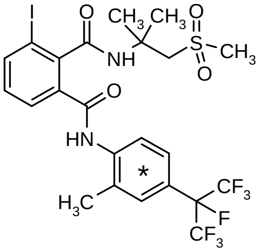
Property	Results	Reference
Dissociation constant	Not to be expected to dissociate within a pH range of 4 to 10	Bogdoll, 2005
Photochemical oxidative degradation	Reactions with OH radicals in the air: half-life of 8.78 hours, corresponding to a chemical lifetime of 12.7 hours. Based on these values no long-range transport and no accumulation in air are expected for flubendiamide	Hellpointner, 2003

METABOLISM AND ENVIRONMENTAL FATE

The metabolism of flubendiamide in plants and animals was investigated using [phthalic acid ring-UL-^{14}C] flubendiamide and [aniline ring-UL-^{14}C] flubendiamide. Residues of flubendiamide in succeeding crops were also investigated using these two labels. A list of the main metabolites and degradate compounds found in the studies are shown in Table 1.



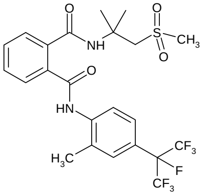
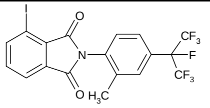
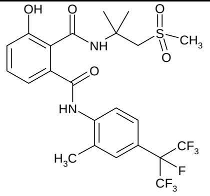
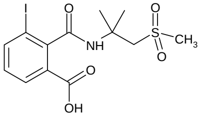
[phthalic acid ring-UL-^{14}C] flubendiamide

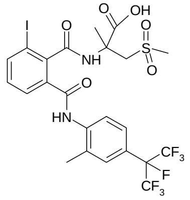
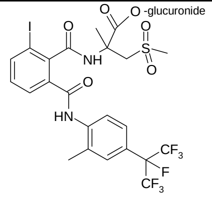
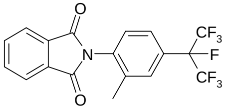
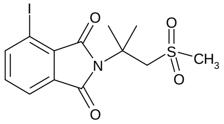
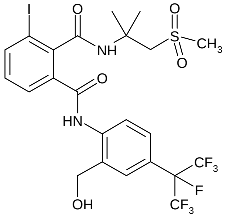
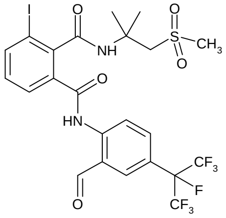
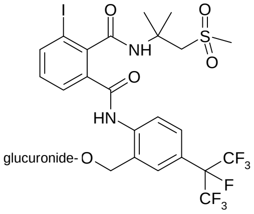
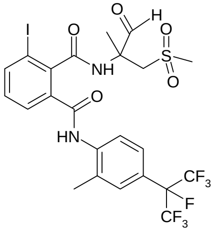


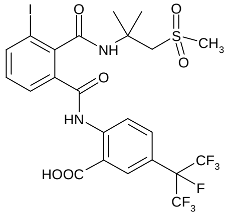
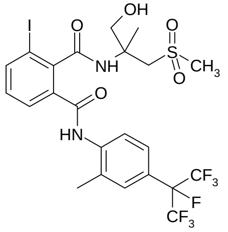
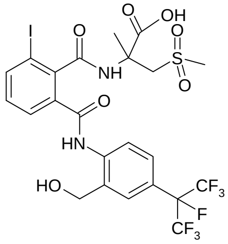
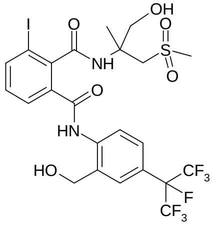
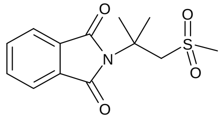
[aniline ring-UL-^{14}C] flubendiamide

Figure 1 ^{14}C labelled positions (*) of flubendiamide used in the metabolism studies

Table 1 Metabolites and degradate compounds found in the metabolism studies conducted with flubendiamide

Common name	Chemical Structure	Found in
flubendiamide-des-iodo		apples (fruits, leaves), cabbage (leaves), corn (forage, fodder), rice (leaves, stems, hulls), tomato (fruits, leaves), rotational crops
flubendiamide-iodophthalimide		goat, laying hen, rat, apples (leaves)
flubendiamide-3-OH		apples (leaves), cabbage (leaves), rice (leaves, stems), tomato (fruits, leaves)
flubendiamide-des-anilino		apples (leaves), tomato (fruits, leaves), rotational crops

Common name	Chemical Structure	Found in
flubendiamide-carboxy		Goat, laying hen
flubendiamide -carboxy-glucuronide		Goat, laying hen
flubendiamide -phthalimide		goat
flubendiamide -iodo-alkyl-phthalimide		goat, laying hen, rotational crops
flubendiamide-benzylalcohol		goat, laying hen, rat, apples (leaves), cabbage (leaves), rice (leaves, stems) tomato (fruits, leaves) rotational crops
flubendiamide -benzaldehyde		Rat, cabbage (leaves), rice (leaves, stems), tomato (fruits, leaves)
flubendiamide -benzylalcohol-glucuronide		goat
flubendiamide -formyl		goat

Common name	Chemical Structure	Found in
flubendiamide -benzoic acid		goat, rat, apples (leaves), cabbage (leaves), rice (leaves, stems), tomato (fruits, leaves), rotational crops
flubendiamide -hydroxy		Goat, laying hen, rat
flubendiamide -carboxy-benzylalcohol		laying hen
flubendiamide -hydroxy-benzylalcohol		laying hen
flubendiamide -desiodo-alkyl-phthalimide		rotational crops

Animal metabolism

The metabolism of flubendiamide was investigated in laying hen and lactating goat using both radiolabels.

Rats

Studies conducted in rats were submitted and evaluated by the WHO panel of the 2010 JMPR and reported in the toxicological evaluations

Poultry

The nature of residues in eggs and foodstuff originating from poultry dosed orally with flubendiamide by gavage was investigated in two studies using similar experimental designs. In one study, 6 laying hens received 14 daily oral doses of [phthalic acid ring-UL-^{14}C]flubendiamide at 1.0 mg/kg b.w./day (16.95 ppm in the diet) (Koester et al., 2005a; MEF-04/159). In the second study, 6 laying hens received 14 daily oral doses of [aniline ring-UL-^{14}C] flubendiamide at 0.71 mg/kg b.w./day (8.86 ppm in the diet) (Koester and Justus, 2004; MEF-04/055).

Eggs and faecal-urine excreta were collected once daily. The treated hens were sacrificed 24 hours after the last dose and liver, bile bladder, kidneys, leg and breast muscle, skin without subcutaneous fat, subcutaneous fat and eggs were sampled. Samples of eggs, excreta and organs/tissues from individual animals were pooled before extraction. The total radioactive residues (TRR) of each pool were determined by combustion/LSC (solid samples) or direct LSC. An aliquot was extracted four times with a mixture of acetonitrile/water or twice with pure acetonitrile followed by acetonitrile/water (eggs). The combined extracts were purified by solid phase extraction (SPE) using a C18 cartridge. The eluates were analysed by reversed phase column HPLC (radioisotope, UV and MS detectors) or and two-dimensional TLC. Solid samples were combusted prior to radioactivity determination and the formed $^{14}CO_2$ absorbed in an alkaline trapping solvent.

About 91% of the administered cumulative dose of [phthalic acid ring -UL-^{14}C]flubendiamide was recovered from organs, tissues, eggs, and combined faecal-urine excreta. The majority of the radioactivity (61.9%) was detected in the excreta until sacrifice, 24.4% in organs and tissues and 5.1% in eggs.

Table 2 shows the levels of parent compound and metabolites found in eggs, muscle, fat and liver. The residue concentration in eggs ranged from 0.135 mg/kg eq. within the first 4 days to 2.7 mg/kg eq. at the end of the dosing period. Flubendiamide was the main residue component in eggs and tissues; flubendiamide-benzylalcohol was detected as a minor metabolite, amounting up to 8.8% of the TRR in liver. Two metabolites, flubendiamide-iodophthalimide and flubendiamide-iodo-alkylphthalimide present in traces were tentatively identified (Table 2). Yolks had 12.14 mg/kg eq.; skin without subcutaneous fat 6.48 mg/kg eq. and kidney 2.41 mg/kg eq. Leg muscle had about three times higher residues than breast muscle (1.47 and 0.59 mg/kg eq., respectively).

Table 2 Distribution of residues in eggs, muscle, fat and liver of laying hens following oral administration of 14 daily doses of [phthalic acid ring -UL-^{14}C]flubendiamide at 1 mg/kg b.w

	Egg pool day 1-4		Egg pool day 5-9		Egg pool day 10-14		Muscle[b]		Fat		Liver	
Compound	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.
flubendiamide	92.2	0.135	92.7	1.098	92.1	2.69	95.0	1.00	97.9	17.69	81.9	3.30
flubendiamide -benzyl-alcohol	5.4	0.008	3.8	0.045	4.5	0.131	4.3	0.046	1.9	0.338	8.8	0.356
flubendiamide-iodophthalimide[a]	---	---	---	---	traces	traces	traces	traces	traces	traces	traces	traces
flubendiamide-iodo-alkyl-phthalimide[a]	---	---	---	---	traces	traces	---	---	---	---	---	---
Sum identified	97.6	0.143	96.5	1.14	96.6	2.83	99.3	1.05	99.8	18.03	90.7	3.66
Solids	2.1	0.003	2.5	0.030	3.2	0.092	0.5	0.005	0.0	0.004	6.7	0.269
Not analysed/losses	0.1	0.000	1.0	0.012	0.2	0.007	0.2	0.002	0.2	0.034	2.6	0.107
Total balance	100.0	0.147	100.0	1.18	100.0	2.92	100.0	1.06	100.0	18.07	100.0	4.04

[a] tentatively identified;

[b] mean of leg and breast muscle residues

About 98% of the orally administered cumulative dose of [aniline ring-UL-^{14}C]flubendiamide was recovered from organs and tissues, eggs and combined faecal-urine excreta of treated hens (Koester and Justus, 2004). The majority of the radioactivity (66.2% of the total administered dose) was detected in the excreta until sacrifice. The excretion rate was high and excretion started soon after the first administration. The absorbed fraction from the intestinal tract was estimated to be at least 32% of the total administered dose, with 24% found in organs and tissues and 7.7% in eggs.

Table 3 shows the levels of parent compound and metabolites found in eggs, muscle, fat and liver. The highest residue concentration in eggs (2.4 mg/kg equ.) was found at the end of the experiment. One day after the first administration, residues in eggs amounted 0.058 mg/kg equ. A continuous increase of the radioactivity level was determined over the whole test period starting after a lag-phase of 2 days. The parent compound was the main residue component, acoounting for 82 – 97% of the TRR in eggs and tissues. Flubendiamide-benzylalcohol was detected as a minor metabolite amounting up to 5.8% of the TRR in eggs (days 1–4) or 5.5% of the TRR in liver. A trace metabolite, flubendiamide-iodo-phthalimide was detected only in fat amounting to 1.6% of the TRR (Table 3).

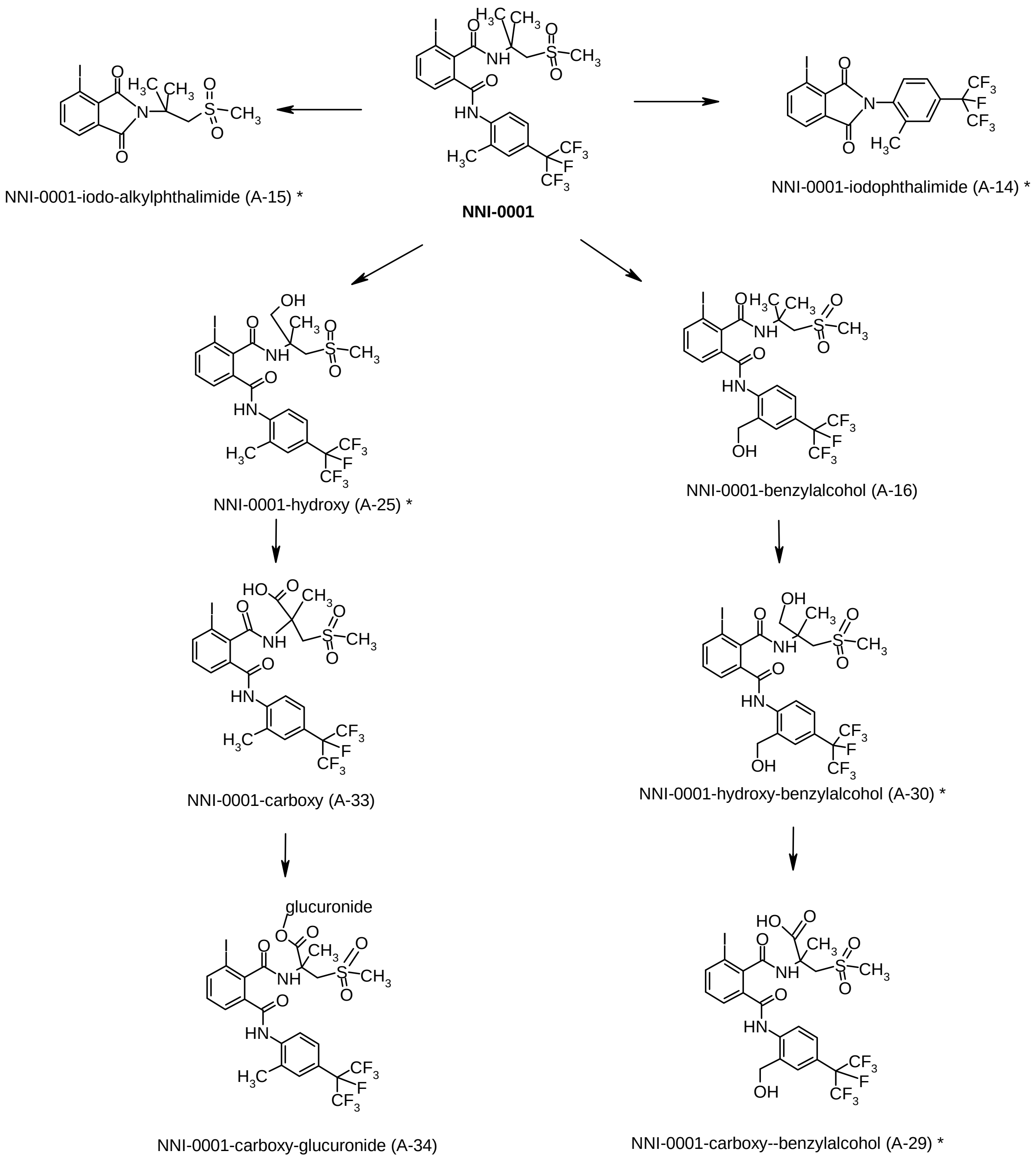
Eggs dissected from the ovary and oviduct had 9.30 mg/kg eq., skin without subcutaneous fat had 3.60 mg/kg eq, and kidney 1.81 mg/kg eq. Residues in leg muscle were higher than in breast muscle (1.02 and 0.38 mg/kg equ., respectively).

Table 3 Distribution of residues in eggs, muscle, fat and liver of laying hens following oral administration of 14 daily doses of [aniline ring-UL-^{14}C] flubendiamide at 0.71 mg/kg bw

Compound	Egg day 1-4		Egg day 5-9		Egg day 10-14		Muscle[a]		Fat		Liver	
	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.
flubendiamide	92.0	0.306	93.5	1.41	92.0	2.36	95.1	0.669	96.9	11.8	82.2	2.49
flubendiamide-benzylalcohol	5.8	0.019	2.5	0.037	2.8	0.071	3.9	0.028	1.0	0.124	5.5	0.167
flubendiamide-iodophthalimide							----	----	1.6	0.20	----	----
flubendiamide -aniline	n.d.	n.d.	n.d.	n.d.	< 0.2	< 0.005	< 0.3	< 0.002	< 0.2	< 0.024	< 0.2	< 0.007
Sum identified	97.8	0.326	95.9	1.44	94.7	2.43	99.0	0.697	99.6	12.17	87.7	2.66
Solids	1.7	0.006	3.0	0.045	3.4	0.088	0.5	0.004	0.0	0.002	9.3	0.283
not analysed/losses	0.5	0.002	1.1	0.016	1.8	0.047	0.5	0.004	0.4	0.052	3.0	0.091
Total balance	100.0	0.333	100.0	1.505	100.0	2.56	100.0	0.704	100.0	12.22	100.0	3.036

[a] mean of leg and breast muscle residues

The metabolism of flubendiamide in laying hens is shown in Figure 2. The major pathway was the oxidation of the methyl groups to form a primary alcohol (hydroxylation), further oxidation of the aliphatic alcohol to a carboxylic acid group followed by conjugation with glucuronic acid. The glucuronic acid conjugate of the carboxylic group was exclusively found in the excreta and in the bile. A minor reaction was the cleavage of the respective amide bond of flubendiamide and the cyclisation to flubendiamide-iodophthalimide and flubendiamide-iodo-alkylphthalimide.



* tentativaly identified as trace metabolite (only partly resolved chromatographic peaks)

Figure 2 Metabolic pathway of flubendiamide (NNI-0001) in the laying hen

Goat

Two studies were conducted to investigate the nature of residues in milk and foodstuffs from lactating goat dosed with labelled flubendiamide given by gavage. In the first study, one goat received 4 daily oral doses of [phthalic acid ring-UL-^{14}C]flubendiamide at a mean rate of 4.83 mg/kg b.w./day (176 ppm in the diet) (Koester *et al.*, 2005b; MEF-04/173). In the second study, one goat received 4 oral doses of [aniline ring UL-^{14}C] flubendiamide at a rate of 5 mg/kg b.w./day (370 ppm in the diet) (Weber *et al*., 2005; MEF-03/173). The studies used similar experimental protocols.

The goat was milked in the morning immediately prior to each dose, 8, 24, 32, 48, 56, 72, and 77 hours (at sacrifice) after the first dose. Urine and faeces fractions were collected in intervals of 24 hours after the first, second, and third dose and at sacrifice. An aliquot of each milk and urine fraction was taken and processed for LSC and the remaining samples stored at about -18 °C for metabolite analysis. Each faeces fraction was freeze-dried, homogenised, an aliquot subjected to combustion/LSC and the remainder stored at room temperature for the metabolite analysis.

Following sacrifice, liver, bile bladder, kidneys, muscle (round, flank, loin) and fat (perirenal, omental, subcutaneous) were sampled. The organs or tissue samples were minced in half-frozen state, a sample was freeze-dried, and three sub-samples prepared for tissue combustion/LSC. Portions of each organ or tissue sample were stored at about -18 °C for metabolite analysis. A pool of milk samples was extracted with acetonitrile, partitioned against n-heptane and again partitioned against acetonitrile. All resulting water/acetonitrile and acetonitrile extracts were combined, concentrated, and submitted to C18 SPE (phthalic acid ring-UL study) or styrene divinylbenzene polymer (aniline ring UL study). Organs, tissue and faeces samples were extracted with acetonitrile/water; fat was additionally extracted with n-heptane and the extracts submitted to C18 SPE. Solid samples were combusted prior to radioactivity determination and the formed $^{14}CO_2$ absorbed in an alkaline trapping solvent. Milk, muscle, fat, liver, and kidney extracts were submitted to hydrolysis under alkaline conditions. Structure elucidation was performed by HPLC (UV and radioisotope detectors), one-dimensional and two-dimensional TLC, HPLC/MS, GC/MS and/or NMR.

Until sacrifice (77 hours after the first dose), 53.7% of the administered radioactivity was recovered, mostly in the feces (44.2%). Tissues accounted for 8.7% of the dose and milk for 0.5%. Table 4 shows the distribution of residues in milk and edible tissues of a goat dosed with [phthalic acid ring-UL-^{14}C]flubendiamide. The highest residue levels at sacrifice were observed in fat (perirenal fat: 11.05 mg/kg eq., omental fat: 10.14 mg equ./kg, subcutaneous fat: 8.44 mg/kg eq.) and liver. The parent compound was the predominant component of the residue, accounting for 78.3–90.6% of TRR. Flubendiamide-iodophthalimide was detected at the highest level in milk and in fat. Liver contained all investigated metabolites, at levels < 5% of TRR.

Table 4 Distribution of residues in milk and tissues of a lactating goat following 4 daily doses of [phthalic acid ring -UL-^{14}C]flubendiamide at a mean dose rate of 4.83 mg/kg b.w (176 ppm in the diet)

Compound/comment	Milk		Muscle		Fat		Liver		Kidney	
	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.
Flubendiamide	78.3	0.547	86.9	0.721	89.2	8.793	79.9	8.103	90.6	2.207
Flubendiamide-iodophthalimide	11.4	0.080	6.5	0.054	10.6	1.045	2.4	0.242	1.6	0.039
Flubendiamide -phthalimide	2.6	0.018	1.3	0.011	---	-----	1.5	0.154	---	-----
Flubendiamide -hydroxy	1.1	0.008	2.7	0.022	---	-----	3.1	0.316	3.7	0.089
Flubendiamide -formyl	1.2	0.008	1.6	0.013	---	-----	2.7	0.276	2.3	0.056
Flubendiamide -carboxy	---	-----	---	-----	---	-----	3.8	0.390	---	-----
Flubendiamide -benzylalcohol	---	-----	---	-----	---	-----	2.1	0.209	---	-----
Flubendiamide -iodo-alkylphthalimide	---	-----	---	-----	---	-----	0.5	0.053	---	-----
Sum identified	94.5	0.660	99.1	0.822	99.8	9.838	96.1	9.743	98.2	2.392
Solids	4.6	0.032	0.5	0.004	0.1	0.005	2.1	0.216	0.7	0.018
not analysed/losses	0.8	0.006	0.5	0.004	0.1	0.015	0.7	0.073	1.0	0.025
Total balance	100.0	0.698	100.0	0.829	100.0	9.857	100.0	10.138	100.0	2.435

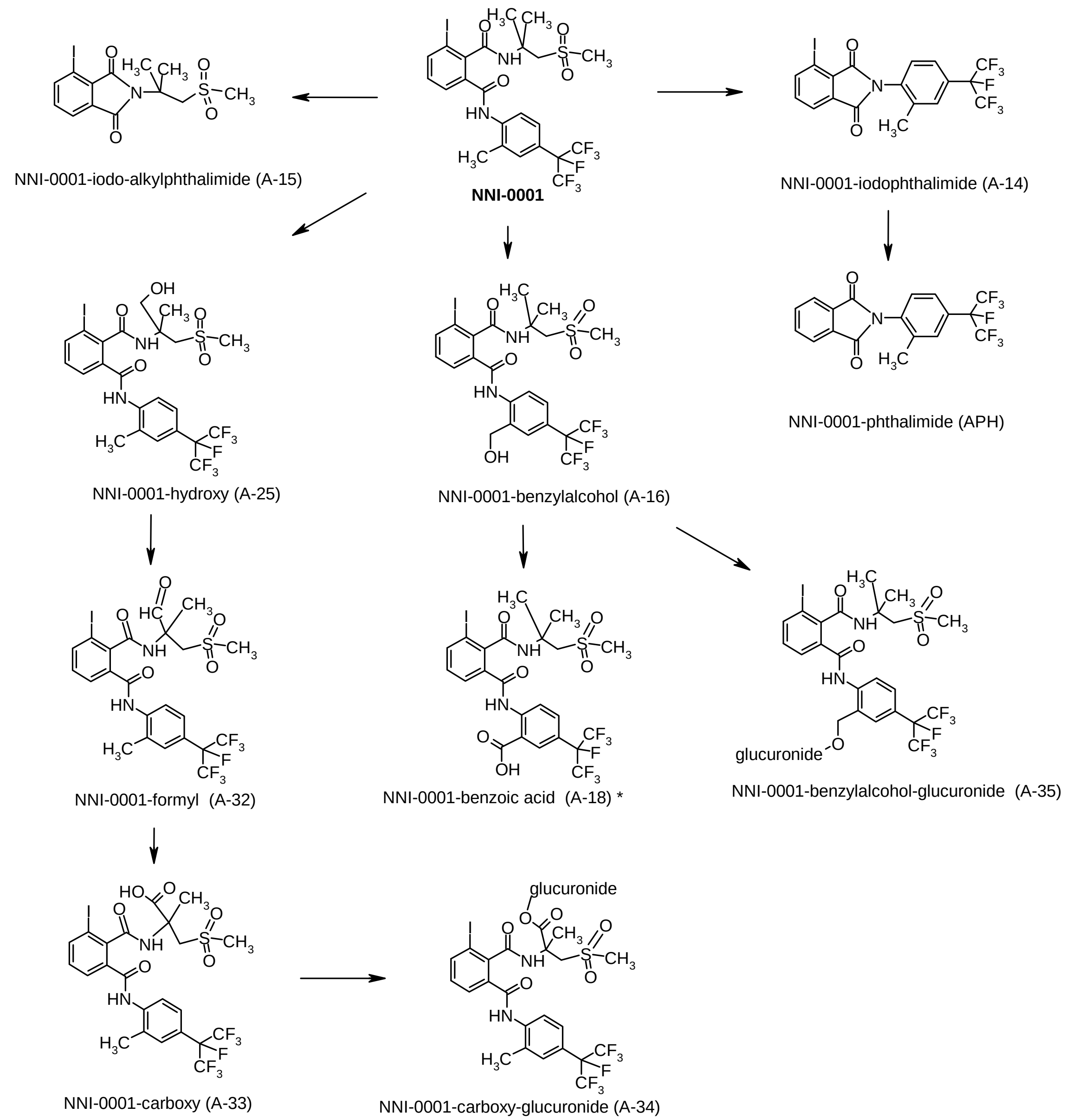
About 25% of the totally administered dose of [aniline ring UL ^{14}C]flubendiamide, was excreted until sacrifice, 96% in the faeces. Milk accounted for 0.4% and tissues for 15% of the totally administered dose. Table 5 shows the distribution of residues in milk and edible tissues of a goat dosed with [aniline ring-UL-^{14}C]flubendiamide. The parent compound was the main residue component. At sacrifice, the highest radioactivity level was measured in fat (omental fat: 21.8 mg/kg eq., perirenal fat: 20.5 mg/kg eq., subcutaneous fat: 16.6 mg/kg eq.). Residues in muscle were: 2.2 mg/kg eq. in flank muscle, 2.0 mg/kg eq. in loin muscle and 1.4 mg/kg eq. in round muscle. The major metabolite, flubendiamide-iodophthalimide accounted for approx. 17% of the TRR in milk, 8.4% in muscle and 24% in fat. Minor identified metabolites accounted for less than 6%TRR each.

Table 5 Distribution of residues in milk and tissues of a lactating goat following 4 daily doses of [aniline ring -UL-^{14}C]flubendiamide at a mean dose rate of 4.9 mg/kg b.w (370 ppm in the diet)

Report name/comment	Milk		Muscle		Fat		Liver		Kidney	
	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.
Flubendiamide	72.4	1.040	83.8	1.278	74.7	15.602	74.9	9.850	92.6	4.093
Flubendiamide-iodophthalimide	17.0	0.244	8.4	0.129	24.0	5.008	2.4	0.313	----	----
Flubendiamide-phthalimide	1.1	0.016	----	----	----	----	1.6	0.210	----	----
Flubendiamide -hydroxy	1.5	0.022	2.9	0.044	----	----	5.1	0.668	4.1	0.182
Flubendiamide -formyl	0.6	0.009	----	----	----	----	2.8	0.369	----	----
Flubendiamide -carboxy	----	----	----	----	----	----	5.3	0.691	----	----
Flubendiamide-benzylalcohol	0.9	0.012	1.0	0.015	----	----	2.4	0.322	----	----
Flubendiamide -aniline	< 0.5	< 0.007	< 0.2	< 0.004	< 0.3	< 0.053	< 0.3	< 0.040	< 0.5	< 0.022
Sum identified	93.5	1.343	96.1	1.466	98.7	20.611	94.5	12.422	96.7	4.275
Solids	0.3	0.004	0.6	0.009	0.1	0.013	3.3	0.440	1.4	0.063
not analysed/losses	6.2	0.088	3.3	0.050	1.2	0.260	2.2	0.290	1.8	0.081
Total balance	100.0	1.436	100.0	1.52	100.0	20.88	100.0	13.15	100.0	4.419

The intestinal absorption of [phthalic acid/aniline ring-UL-^{14}C]flubendiamide was estimated to be approximately 20/30% of the cumulative dose as derived from the amount of radioactivity detected in milk (0.53/0.4% of the total administered dose), urine (0.25/0.5% of the total administered dose), edible organs and tissues of the body at sacrifice (8.7/15% of the total administered dose), and the portion of metabolites in the faeces. The equivalent concentration in the plasma at 24 hours after dosing was about three times higher compared with the radioactivity level of the milk, indicating a slow absorption process from the intestinal tract and slow distribution into organs and tissues.

The metabolism of flubendiamide in goats is shown in Figure 3. It can be characterised by the oxidation of the methyl groups to a primary alcohol (hydroxylation) and further oxidation of the aliphatic alcohol group to a carboxylic acid which was partly conjugated with glucuronic acid. The glucuronic acid conjugate was exclusively found in bile and excreta. The cleavage of the amide bonds of flubendiamide followed by cyclisation to different flubendiamide-imides was a significant metabolic site. Deiodination was a minor pathway



* tentativaly identified as trace metabolite (only partly resolved chromatographic peaks)

Figure 3 Proposed metabolic pathway of flubendiamide (NNI-0001) in the lactating goat

Plants

The metabolism of flubendiamide after spray applications was investigated in five different crops – head cabbage, tomato, apple, corn and rice.

Cabbage

In the metabolism study conducted in cabbage (Motoba, 2002a), the test substances [phthalic acid ring-UL-^{14}C]-flubendiamide and [aniline ring-UL-^{14}C]-flubendiamide were dissolved in acetonitrile and applied to immature cabbage at an application rate of 300 µg/plant (5 mL/plant). For each label, 5 plants were treated. The treated plants were placed in a greenhouse at an average temperature of 20.6 °C and at an average humidity of 56.2%. The test systems were placed in the area under the quartz ceiling of a glasshouse to make the light irradiation conditions similar to field conditions. Samples were collected 3 weeks and 6 weeks after application (maturity). Two whole plants were sampled for each label at each sampling date. The edible parts (heads) and the non-edible parts (loose outer leaves, stems and roots) were collected separately. The outer leaves were rinsed (surface washed) with acetonitrile, the rinsed leaves, the head and the stems homogenized and extracted three times with acetonitrile/water. Solid residues were separated by centrifugation and further extracted with acetonitrile-0.1*N* HCl *aq*. Roots were cut and directly subjected to radioanalysis by combustion. Extracts and solid residues were subjected to LSC. The metabolites were analysed by TLC followed by radioluminography and HPLC.

The distribution of total radioactive residues (TRR) in cabbage head and loose outer leaves is shown in Table 6. All the applied radioactivity (AR) was recovered in the two experiments. Cabbage heads contained < 0.1% AR.

Table 6 Total radioactive residues (TRR) in edible and non-edible parts of cabbage after foliar application with [phthalic acid ring-UL-^{14}C]- and [aniline ring-UL-^{14}C]flubendiamide

	Immature plants, 3 weeks PHI		Mature plants, 6 weeks PHI	
	head	loose outer leaves	head	loose outer leaves
[phthalic acid ring-UL-^{14}C]-label, mg/kg eq. (% of AR).				
ACN rinsate	0.0001 (< 0.01%)	0.459 (77.8%)	< 0.0001 (< 0.01%)	0.480 (80.8%)
ACN /water extract (4/1)	0.0002 (0.01%)	0.088 (15.3%)	0.0001 (0.04%)	0.075 (13.3%)
ACN/0.1N HCl extract (4/1)	n.c.	0.012 (2.1%)	n.c.	0.012 (2.1%)
non-extracted residue	0.0004 (0.03%)	0.033 (5.8%)	0.0001 (0.02%)	0.027 (4.7%)
AR	0.0006 (0.05%)	0.593 (101%)	0.0004 (0.06%)	0.594 (101%)
[aniline ring-UL-^{14}C]-label, mg/kg eq. (% of AR).				
ACN rinsate	0.0001 (0.01%)	0.566 (86.6%)	< 0.0001 (< 0.01%)	0.493 (87.9%)
ACN/water extract (4/1)	0.0001 (0.01%)	0.088 (13.8%)	0.0001 (0.01%)	0.070 (12.4%)
ACN/0.1N HCl extract (4/1)	n.c. .	0.015 (2.3%)	n.c.	0.012 (2.2%)
non-extracted residue	0.0008 (0.06%)	0.032 (5.4%)	n.d.	0.030 (5.3%)
AR	0.0010 (0.07%)	0.701 (108%)	0.0001 (0.01%)	0.605 (108%)

n.c. = extraction was not conducted; n.d. = no radioactivity detected

Table 7 shows the characterisation of the residues extracted from the leaves. Due to the low residue levels, cabbage head extracts were not further analysed. Flubendiamide was the main compound detected in immature and mature plants treated with both radiolabels (> 90% of the applied radioactivity, AR). Flubendiamide-des-iodo and flubendiamide-3-OH were the main metabolites found in the leaf extracts, reaching up to 1.7% AR. Unidentified and non-extracted residues accounted for < 0.05 mg/kg eq. (< 10% AR).

Table 7 Parent compound and metabolites of [phthalic acid ring-UL-^{14}C]- and [aniline ring-UL-^{14}C]flubendiamide in cabbage leaves after foliar application

	Immature plants, 3 weeks PHI		Mature plants, 6 weeks PHI	
	mg/kg eq.	% AR	mg/kg	% AR
[phthalic acid ring-UL-^{14}C]-label				
Flubendiamide	0.5345	91.0	0.5380	91.2)
Flubendiamide -des-iodo	0.0076	1.3	0.0089	1.5
Flubendiamide -3-OH	0.0044	0.7	0.0075	1.3
Flubendiamide -benzylalcohol	0.0019	0.3	0.0032	0.6
Flubendiamide -benzoic acid	0.0006	0.1	0.0018	0.3
unidentified in extracts [a)]	0.0104	1.8	0.0096	1.3
non-extracted residue	0.0331	5.8	0.0271	4.7
Total	0.5926	101	0.5940	101
[aniline ring-UL-^{14}C]label				
Flubendiamide	0.6364	97.7	0.5408	92.3
Flubendiamide -des-iodo	0.0092	1.4	0.0093	1.7
Flubendiamide -3-OH	0.0068	1.0	0.0091	1.6

	Immature plants, 3 weeks PHI		Mature plants, 6 weeks PHI	
	mg/kg eq.	% AR	mg/kg	% AR
Flubendiamide -benzylalcohol	0.0027	0.4	0.0045	0.8
Flubendiamide -benzoic acid	0.0014	0.2	0.0028	0.5
unidentified in extracts [b]	0.0124	1.9	0.0091	1.6
non-extracted residue	0.0324	5.4	0.0298	5.3
Total	0.7013	108	0.6053	108

[a] each component ≤ 0.004 mg/kg (≤ 0.4%) for the phthalic acid label

[b] each component ≤ 0.004 mg/kg (≤ 0.6%) for the aniline label

Tomato

Immature fruits and leaves of cherry tomato plants were either treated with [phthalic acid ring-UL-^{14}C] or [aniline ring-UL-^{14}C]-labelled flubendiamide dissolved in acetonitrile (Motoba, 2002b). Eight plants were treated, 4 with each label. On each plant, two or three selected branches of fruits (5 fruits per branch) and of leaves were treated. Application was done with an auto-pipette directly onto the leaves or fruits. The application rates were 125 µg/branch of fruits (25 µg/fruit) and 800 µg/branch of leaves. Treated plants were placed under the quartz ceiling of a glasshouse at an average temperature of 27.2 °C and average humidity of 52.1%. Samples were collected at day 0, and 1, 2 and 4 weeks after application. At each sampling date, samples from two plants were taken per radiolabel, two branches per plant, one of treated fruits and one of leaves. In addition, 4 weeks after application whole plants were sampled and separated into different parts (untreated fruits, untreated leaves, stem, root) in order to investigate the translocation of the test substance into untreated plant parts. For this purpose, the plants from day 0 were used, from which either the treated leaves or treated fruits had been dissected immediately after application.

The surface of treated fruits and treated leaves was rinsed with acetonitrile and the rinsed fruit and leaves extracted with acetonitrile/water. The resultant solid residue of fruits was further extracted with acetonitrile-0.1*N* HCl *aq*. and acetonitrile-1*N* HCl *aq*. All samples were subjected to LSC and the metabolites analysed by TLC followed by radioluminography and HPLC.

The distribution of TRR is shown for fruits and leaves in Table 8 at all sampling dates and for both radiolabels. The ACN rinsates contained most of the radioactive residues; in fruits, the radioactivity decreased from 98.8% TRR at day 0 to 64.2% TRR four weeks later. Analysis of untreated plant parts four weeks after treatment showed less than 0.5% of the AR.

Table 8 Total radioactive residues (TRR) in fruits and leaves of tomato plants after application of [phthalic acid ring-UL-^{14}C] and [aniline ring-UL-^{14}C]flubendiamide onto immature fruits and leaves

	day 0		1 week		2 weeks		4 weeks	
	fruits	leaves	fruits	leaves	fruits	leaves	fruits	leaves
[phthalic acid ring-UL-^{14}C]-label, *mg/kg eq. (% of TRR)*								
ACN rinsate	3.226 (98.8%)	43.84 (106%)	2.364 (81.1%)	37.88 (99.2%)	1.591 (78.4%)	27.22 (96.5%)	1.287 (64.2%)	15.60 (84.8%)
ACN/water extract (4/1)	0.009 (0.3%)	0.248 (0.6%)	0.032 (1.1%)	0.633 (1.7%)	0.011 (0.6%)	0.564 (2.0%)	0.0315 (1.5%)	0.878 (4.9%)
ACN/0.1N HCl extract (4/1)	n.d. n.d.	n.c. n.c.	0.0001 (< 0.1%)	n.c. n.c.	0.0002 (< 0.1%)	n.c. n.c.	0.0016 (< 0.1%)	n.c. n.c.
ACN/1N HCl extract (4/1)	0.0006 (< 0.1%)	n.c. n.c.	n.d. n.d.	n.c. n.c.	n.d. n.d.	n.c. n.c.	0.0001 (< 0.1%)	n.c. n.c.
non-extracted residue	n.d. n.d.	0.0093 (< 0.1%)	n.d. n.d.	0.0278 (< 0.1%)	n.d. n.d.	0.0129 (< 0.1%)	0.0006 (< 0.1%)	0.0491 (0.3%)
Total radioactive residues	3.236 (99.1%)	44.10 (106%)	2.39 (82.1%)	38.54 (101%)	1.602 (78.9%)	27.80 (98.5%)	1.321 (65.9%)	16.53 (89.9%)
[aniline ring-UL-^{14}C]-label, *mg/kg eq. (% of TRR)*								
ACN rinsate	3.363 (98.9%)	45.05 (99.2%)	2.141 (83.0%)	36.98 (99.4%)	1.422 (77.6%)	26.66 (97.7%)	1.449 (66.9%)	14.60 (91.6%)
ACN/water extract (4/1)	0.012 (0.4)	0.3192 (0.7%)	0.0257 (1.0%)	0.5571 (1.5%)	0.0171 (1.0%)	0.3513 (1.3%)	0.0340 (1.6%)	0.280 (1.8%)
ACN/0.1N HCl	n.d.	n.c.	0.0006	n.c.	n.d.	n.c.	0.0019	n.c.

	day 0		1 week		2 weeks		4 weeks	
	fruits	leaves	fruits	leaves	fruits	leaves	fruits	leaves
extract (4/1)	n.d.	n.c.	(< 0.1%)	n.c.	n.d.	n.c.	(< 0.1%)	n.c.
ACN/1N HCl extract (4/1)	0.0002 (< 0.1%)	n.c. n.c.	n.d. n.d.	n.c. n.c.	n.d. n.d.	n.c. n.c.	0.0003 (< 0.1%)	n.c. n.c.
non-extracted residue	n.d. n.d.	0.0083 (< 0.1%)	n.d. n.d.	0.0145 (< 0.1%)	0.0129 (< 0.1%)	0.0133 (< 0.1%)	0.0006 (< 0.1%)	0.0199 (0.1%)
Total radioactive residues	3.375 (99.3%)	45.38 (99.9%)	2.167 (84.1%)	37.55 (101%)	1.452 (78.5%)	27.02 (99.1%)	1.486 (68.7%)	14.89 (93.5%)

n.c. = extraction was not conducted n.d. = no radioactivity detected

Table 9 shows the characterisation of the residues extracted from the tomato fruit. Unchanged parent compound was the main component detected in fruits and amounted to 1.27 and 1.43 mg/kg eq. after four weeks for the phthalic acid and aniline label, respectively. This corresponded to 63.4% and 66.3% of the applied radioactivity. Only small amounts of metabolites (each less than 0.01 mg/kg) were detected in fruits for both labels.

Table 9 Parent compound and metabolites of [phthalic acid ring-UL-^{14}C] and [aniline ring-UL-^{14}C]flubendiamide in tomato fruits

	fruits, day 0		fruits, 1 week		fruits, 2 weeks		fruits, 4 weeks	
	mg/kg	% AR	mg/kg	% AR	mg/kg	% AR	mg/kg	% AR
[phthalic acid ring-UL-^{14}C]-label								
Flubendiamide	3.218	98.6	2.371	81.3	1.584	78.0	1.271	63.4
Flubendiamide -des-iodo	0.002	< 0.1	0.004	0.1	0.003	0.2	0.007	0.3
Flubendiamide -3-OH	0.001	< 0.1	0.002	< 0.1	0.002	0.1	0.003	0.2
Flubendiamide -des-anilino	n.d.	n.d.	n.d.	n.d.	0.001	< 0.1	0.001	< 0.1
Flubendiamide-benzylalcohol	n.d.	n.d.	0.0013	< 0.1	0.002	0.1	0.005	0.3
Flubendiamide -benzoic acid	n.d.	n.d.	0.001	< 0.1	0.002	< 0.1	0.003	0.2
unidentified extracted	0.015	0.5	0.002	0.6	0.009	0.4	0.031	1.5
non-extracted residue	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.001	< 0.1
Total	3.236	99.1	2.395	82.1	1.602	78.9	1.321	65.9
[aniline ring-UL-^{14}C]-label								
Flubendiamide	3.359	98.8	2.147	81.3	1.420	77.4	1.435	66.3
Flubendiamide -des-iodo	n.d.	n.d.	0.002	< 0.1	0.003	0.2	0.005	0.2
Flubendiamide -3-OH	0.001	< 0.1	0.002	< 0.1	0.002	0.1	0.003	0.1
Flubendiamide-benzylalcohol	n.d.	n.d.	0.001	< 0.1	0.002	0.1	0.005	0.2
Flubendiamide -benzoic acid	n.d.	n.d.	0.002	< 0.1	0.002	0.1	0.004	0.2
unidentified extracted	0.015	0.4	0.014	0.6	0.010	0.5	0.034	1.6
non-extracted residue	n.d.	n.d.	n.d.	n.d.	0.013	< 0.1	0.001	< 0.1
Total	3.3750	99.3	2.164	84.1	1.452	78.5	1.486	68.7

Flubendiamide was also the main component found in leaves (Table 10). Only small amounts of metabolites (each less than 0.1 mg/kg) were detected in leaves for both labels. The sum of unidentified metabolites for both labels amounted to up to 0.034 mg/kg eq. (1.6% AR) in fruit and 1.19 mg/kg eq. (6.5% AR) in leaves. The maximum for a single unidentified compound amounted to less than 0.01 mg/kg (less than 10%) in fruits and leaves for both labels.

Table 10 Parent compound and metabolites of [phthalic acid ring-UL-^{14}C] and [aniline ring-UL-^{14}C]flubendiamide in tomato leaves after application onto immature fruits and leaves

	leaves, day 0		leaves, 1 week		leaves, 2 weeks		leaves, 4 weeks	
	mg/kg	% AR	mg/kg	% AR	mg/kg	% AR	mg/kg	% AR
[phthalic acid ring-UL-^{14}C]-label								
Flubendiamide	43.69	105	37.49	98.1	26.72	94.7	15.03	81.7
Flubendiamide -des-iodo	0.016	< 0.1	0.069	0.2	0.095	0.3	0.087	0.5
Flubendiamide -3-OH	0.002	< 0.1	0.039	0.1	0.060	0.2	0.039	0.2
Flubendiamide -des-anilino	n.d.	n.d.	0.008	< 0.1	0.022	< 0.1	0.017	< 0.1
Flubendiamide -benzylalcohol	n.d.	n.d.	0.004	0.1	0.066	0.2	0.066	0.4
Flubendiamide -benzoic acid	n.d.	n.d.	0.036	< 0.1	0.053	0.2	0.055	0.3
unidentified extractables	0.365	0.9	0.833	2.2	0.768	2.7	1.189	6.5

	leaves, day 0		leaves, 1 week		leaves, 2 weeks		leaves, 4 weeks	
	mg/kg	% AR	mg/kg	% AR	mg/kg	% AR	mg/kg	% AR
non-extracted residue	0.009	< 0.1	0.028	< 0.1	0.013	< 0.1	0.049	0.3
Total	44.10	106	38.54	101	27.80	98.5	16.53	89.9
[aniline ring-UL-^{14}C]-label								
Flubendiamide	44.99	99.1	36.61	98.4	26.27	96.3	14.18	89.1
Flubendiamide -des-iodo	n.d.	n.d.	0.045	0.1	0.064	0.2	0.054	0.3
Flubendiamide -3-OH	n.d.	n.d.	0.032	0.1	0.050	0.2	0.030	0.2
Flubendiamide -benzylalcohol	n.d.	n.d.	0.040	0.1	0.041	0.2	0.048	0.3
Flubendiamide -benzoic acid	n.d.	n.d.	0.050	0.1	0.059	0.2	0.053	0.3
unidentified extractables	0.380	0.8	0.754	2.0	0.524	2.0	0.506	3.2
non-extractaed residue	0.008	< 0.1	0.014	< 0.1	0.013	< 0.1	0.020	0.1
Total	45.38	99.9	37.55	101	27.02	99.1	14.89	93.5

Apples

The metabolism of flubendiamide in apples was investigated by Baker et al (2002). [Phthalic acid ring-UL-^{14}C]flubendiamide and [aniline ring-UL-^{14}C] flubendiamide were applied as an EC formulation (2.5% a.i.) to two apple trees (one for each label) by spray application at a rate corresponding to 0.11 kg ai/ha. Samples of apples and leaves were collected at 0, 7, 14, 28 and 56 days after treatment. Apple samples were rinsed in acetonitrile and first and second rinses were stored separately in freezers. Samples of rinsed apples and unrinsed leaves were subjected to combustion. A portion of processed (homogenised with dry ice) rinsed apples were extracted with acetonitrile, centrifuged and the pellet combusted. Homogenised leaves were extracted with acetonitrile followed by ACN/0.1N HC1 (4:1, v/v). The rinse solutions or extracts were analysed by HPLC and in some cases additionally by one- or two-dimensional TLC.

TRRs in apples were below 0.05 mg/kg at each harvest date and in each label, mostly present in the apple rinses (Table 11). The TRR in apple extracts were between 38 to 44%TRR 28 to 56 days after treatment. Residues in pellet were at 0.001-0.002 mg/kg eq.

Table 11 Total radioactive residues (TRR) in apples after spray application with [phthalic acid ring-UL-^{14}C]- and [aniline ring-UL-^{14}C]flubendiamide

DAT days	TRR	Apple rinses		Apple extracts		pellet		Recovery	
	mg/kg	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR	mg/kg eq.	% TRR
[phthalic acid ring-UL-^{14}C]-label									
0	0.016	0.013	81.25	0.003	16.88	0.001	6.25	0.017	104.4
7	0.017	0.012	70.59	0.005	31.76	0.002	11.76	0.019	114.1
14	0.018	0.012	66.67	0.005	27.78	0.002	11.11	0.019	105.6
28	0.018	0.010	55.56	0.007	38.89	0.002	11.11	0.019	105.6
56	0.011	0.004	36.36	0.004	40.18.	0.002	18.18	0.010	94.7
[aniline ring-UL-^{14}C]-label									
0	0.043	0.030	69.77	0.009	19.91	0.001	2.33	0.040	92.0
7	0.024	0.014	58.33	0.007	28.38	0.002	8.33	0.023	95.0
14	0.021	0.013	61.90	0.005	23.57	0.001	4.76	0.019	90.2
28	0.017	0.008	47.06	0.006	35.88	0.002	11.76	0.016	94.7
56	0.010	0.004	40.00	0.004	43.70	0.002	20.00	0.010	103.7

TRR in apple leaves are summarised in Table 12. Residues at day 0 were 4.5 mg/kg, dropping to 1.4–1.6 mg/kg at day 56, mostly recovered in the ACN leaf extracts. Residues in the pellet increased during the experiment to about 10%TRR.

Table 12 Total radioactive residues (TRR) in <u>leaves</u> after spray application with [phthalic acid ring-UL-^{14}C]- and [aniline ring-UL-^{14}C]flubendiamide

DAT	TRR	Leaf extracts ACN		Leaf extracts, ACN/HCl		pellet		Recovery	
days	mg/kg	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
[phthalic acid ring-UL^{-14}C]-label									
0	4.508	4.291	95.19	0.697	15.46	0.051	1.131	5.039	111.8
7	4.206	3.423	81.38	1.196	28.44	0.217	5.159	4.836	115
14	2.970	2.503	84.28	0.518	17.44	0.192	6.465	3.213	108.2
28	2.839	2.323	81.82	0.542	19.09	0.299	10.53	3.164	111.4
56	1.443	0.991	68.68	0.226	15.66	0.163	11.30	1.380	95.6
[aniline ring-UL-^{14}C]-label									
0	4.452	4.146	93.13	0.824	18.51	0.068	1.527	5.038	113.2
7	4.279	3.502	81.84	0.906	21.17	0.163	3.809	4.571	106.8
14	4.801	3.481	72.51	0.901	18.77	0.186	3.874	4.568	95.2
28	3.425	2.841	82.95	0.567	16.55	0.295	8.613	3.703	108.1
56	1.643	1.288	78.39	0.289	17.59	0.171	10.41	1.748	106.4

Parent compound and metabolites identified by HPLC apples extract is shown in Table 13. Flubendiamide was the major compound detected in both label experiments; flubendiamide-des-iodo was detected at ≤ 0.002 mg/kg.

Table 13 Parent compound and metabolites of [phthalic acid ring-UL-^{14}C] and [aniline ring-UL-^{14}C]flubendiamide in <u>apples</u> after spray application

Metabolites	apples, day 0		apples, day 7		apples, day 14		apples, day 28		apples, day 56	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
[phthalic acid ring-UL-^{14}C]-label										
Flubendiamide	0.015	93.8	0.016	94.1	0.014	77.8	0.013	72.2	0.006	54.5
Flubendiamide-des-iodo	n.d.	n.d.	< 0.001	< 5.9	< 0.001	< 5.6	0.001	5.6	< 0.002	< 18.2
Peak at 22.25 min	< 0.001	< 6.3	< 0.001	< 5.9	< 0.001	< 5.6	< 0.001	< 5.6	< 0.001	< 9.1
Other unidentified	< 0.001	< 6.3	< 0.001	< 5.9	< 0.001	< 5.6	< 0.002	< 11.1	< 0.002	<18.2
non-extracted residue	0.001	6.3	0.002	11.8	0.002	11.1	0.002	11.1	0.002	18.2
TRR	0.016		0.017		0.018		0.018		0.011	
[aniline ring-UL-^{14}C]-label										
Flubendiamide	0.035	81.4	0.018	75.0	0.014	66.7	0.009	52.9	0.005	50.0
Flubendiamide-des-iodo	< 0.002	<4.7	< 0.001	< 4.2	< 0.002	< 9.5	0.002	11.8	< 0.001	<10.0
Peak at 22.25 min	< 0.002	<4.7	< 0.001	<4.2	< 0.001	< 4.8	< 0.001	< 5.9	< 0.001	<10.0
Other unidentified	< 0.001	<2.3	< 0.001	<4.2	< 0.001	< 4.8	< 0.002	< 11.8	< 0.001	<10.0
non-extracted residue	0.001	2.3	0.001	4.2	0.001	4.8	0.002	11.8	0.002	20.0
TRR	0.043		0.024		0.021		0.017		0.010	

Flubendiamide was the predominant analyte detected in leaves, accouting for over 50% TRR at day 56 (Table 14). Flubendiamide-des-iodo was the major metabolites detected in leaf extracts (0.167–0.205 mg/kg). Several other unidentified metabolites were also detected; a maximum residue of 0.094 mg/kg was associated with a polar region. Other unidentified metabolites observed in leaves were < 0.05 mg/kg.

Table 14 Parent compound and metabolites of [phthalic acid ring-UL-^{14}C]- and [aniline ring-UL-^{14}C]flubendiamide in apple leaves after spray application

Metabolites [a]	leaves, day 0		leaves, day 7		leaves, day 14		leaves, day 28		leaves, day 56	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
[phthalic acid ring-UL-^{14}C]-label										
Flubendiamide	4.763	105.7	4.191	99.6	2.358	79.4	2.087	73.5	0.763	52.9
Flubendiamide-des-iodo	0.035	0.8	0.167	4.0	0.093	3.1	0.135	4.8	0.104	7.2
Flubendiamide -3-OH	n.d.	n.d.	0.022	0.5	0.113	3.8	0.072	2.5	0.010	0.7

Metabolites a)	leaves, day 0		leaves, day 7		leaves, day 14		leaves, day 28		leaves, day 56	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Flub-des-anilino	n.d.	n.d.	n.d.	n.d.	0.075	2.5	0.011	0.4	n.d.	n.d.
Flub-iodo-phthalimide	0.011	0.2	0.008	0.2	0.005	0.2	n.d.	n.d.	n.d.	n.d.
Flub-benzyl alcohol	0.004	0.1	0.039	0.9	0.047	1.6	0.035	1.2	0.031	2.1
Flub-benzoic acid	0.017	0.4	n.d.	n.d.	0.045	1.5	0.038	1.3	0.050	3.5
Flub-3-iodo-phthalic acid	n.d.	n.d.	n.d.	n.d.	0.046	1.5	0.176	6.2	0.030	2.1
Peak at 5.25 min	n.d.	n.d.	n.d.	n.d.	0.004	0.1	0.049	1.7	0.021	1.5
Peak at 19.25 min	0.031	0.7	0.026	0.6	0.027	0.9	n.d.	n.d.	0.030	2.1
Peak at 22.25 min	0.082	1.8	0.021	0.5	0.016	0.5	0.032	1.1	0.023	1.6
Other unidentified	0.044	1.0	0.153	3.6	0.261	8.8	0.222	7.8	0.188	13.0
non-extracted residue	0.051	1.13	0.217	5.16	0.192	6.47	0.299	10.53	0.163	11.30
TRR	4.51		4.21		2.97		2.84		1.44	
[aniline ring-UL-^{14}C]-label										
Flubendiamide	4.611	103.6	4.024	94.0	3.695	77.0	2.650	77.4	1.026	62.4
Flubendiamide-des-iodo	0.069	1.5	0.205	4.8	0.181	3.8	0.182	5.3	0.114	6.9
Flubendiamide -3-OH	n.d.	n.d.	n.d.	n.d.	0.109	2.3	0.097	2.8	0.072	4.4
Flub-iodo-phthalimide	0.103	2.3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Flub-benzyl alcohol	0.013	0.3	0.019	0.4	0.055	1.1	0.052	1.5	0.043	2.6
Flub-benzoic acid	0.013	0.3	0.016	0.4	0.066	1.4	0.086	2.5	0.054	3.3
Peak at 5.25 min	n.d.	n.d.	n.d.	n.d.	0.080	1.7	0.033	1.0	0.094	5.7
Peak at 19.25 min	0.024	0.5	0.007	0.2	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Peak at 22.25 min	n.d.	n.d.	0.015	0.4	0.056	1.2	0.041	1.2	0.018	1.1
Other unidentified	0.139	3.1	0.121	2.8	0.141	2.9	0.271	7.9	0.153	9.3
non-extracted residue	0.068	1.53	0.163	3.81	0.186	3.87	0.295	8.61	0.171	10.41
TRR	4.45		4.28		4.80		3.42		1.64	

[a] in sum of rinses (ACN) and extracts (ACN/0.1 N HCl)

[b] no individual metabolite > 0.1 mg/kg;

n.d. - not detected.

Sweet Corn

The metabolism of flubendiamide in sweet corn was investigated by Krolski and Nguyen (2005). [phthalic acid ring-UL-^{14}C]flubendiamide and [aniline ring-UL-^{14}C] flubendiamide were applied 4 times, at a 7-day interval, at 0.16 kg ai/ha. Forage (that includes husks) and sweet corn samples were collected one day after the fourth treatment. Corn grain and fodder (cobs plus stalk plus husk) were collected at 22 days PHI. Grain and fodder were processed with dry ice, portions taken for radioassay and the remainders stored freezen for analysis. An aliquot of pulverized forage was blended with ACN/water, centrifuged and the remaining dried solids extracted with acetonitrile heated at reflux. The suspension was filtered and extracted with methanol heated at reflux. The filtered solids were removed, the remaining solids from the refluxing methanol extraction were suspended in 2*N* NaOH and heated at reflux. Other samples (sweet corn, fodder and grain) were also extracted in several steps. Extracts and solid residues after combustion were subjected to LSC. The main extracts (ACN/water) were analysed by HPLC/MS. The total radioactive residue (TRR) of all samples was determined by combustion of aliquots.

TRR of forage and fodder was within the range of 0.288 to 0.6 mg/kg eq., mostly found in the acetonitrile/water extracts (Table 15). TRR derived by combustion of sweet corn and corn grain samples were 0.01 and 0.016 mg/kg eq., from the phtalic acid ring label, respectively, and 0.001 and 0.003 mg/kg eq. in the aniline ring label, respectively. ACN/water extracts of sweet corn and corn grain of the phthalic acid labelrepresented 37 and 15% TRR, respectively; methanol under reflux and alkaline conditions extracted about 20 and 13% TRR of sweet corn and grain, respectively. Due to the low TRRs, the sweet corn and corn grain samples of the aniline label were not further analysed.

Table 15 Total radioactive residues (TRR) and results of extraction in corn after spray application with [phthalic acid ring-UL-^{14}C] and [aniline ring-UL-^{14}C]flubendiamide

	forage		sweet corn		fodder		corn grain	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
[phthalic acid ring-UL-^{14}C]-label								
Hexane	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	< 0.001	3
ACN/H2O	0.275	95	0.004	37	0.415	87	0.002	15
ACN reflux	0.003	1	n.g.	n.g.	0.018	4	n.g.	n.g.
MeOH reflux	0.002	1	0.002	20	0.009	2	0.002	13
1N HCl, RT	n.g.	n.g.	< 0.001	2	n.g.	n.g.	n.g.	n.g.
1N HCl, reflux	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	0.001	4
2N NaOH, RT	n.g.	n.g.	0.003	33	0.027	6	n.g.	n.g.
2N NaOH, reflux	0.006	2	n.g.	n.g.	n.g.	n.g.	< 0.001	<1
6N NaOH, reflux	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	0.009	53
6N HCl, reflux	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	0.002	14
non-extracted residue	0.001	0.4	0.001	8	0.008	2	-	-
TRR	0.288	100	0.010	100	0.476	100	0.016	100
[aniline ring-UL-^{14}C]-label								
Hexane	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.
ACN/H2O	0.570	95	n.g.	n.g.	0.369	94	n.g.	n.g.
ACN reflux	n.g.	n.g.	n.g.	n.g.	0.01	3	n.g.	n.g.
MeOH reflux	n.g.	n.g.	n.g.	n.g.	< 0.01	2	n.g.	n.g.
1N HCl, RT	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.
1N HCl, reflux	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.
2N NaOH, RT	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.
2N NaOH, reflux	n.g.	n.g.	n.g.	n.g.	< 0.01	1	n.g.	n.g.
6N NaOH, reflux	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.
6N HCl, reflux	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.	n.g.
non-extracted residue	0.029	5	n.g.	n.g.	< 0.01	<1	n.g.	n.g.
TRR	0.599	100	0.001	100	0.394	100	0.003	100

ng = fraction not generated.

The distribution of total radioactive residues in corn forage and fodder is shown in Table 16. TRR levels were between 0.3 to 0.6 mg/kg eq., with over 75% TRR as parent compound. Flubendiamide-des-iodo was detected at levels from 0.03 to 0.05 mg/kg eq. No unidentified residues represented > 1% of the TRR in any matrix.

Table 16 Parent compound and metabolites of [phthalic acid ring-UL-^{14}C] and [aniline ring-UL-^{14}C]flubendiamide in corn after spray application

	forage		fodder	
	mg/kg	% TRR	mg/kg	% TRR
[phthalic acid ring-UL-^{14}C]-label				
Flubendiamide	0.212	77	0.324	78
Flub-des-iodo	0.050	18	0.036	9
total	0.262	95	0.360	87
[aniline ring-UL-^{14}C]-label				
Flubendiamide	0.513	90	0.308	84
Flub-des-iodo	0.029	5	0.037	10
total	0.542	95	0.345	94

Rice

The metabolism of flubendiamide in rice was investigated by Motoba (2004). Application of [phthalic acid ring-UL-^{14}C]flubendiamide was done just before ear emergence 76 days after seeding and 63 days before the final sampling at maturity. The application suspensions (49.6 ± 0.5 µg eq./mL) were sprayed to the test plants and after drying of the droplets on the plant surface, the plants were transferred to the greenhouse. The average temperature and humidity were 27.6°C and 66.4%, respectively. Light intensity was not monitored. Four pots of rice plants were sampled. The first plant sample (one pot) was taken at time zero, and the second four weeks after application (sampling of immature plants). For the last sample (9 weeks, mature rice plants) two pots were used. Immediately after the treatment, when the droplets had dried, leaves with stems were sampled. Four weeks after treatment, rice plants were sampled separately in parts (leaves with stems, ear and root). Nine weeks after application the samples of one pot were immediately rinsed and extracted, and samples from the other pot were dried for a week in the glasshouse simulating the normal horticultural procedure before they were rinsed and extracted.

The surface of leaves, stems, immature and mature ears were rinsed with acetonitrile; the rinsed plant samples were extracted with ACN/water and further with ACN/0.1N HCl and ACN/1N HCl. Roots were subjected to combustion radioanalysis. Dried mature ears were separated into rough (unhulled) rice and ear stems, and the ear stems were combined with the stems. The surface of unhulled grain was rinsed with acetonitrile and then separated into hulls and grain by forceps. The obtained hulls and grain were extracted with ACN/water. Extracts containing enough radioactivity for further analysis (> 0.01 mg/kg eq.) were subjected to TLC analysis followed by radioluminography. Identification of metabolites was confirmed by HPLC and LC-MS.

The distribution of radioactivity in the fractions and the total radioactive residues are shown in Table 17. Significant radioactive residues were only found in leaves and stems. The TRRs in stems and leaves decreased significantly from 2.1 mg/kg eq. at time zero to around one third of the initial value at sampling of immature plants (four weeks after application). At the last sampling, the TRR increased compared to immature samples. The TRRs in grain and hulls were low. The radioactive residues in stems and leaves were almost completely extracted by surface rinse and the extraction procedure.

Table 17 Total radioactive residues (TRR) in rice plants after foliar application with [phthalic acid ring-UL-^{14}C]flubendimide

		Radioactive residue, mg/kg eq. (% of TRR)			
		Seed		Stems and leaves	Root
Sampling, PHI	Fractions	Grain	Hull		
0	Rinsate	N.S.	N.S.	2.036 (95.9)	
	ACN/water extract	N.S.	N.S.	0.088 (4.1)	N.S.
	Post extraction solids	N.S.	N.S.	- (-)	N.S.
	Total radioactive residue	N.S.	N.S.	2.123 (100)	N.S.
4 weeks	Rinsate	0.002 (42.0)		0.512 (82.1)	N.D.
	ACN/water extract	0.002 (44.4)		0.094 (15.2)	N.D.
	ACN/0.1 *N* HCl extracts	- (-)		0.012 (1.9)	N.D.
	ACN/1 *N* HCl extracts	- (-)		0.002 (0.3)	N.D.
	Post extraction solids	0.001 (13.6)		0.003 (0.5)	< 0.001 (100.0)
	Total radioactive residue	0.005 (100.0)		0.623 (100.0)	< 0.001 (100.0)
9 weeks (fresh samples)	Rinsate	0.004 (18.2)		1.026 (72.8)	N.D.
	ACN/water extract	N.D.		0.319 (22.7)	N.D.
	ACN /0.1 *N* HCl extracts	N.D.		0.046 (3.3)	N.D.
	ACN /1 *N* HCl extracts	N.D.		0.006 (0.4)	N.D.
	Post extraction solids	0.019 (81.8)		0.011 (0.8)	0.001 (100.0)
	Total radioactive residue	0.023 (100.0)		1.408 (100.0)	0.001 (100.0)
9 weeks after drying	Rinsate	N.D.	0.015 (33.3)	2.103 (69.2)	N.D.
	ACN/water extract	< 0.001 (19.3)	0.036 (62.3)	0.625 (20.6)	N.D.
	ACN /0.1 *N* HCl extracts	N.D.	N.D.	0.161 (5.3)	N.D.
	ACN /1 *N* HCl extracts	N.D.	N.D.	0.093 (3.0)	N.D.
	Post extraction solids	0.001 (80.7)	0.002 (4.4)	0.057 (1.9)	0.008 (100.0)
	Total radioactive residue	0.001 (100.0)	0.052 (100.0)	3.038 (100.0)	0.008 (100.0)

N.S. = No sample was available.

- = Not detected.

N.D. = Not determined.

The metabolites identified in rice samples are summarised in Table 18. Flubendiamide was the predominant constituent of the residue in all matrices and for all sampling times. Flubendiamide-des-iodo accounted for 4.1% TRR in leaves and stems and fubendiamide-3-OH was identified as a minor constituent. Two metabolites formed by oxidation were also identified in leaves and stems: flubendiamide-benzylalcohol and flubendiamide-benzoic acid.

Table 18 Parent compound and metabolites of [phthalic acid ring-UL-^{14}C]flubendiamide in rice

Metabolites	Concentrations as mg/kg eq. (% of TRR)				
	O day PHI	4 weeks PHI	9 weeks PHI	9 weeks PHI, after drying	
	Stem and leaves	Stem and leaves (immature)	Stem and leaves (mature)	Stem and leaves (straw, dried)	Hulls
Flubendiamide	2.12 (100.0)	0.596 (95.7)	1.34 (95.4)	2.82 (92.9)	0.046 (88.8)
Flubendiamide -des-iodo	-	0.019 (3.1)	0.041 (2.9)	0.126 (4.1)	0.002 (4.0)
Flubendiamide -3-OH	-	0.002 (0.3)	0.007 (0.5)	0.014 (0.5)	-
Flubendiamide -benzyl alcohol	-	0.001 (0.1)	0.004 (0.3)	0.014 (0.5)	-
Flubendiamide -benzoic acid	-	-	0.001 (0.1)	0.004 (0.1)	-
Others unknowns [a)]	-	0.002 (0.3)	-	-	0.002 (3.4)
Post extraction solid (PES)	-	0.003 (0.5)	0.011 (0.8)	0.057 (1.9)	0.002 (3.8)
Total (TRR)	2.12 (100.0)	0.623 (100.0)	1.41 (100.0)	3.04 (100.0)	0.052 (100.0)

[a] Other unknowns included extractable fractions that were not further analysed, or residues in aqueous phases obtained by partitioning prior to TLC analysis

- = Not detected.

From the results of the metabolism studies, it was concluded that the metabolism of flubendiamide after foliar application on plants involved mostly two different routes, shown in Figure 4. The first route was the des-iodination of the parent compound to yield flubendiamide-des-iodo followed by hydroxylation to flubendiamide-3-OH. Another route involved the stepwise oxidation of the methyl group at the aniline ring leading to flubendiamide-benzylalcohol and flubendiamide-benzoic acid. While oxidation of the methyl group is most likely due to enzymatic processes, the des-iodination and the consequent hydroxylation of the phthalic acid ring are known from both, photolytic and biotic degradation processes.

In tomato, the label-specific metabolite flubendiamide-des-anilino (A-13) was also observed indicating that a small amount of the parent compound was cleaved (Figure 5). In apple fruits, a third route was also observed, involving the elimination of the amino-ethyl-sulfonyl substituent leading to flubendiamide-iodo-phthalimide. The label-specific metabolite flubendiamide-3-iodo-phthalic acid was also observed indicating that the parent compound was cleaved in or on leaves of apple trees (Figure 5). In corn, the only metabolic reaction observed was the reductive deiodination to yield flubendiamide-des-iodo. Additionally, small amounts of bound residues were observed.

Environmental Fate

The supported uses of flubendiamide concern foliar application only. Based onthe 'FAO Manual on Submission and Evaluation of Pesticide Residue Data for the Estimation ofMaximum Residue Levels in Food and Feed', 2009, no studies on the fate and behavior in soil arerequired for this type of use. Any metabolite from a field dissipation study that may have an impact on plant residues is covered by the rotational crop study.

Hydrolysis

Flubendiamide comprised more than 95% of the residue at 25 ± 1 °C in pH 4.0, 5.0, 7.0 and 9.0 buffer solutions over a 30-day study period; and more than 95% of the residue at 50.0 ± 0.1 °C in pH 4.0, 7.0 and 9.0 buffer solutions over a 5-day study period (A.Yamashita, 2003). Therefore flubendiamide is hydrolytically stable from pH 4.0 to 9.0.

Photolysis

Flubendiamide was irradiated in distilled water, natural water, and distilled water containing 1% acetone with artificial light for up to 168 hours. An average half-life of 5.5 days was determined in distilled water and distilled water with acetone, while a half-life of 4.3 days was reported in natural waters. The results of the environmental fate studies indicate that degradation of flubendiamide is more likely to occur by photolysis than hydrolysis.

Residues in succeeding crops

The metabolism of flubendiamide after spray application onto bare soil was investigated in three different crops – spring wheat, Swiss chard, and turnips (Reiner, 2004). [Phthalic acid ring-UL-^{14}C]flubendiamide was applied uniformly to the soil of a planting container (area 1 m²) by spray application (day 0). The application rate corresponded to 0.437 kg ai/ha and was based on the projected annual field rate of 0.420 kg ai/ha. Crops of the first, second and third rotation were sown at day 29, day 135 and day 274, respectively. Plants of the first rotation were grown under natural temperature and light conditions and for the second and third rotation, in the greenhouse. One row of wheat plants was cut shortly above the soil surface to sample the forage and one row was cut shortly above the soil surface at the soft dough stage to sample hay. Grain and straw were harvested at maturity. The seeds were collected by hand yielding the grain sample and the remaining ears and chaff were combined with the straw sample. Swiss chard of each rotation was sampled at maturity. Only the leaves were cut above the soil surface. The turnips of each rotation were sampled at maturity. Leaves and roots were separated.

Samples were homogenised with liquid nitrogen, an aliquot extracted with ACN/water and the radioactivity determined by LSC. The undissolved residue was dried at room temperature yielding solids 1. Aliquots were combusted and the radioactivity measured by LSC. For further analysis, the combined ACN/water extracts were concentrated and the radioactivity partitioned with dichloromethane. The total radioactive residues (TRR) in rotational crops, expressed as mg/kg parent compound equivalents, are shown in Table 19. The maximum TRR (0.070 mg/kg) was observed in wheat straw of the first rotation, which decreased in the second rotation and the third rotation.

Table 19 Total radioactive residues (TRRs) in rotational crops grown in soil treated with [phthalic acid ring-UL-^{14}C]-flubenidamide. Values are expressed as mg/kg parent compound equivalents

	wheat				Swiss	Turnips	
	forage	hay	straw	grain	chard	leaves	roots
first rotation	0.013	0.045	0.070	0.003	0.022	0.011	0.006
second rotation	0.008	0.032	0.063	0.002	0.019	0.005	0.002
third rotation	0.016	0.022	0.050	0.003	0.015	0.006	0.002

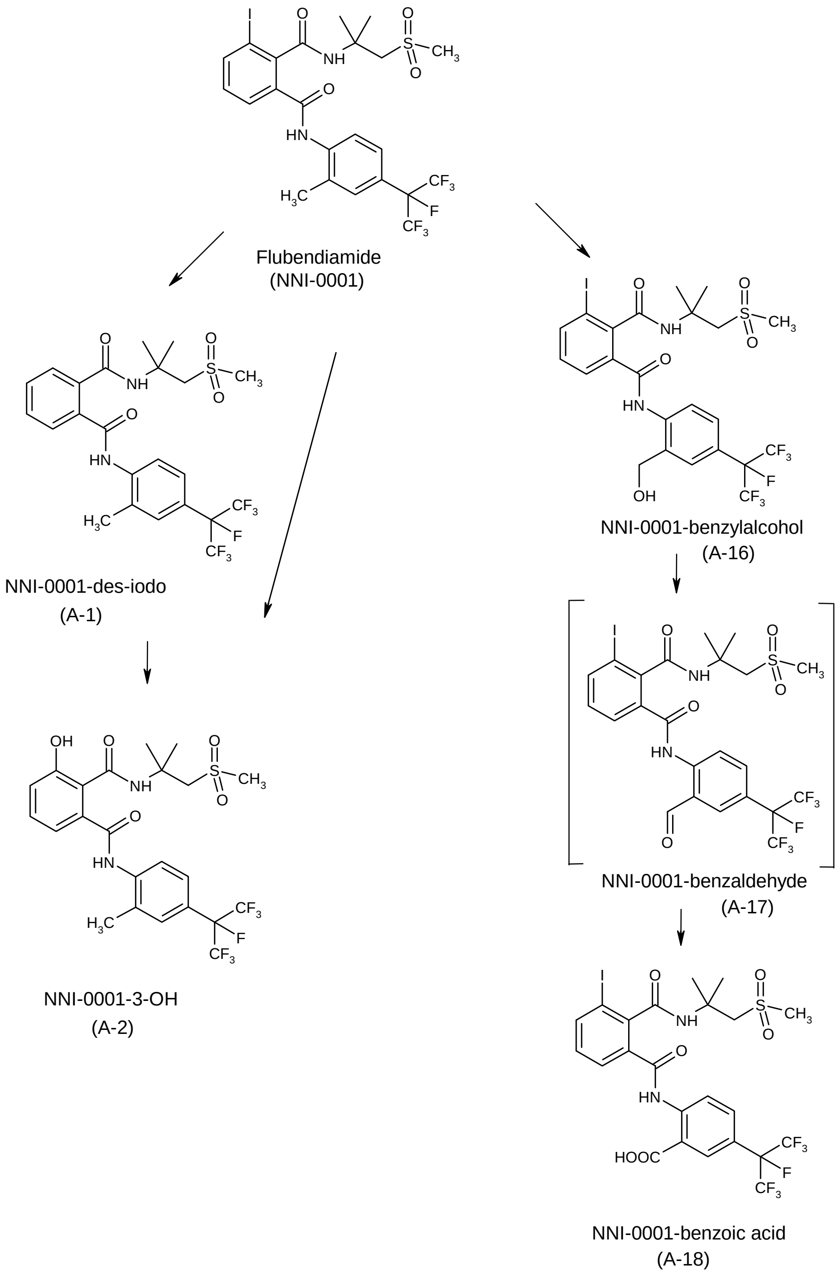
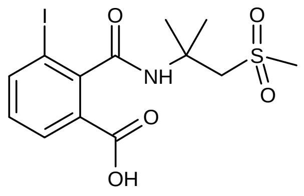
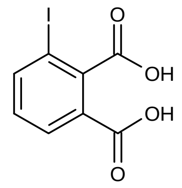


Figure 4 Proposed metabolic pathway of flubendiamide in plants [ ] = postulated intermediate



NNI-0001-des-anilino

(A-13)



NNI-0001-3-iodo-phthalic acid

(A-21)

or similar metabolites

Figure 5 Metabolites found in tomato (A13) and apple (A-21) treated with flubendiamide (NNI-0001)

About 80–90% of the TRR was extracted from the majority of samples using acetonitrile/water. In grain, only a relatively small portion of the TRR was extracted using acetonitrile/water, with the solids 1 (undissolved residue following conventional extraction) of the first rotation accounting for 69.3% TRR. After enzymatic treatment with diastase, 16.2% TRR (< 0.001 mg/kg) remained undissolved in solids.

Tables 20 to 26 show the components of all plant samples treated with [phthalic acid ring-UL-^{14}C] flubendiamide. Unchanged parent compound was the main component of all plant samples and accounted for 22-77% of the TRR, except for grain. In grain, only 4% (< 0.001 mg/kg) of the TRR (0.003 mg/kg) was due to unchanged flubendiamide in the first rotation. The amount of flubendiamide decreased to 2.2% in the second rotation and 0.5% in the third rotation. The main portion of the TRR in grain was due to very polar radioactivity found in aqueous phases following conventional and enzymatic extraction. It was concluded, that approximately 62% of the TRR in grain of the first rotation consisted of natural compounds, formed after mineralisation of flubendiamide residues to $^{14}CO_2$ in soil. This was supported by comparison of the TRRs of grain of all three rotations (0.003 mg/kg, 0.002 mg/kg, 0.003 mg/kg, respectively), by the constant decrease of flubendiamide, by the constant decrease of metabolites flubendiamide-des-iodo and flubendiamide-des-iodo-alkylphthalimide, and by the relative amount of radioactivity in organic/aqueous phases.

A major metabolite in confined rotational crops was flubendiamide-des-iodo, accounting for up to 8.0% of the TRR in Swiss chard of the second rotation. The highest absolute amount of flubendiamide-des-iodo-alkylphthalimide was 0.010 mg/kg in straw of the second rotation corresponding to 16.0% of the TRR. Further identified metabolites were metabolite flubendiamide-benzyl alcohol, flubendiamide-benzoic acid, flubendiamide-iodo-alkylphthalimide) and flubendiamide-des-anilino), each of them < 0.01 mg/kg in all crops of all rotations.

Table 20 Amount of parent compound and metabolites in wheat forage grown in soil treated with [phthalic acid ring-UL-^{14}C] flubendiamide

	first rotation		second rotation		third rotation	
code (report name)	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Flubendiamide	65.3	0.008	77.4	0.006	67.2	0.011
flubendiamide -des-iodo	1.9	< 0.001	2.8	< 0.001	4.3	0.001
flubendiamide -benzyl alcohol	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
flubendiamide -benzoic acid	1.4	< 0.001	0.4	< 0.001	0.7	< 0.001
flubendiamide -iodo-alkylphthalimide	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
flubendiamide -des-anilino	n.d.	n.d.	n.a.	n.a.	n.d.	n.d.
flubendiamide -des-iodo-alkylphthalimide	2.3	< 0.001	6.8	0.001	3.4	0.001
metabolites in the dichloromethane phase	8.1	0.001	2.1	< 0.001	3.4	0.001
metabolites in the aqueous phase	11.3	0.001	3.6	< 0.001	16.4	0.003

	first rotation		second rotation		third rotation	
code (report name)	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
total extracted	90.2	0.012	93.1	0.007	95.4	0.015
solids 1	9.8	0.001	6.9	0.001	4.6	0.001

n.a. = not analysed; n.d. = not detected

Table 21 Amount of parent compound and metabolites in wheat hay grown in soil treated with [phthalic acid ring-UL-^{14}C] flubendiamide

	first rotation		second rotation		third rotation	
Compound	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Flubendiamide	59.6	0.027	64.0	0.020	53.9	0.012
flubendiamide -des-iodo	2.3	0.001	2.5	0.001	2.9	0.001
flubendiamide -benzyl alcohol	0.4	< 0.001	n.d.	n.d.	0.5	< 0.001
flubendiamide -benzoic acid	0.9	< 0.001	0.6	< 0.001	1.0	< 0.001
flubendiamide -iodo-alkylphthalimide	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
flubendiamide -des-anilino	1.3	0.001	n.d.	n.d.	n.d.	n.d.
flubendiamide -des-iodo-alkylphthalimide	8.4	0.004	14.9	0.005	7.1	0.002
metabolites in the dichloromethane phase	5.8	0.003	3.1	0.001	4.3	0.001
metabolites in the aqueous phase	11.6	0.005	6.8	0.002	13.4	0.003
total extracted	90.4	0.041	91.9	0.029	83.2	0.018
solids 1	9.6	0.004	8.1	0.003	16.8	0.004

n.d. = not detected;

Table 22 Amount of parent compound and metabolites in wheat straw grown in soil treated with [phthalic acid ring-UL-^{14}C] flubendiamide

	first rotation		second rotation		third rotation	
Compound	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Flubendiamide	38.9	0.027	60.5	0.038	55.1	0.027
flubendiamide -des-iodo	2.6	0.002	2.2	0.001	3.3	0.002
flubendiamide -benzyl alcohol	0.5	< 0.001	0.5	< 0.001	1.2	0.001
flubendiamide -benzoic acid	0.8	0.001	0.6	< 0.001	0.8	< 0.001
flubendiamide -iodo-alkylphthalimide	0.8	0.001	0.3	< 0.001	n.d.	n.d.
flubendiamide -des-anilino	7.0	0.005	0.7	< 0.001	n.d.	n.d.
flubendiamide -des-iodo-alkylphthalimide	5.9	0.004	16.0	0.010	9.6	0.005
metabolites in the dichloromethane phase	4.7	0.003	3.7	0.002	5.1	0.003
metabolites in the aqueous phase	23.1	0.016	6.6	0.004	9.9	0.005
total extracted	84.3	0.059	91.1	0.058	85.0	0.042
solids 1	15.7	0.011	8.9	0.006	15.0	0.007

Table 23 Amount of parent compound and metabolites in wheat grain grown in soil treated with [phthalic acid ring-UL-^{14}C] flubendiamide

	first rotation		second rotation		third rotation	
code (report name)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Flubendiamide	4.0	< 0.001	2.2	< 0.001	0.5	< 0.001
flubendiamide -des-iodo	0.5	< 0.001	0.5	< 0.001	0.2	< 0.001
flubendiamide -benzyl alcohol	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
flubendiamide -benzoic acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
flubendiamide -iodo-alkylphthalimide	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
flubendiamide -des-anilino	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
flubendiamide -des-iodo-alkylphthalimide	3.0	< 0.001	2.6	< 0.001	0.6	< 0.001

	first rotation		second rotation		third rotation	
code (report name)	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
metabolites in the dichloromethane phase	11.5	< 0.001	3.9	< 0.001	5.4	< 0.001
metabolites in the aqueous phase	11.7	< 0.001	11.0	< 0.001	6.7	< 0.001
in the ethyl acetate phase (only first rotation)	2.4	< 0.001	-	-	-	-
in the aqueous phase 2 (only first rotation)	50.7	0.001	-	-	-	-
total extracted	83.8	0.003	20.3	< 0.001	13.4	< 0.001
solids 1 (following conventional extraction)	(69.3)	(0.002)	79.7	0.001	86.6	0.003
solids 2 (following diastase treatment)	16.2	< 0.001	-	-	-	-

n.a. = not analysed; n.d. = not detected

- = phase not available / experiment not conducted

Table 24 Amount of parent compound and metabolites in Swiss chard grown in soil treated with [phthalic acid ring-UL-^{14}C] flubendiamide

	first rotation		second rotation		third rotation	
Compound	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
flubendiamide	69.1	0.015	73.2	0.014	53.8	0.008
flubendiamide -des-iodo	2.2	< 0.001	8.0	0.002	5.4	0.001
flubendiamide -benzyl alcohol	0.8	< 0.001	0.5	< 0.001	n.d.	n.d.
flubendiamide -benzoic acid	0.4	< 0.001	0.5	< 0.001	0.3	< 0.001
flubendiamide -iodo-alkylphthalimide	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
flubendiamide -des-anilino	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
flubendiamide -des-iodo-alkylphthalimide	2.0	< 0.001	5.4	0.001	12.7	0.002
metabolites in the dichloromethane phase	4.0	0.001	2.4	< 0.001	1.9	< 0.001
metabolites in the aqueous phase	13.8	0.003	7.5	0.001	22.1	0.003
total extracted	92.2	0.020	97.4	0.019	96.1	0.014
solids 1	7.8	0.002	2.6	< 0.001	3.9	0.001

n.d. = not detected

Table 25 Amount of parent compound and metabolites in turnip leaves grown in soil treated with [phthalic acid ring-UL-^{14}C] flubendiamide

	first rotation		second rotation		third rotation	
Compound	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
flubendiamide	68.1	0.008	64.2	0.003	22.4	0.001
flubendiamide -des-iodo	2.8	< 0.001	3.2	< 0.001	2.0	< 0.001
flubendiamide -benzyl alcohol	1.1	< 0.001	n.d.	n.d.	n.d.	n.d.
flubendiamide -benzoic acid	0.6	< 0.001	0.3	< 0.001	n.d.	n.d.
flubendiamide -iodo-alkylphthalimide	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
flubendiamide -des-anilino	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.
flubendiamide -des-iodo-alkylphthalimide	6.4	0.001	16.2	0.001	20.7	0.001
metabolites in the dichloromethane phase	1.5	< 0.001	1.9	< 0.001	23.6	0.001
metabolites in the aqueous phase	8.2	0.001	7.3	< 0.001	23.7	0.001
sum characterised	9.7	0.001	9.2	< 0.001	47.3	0.003
total extracted	88.7	0.010	93.0	0.005	92.4	0.006
solids 1	11.3	0.001	7.0	< 0.001	7.6	< 0.001

n.a. = not analysed; n.d. = not detected

Table 26 Amount of parent compound and metabolites in turnip roots grown in soil treated with [phthalic acid ring-UL-^{14}C] flubendiamide

Compound	first rotation		second rotation		third rotation	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Flubendiamide	69.0	0.004	55.0	0.001	32.6	0.001
flubendiamide des-iodo	0.8	< 0.001	2.1	< 0.001	1.5	< 0.001
flubendiamide -benzyl alcohol	0.7	< 0.001	n.d.	n.d.	n.d.	n.d.
flubendiamide -benzoic acid	0.3	< 0.001	0.6	< 0.001	n.d.	n.d.
flubendiamide -iodo-alkylphthalimide	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
flubendiamide -des-anilino	n.d.	n.d.	n.a.	n.a.	n.a.	n.a.
flubendiamide -des-iodo-alkylphthalimide	1.2	< 0.001	2.8	< 0.001	3.4	< 0.001
metabolites in the dichloromethane phase	0.6	< 0.001	0.2	< 0.001	13.0	< 0.001
metabolites in the aqueous phase	14.1	0.001	29.9	0.001	37.6	0.001
total extracted	86.7	0.005	90.6	0.002	88.1	0.002
solids 1	13.3	0.001	9.4	< 0.001	11.9	< 0.001

n.a. = not analysed; n.d. = not detected

The same experiment was repeated using [aniline ring-UL-^{14}C]flubendiamide at the application rate corresponding to 0.442 kg ai/ha (Reiner, 2004). Crops of the first, second and third rotation were sown at day 29, day 135 and day 274, respectively. Plants of the first rotation were grown under natural temperature and light conditions, and for the second and third rotation, in the greenhouse. Sampling and sample analysis were performed as previously described for the phthalic acid label experiment.

The TRRs were relatively low for all crops and all rotations. The maximum TRR (0.137 mg/kg) was observed in wheat straw of the first rotation, decreasing in the second rotation (0.068 mg/kg) and in the third rotation (0.039 mg/kg). Similarly, the TRRs in wheat hay decreased from 0.045 mg/kg (first rotation) to 0.034 mg/kg (second rotation) and 0.021 mg/kg (third rotation). The TRRs in forage and Swiss chard were on a very low level ranging from 0.009 mg/kg to 0.019 mg/kg for all rotations. The lowest residues were present in grain, turnip leaves and turnip roots amounting to ≤ 0.006 mg/kg for all rotations.

The major amount of radioactivity (ca. 80–90% of the TRR) was extracted for the majority of samples using ACN/water. The solids 1 of grain of the first rotation, accounted for 62.0% TRR. After enzymatic treatment, 14.2% TRR (< 0.001 mg/kg) remained undissolved. Unchanged parent compound was the main component of all plant samples and accounted for 44–88% TRR, except for grain. In grain, only 8% TRR (< 0.001 mg/kg) was due to flubendiamide in the first rotation and was not detectable in grain of the third rotation. The main portion of the TRR in grain was due to very polar radioactivity found in aqueous phases following conventional and enzymatic extraction. It was concluded, that approximately 74% of the TRR in grain of the first rotation consisted of natural compounds, formed after mineralisation of flubendiamide residues to $^{14}CO_2$ in soil.

Tables 27 to 33 show the components of all plant samples treated with [aniline ring-UL-^{14}C] flubendiamide. The main metabolite in confined rotational crops was flubendiamide-des-iodo, accounting for 10.8% TRR in Swiss chard of the second rotation. Flubendiamide-benzyl alcohol and -benzoic acid were detected in some of the plant samples up to 1.4% TRR, each accounting for 0.001 mg/kg as a maximum. Special investigations conducted for straw proved that flubendiamide-aniline was not present as a metabolite.

Table 27 Amount of parent compound and metabolites in wheat forage grown in soil treated with [aniline ring-UL-^{14}C]flubendiamide

	first rotation		second rotation		third rotation	
Compound	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Flubendiamide	72.7	0.008	88.0	0.009	59.4	0.008
Flubendiamide -des-iodo	1.4	< 0.001	3.0	< 0.001	4.3	0.001
Flubendiamide-benzyl alcohol	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Flubendiamide -benzoic acid	1.0	< 0.001	0.5	< 0.001	0.7	< 0.001
metabolites in the dichloromethane phase	14.2	0.002	c.i.	c.i.	1.0	< 0.001
metabolites in the aqueous phase	3.8	< 0.001	3.5	< 0.001	30.1	0.004
total extracted	93.0	0.010	95.0	0.010	95.4	0.012
solids 1	7.0	0.001	5.0	0.001	4.6	0.001

c.i. = complete identification was achieved;

n.d. = not detected

Table 28 Amount of parent compound and metabolites in wheat hay grown in soil treated with [aniline ring-UL-^{14}C] flubendiamide

	first rotation		second rotation		third rotation	
Compound	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Flubendiamide	78.4	0.035	85.5	0.029	44.4	0.009
Flubendiamide -des-iodo	2.4	0.001	2.9	0.001	2.7	0.001
Flubendiamide-benzyl alcohol	0.4	< 0.001	n.d.	n.d.	0.4	< 0.001
Flubendiamide -benzoic acid	1.4	0.001	0.7	< 0.001	0.8	< 0.001
metabolites in the dichloromethane phase	0.8	< 0.001	c.i.	c.i.	3.4	0.001
metabolites in the aqueous phase	7.0	0.003	5.2	0.002	27.6	0.006
total extracted	90.4	0.040	94.4	0.032	79.4	0.016
solids 1	9.6	0.004	5.6	0.002	20.6	0.004

Table 29 Amount of parent compound and metabolites in wheat straw grown in soil treated with [aniline ring-UL-^{14}C]flubendiamide

	first rotation		second rotation		third rotation	
Compound	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Flubendiamide	72.3	0.099	79.9	0.054	46.7	0.018
Flubendiamide -des-iodo	2.4	0.003	2.7	0.002	3.0	0.001
Flubendiamide-benzyl alcohol	0.6	0.001	0.9	0.001	0.9	< 0.001
Flubendiamide -benzoic acid	1.1	0.001	0.9	0.001	1.2	< 0.001
metabolites in the dichloromethane phase	0.8	0.001	2.3	0.002	6.9	0.003
metabolites in the aqueous phase	9.5	0.013	4.3	0.003	19.9	0.008
total extracted	86.6	0.119	91.0	0.062	78.6	0.031
solids 1	13.4	0.018	9.0	0.006	21.4	0.008

Table 30 Amount of parent compound and metabolites in wheat grain grown in soil treated with [aniline ring-UL-^{14}C] flubendiamide

	first rotation		second rotation		third rotation	
Compounds	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Flubendiamide	8.0	< 0.001	2.8	< 0.001	n.d.	n.d.
Flubendiamide -des-iodo	0.2	< 0.001	n.d.	n.d.	n.d.	n.d.
Flubendiamide-benzyl alcohol	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Flubendiamide -benzoic acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

	first rotation		second rotation		third rotation	
Compounds	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
metabolites in the dichloromethane phase	1.0	< 0.001	6.0	< 0.001	3.3	< 0.001
metabolites in the aqueous phase	28.8	0.001	2.7	< 0.001	3.0	< 0.001
in the ethyl acetate phase (only first rotation)	3.0	< 0.001	-	-	-	-
in the aqueous phase 2 (only first rotation)	44.8	0.001	-	-	-	-
solids 1 (following conventional extraction)	(62.0)	(0.001)	88.5	0.002	93.7	0.004
total extracted	85.8	0.002	11.5	< 0.001	6.3	< 0.001
solids 2 (following diastase treatment)	14.2	< 0.001	-	-	-	-

n.d. = not detected

- = phase not available / experiment not conducted

Table 31 Parent compound and metabolites in Swiss chard grown in soil treated with [aniline ring-UL-^{14}C]flubendiamide

	first rotation		second rotation		third rotation	
Compound	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Flubendiamide	62.5	0.008	77.4	0.007	67.6	0.013
Flubendiamide -des-iodo	2.1	< 0.001	10.8	0.001	6.2	0.001
Flubendiamide-benzyl alcohol	0.6	< 0.001	n.d.	n.d.	n.d.	n.d.
Flubendiamide -benzoic acid	0.3	< 0.001	n.d.	n.d.	0.3	< 0.001
metabolites in the dichloromethane phase	6.3	0.001	c.i.	c.i.	c.i.	c.i.
metabolites in the aqueous phase	20.1	0.003	8.8	0.001	22.9	0.004
total extracted	92.0	0.012	96.9	0.009	97.1	0.018
solids 1	8.0	0.001	3.1	< 0.001	2.9	0.001

c.i. = complete identification was achieved

Table 32 Parent compound and metabolites in turnip leaves grown in soil treated with [aniline ring-UL-^{14}C]flubendiamide

	first rotation		second rotation		third rotation	
Compound	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Flubendiamide	64.0	0.002	60.1	0.001	53.5	0.003
Flubendiamide -des-iodo	2.6	< 0.001	6.5	< 0.001	7.6	< 0.001
Flubendiamide-benzyl alcohol	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Flubendiamide -benzoic acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
metabolites in the dichloromethane phase	5.3	< 0.001	c.i.	c.i.	4.9	< 0.001
metabolites in the aqueous phase	16.2	0.001	22.3	< 0.001	25.9	0.002
total extracted	88.1	0.003	88.9	0.002	91.9	0.005
solids 1	11.9	< 0.001	11.1	< 0.001	8.1	< 0.001

c.i. = complete identification was achieved; n.d. = not detected

Table 33 Parent compound and metabolites in turnip roots grown in soil treated with [aniline ring-UL-^{14}C] flubendiamide

	first rotation		second rotation		third rotation	
Compound	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Flubendiamide	66.2	0.001	61.8	0.001	62.1	0.002
Flubendiamide -des-iodo	0.5	< 0.001	2.3	< 0.001	n.d.	n.d.
Flubendiamide-benzyl alcohol	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Flubendiamide -benzoic acid	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
metabolites in the dichloromethane phase	10.0	< 0.001	c.i.	c.i.	12.6	< 0.001

Compound	first rotation		second rotation		third rotation	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
metabolites in the aqueous phase	10.6	< 0.001	23.1	< 0.001	14.3	< 0.001
total extracted	87.2	0.002	87.3	0.001	89.0	0.002
solids 1	12.8	< 0.001	12.7	< 0.001	11.0	< 0.001

The main metabolic reaction of flubendiamide in confined rotational crops was the reduction of the parent compound by elimination of the iodine-substituent. Another important metabolic reaction was the elimination of the N-aryl-moiety. Hydroxylation of the parent compound was also observed, however, only a small amount of metabolite flubendiamide-benzyl alcohol was generally detected. A significant amount was further oxidised to the carboxylic acid, probably in soil. Metabolite flubendiamide-benzoic acid was found in relatively high amounts. The proposed metabolic pathway of flubendiamide is shown is shown in Figure 6.

METHODS OF RESIDUE ANALYSIS

Plant material

The use of the GC multi residue method DFG S19 (Specht et al. 1995) and FDA Multi-Residue Method Test guidelines in PAM (Third Edition, January 1994) was not applicable for the analysis of flubendiamide due to the thermolability of flubendiamide, giving recoveries below 70%.

The analytical method 00816/M001 developed for the determination of flubendiamide and flubendiamide-des-iodo residues in/on plant material by Billian (2004) is the recommended monitoring method for flubendiamide residues. In this method, flubendiamide and the des-iodo metabolite are extracted from the sample material using two successive microwave extractions, the first with acetonitrile/0.01%HCl and the second with acetonitrile/0.01%HCl/water. After evaporation, the extract is cleaned-up using diatomaceous earth columns, the residues eluted with cyclohexane/ethyl acetate, and dissolved in acetonitrile/water for quantification by LC-MS/MS. For the determination of flubendiamide in/on oil of plant origin, the samples are dissolved in hexane, extracted with acetonitrile and partionated with hexane before LC-MS/MS. Two MRM transitions for quantitation and confirmation were monitored for each analyte (flubendiamide: m/z 681→254 and m/z 681→274; flubendiamide-des-iodo: m/z 555→254 and m/z 555→274). As matrix effects were observed, matrix-matched standards were used for quantification of residues. Validation data is shown on Table 34. The limit of quantification (LOQ) for both analytes is 0.01 mg/kg for all sample materials. The limit of detection (LOD) was estimated to be at least 0.001 mg/kg.

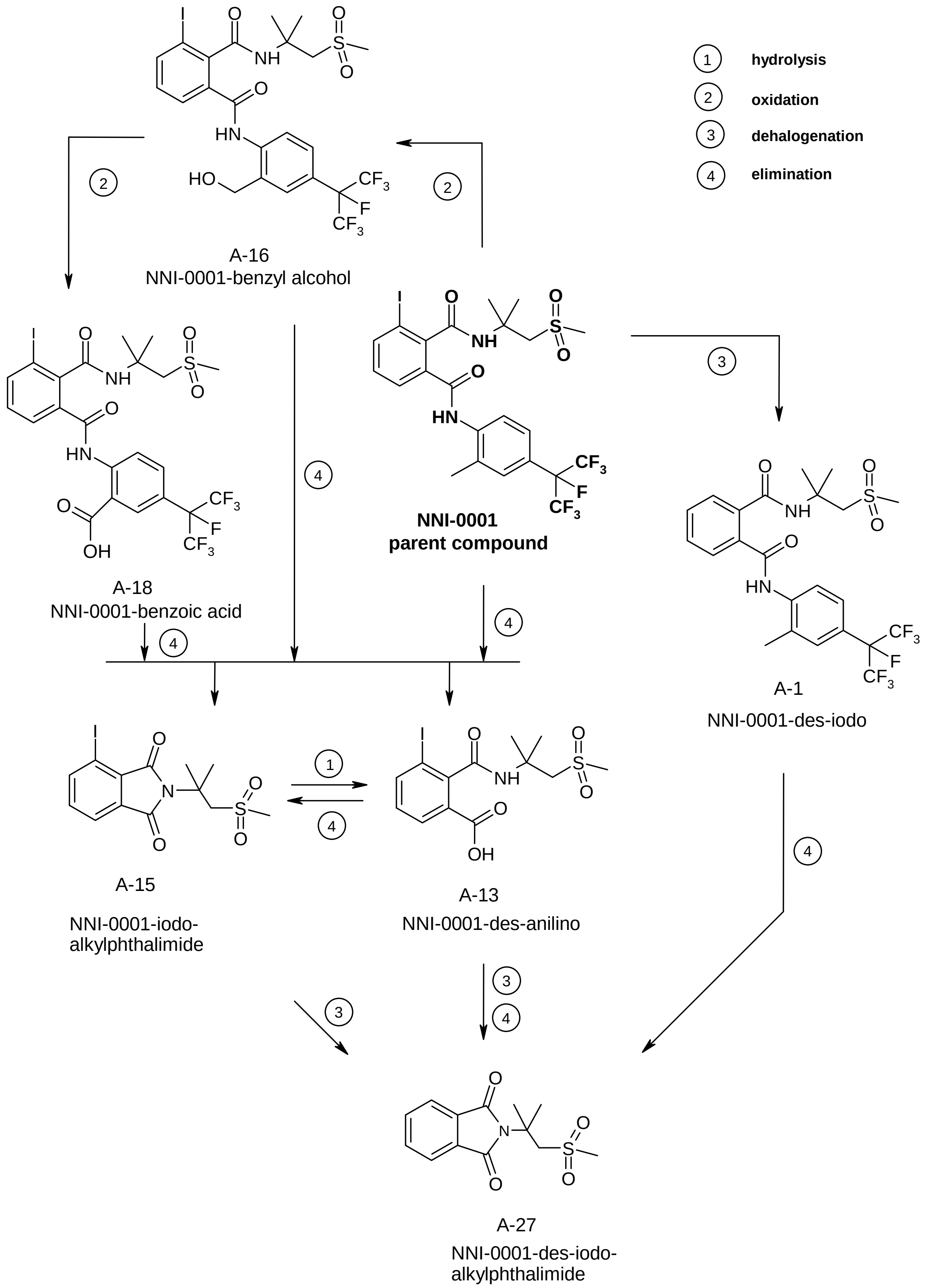


Figure 6 Proposed metabolic pathway of flubendiamide in confined rotational crops

Table 34 Validation data for flubendiamide and flubendiamide-des-iodo in plant material using the method 00816/M001

Analyte	Substrate	Fortification level (mg/kg)	*n*	Recovery		
				Individual values (%)	Mean (%)	RSD (%)
Flubendiamide	Citrus (fruit)	0.01	5	96, 99, 95, 81, 84	91	8.8
		0.10	5	91, 91, 89, 86, 87	89	2.6
		overall	10	-	90	6.3
	Head cabbage (head)	0.01	5	88, 80, 87, 90, 89	87	4.6
		0.10	5	90, 89, 91, 95, 93	92	2.6
		overall	10	-	89	4.5
	Oil (olive, cotton)	0.01	5	95, 94, 93, 87, 84	91	5.3
		0.10	5	98, 95, 97, 92, 86	94	5.1
		overall	10	-	92	5.2
	Bean (with pod)	0.01	3	93, 93, 98	95	3.1
		0.10	3	96, 96, 105	99	5.3
		overall	6	-	97	4.6
	Tomato	0.01	5	103, 90, 89, 86, 89	91	7.3
		0.10	5	96, 93, 96, 96, 93	95	1.7
		overall	10	-	93	5.3
	Wheat (grain)*	0.01	6	80, 92, 93, 99, 82, 77	87	10.0
		0.10	6	70, 87, 78, 89, 92, 71	81	11.7
		overall	12	-	84	11.0
Flubendiamide-des-iodo	Citrus (fruit)	0.01	5	94, 95, 95, 85, 90	92	4.7
		0.10	5	95, 95, 94, 88, 91	93	3.3
		overall	10	-	92	3.9
	Head cabbage (head)	0.01	5	92, 89, 94, 94, 95	93	2.6
		0.10	5	93, 94, 94, 96, 93	94	1.3
		overall	10	-	93	2.0
	Oil (olive, cotton)	0.01	5	97, 94, 96, 86, 86	92	5.9
		0.10	5	98, 95, 97, 91, 85	93	5.7
		overall	10	-	93	5.5
	Bean (with pod)	0.01	3	95, 93, 97	95	2.1
		0.10	3	98, 99, 100	99	1.0
		overall	6	-	97	2.7
	Tomato	0.01	5	106, 89, 88, 86, 86	91	9.0
		0.10	5	98, 95, 95, 97, 93	96	2.0
		overall	10	-	94	6.5
	Wheat (grain)	0.01	7	74, 81,82, 84,83,81,75	80	4.9
		0.10	7	66, 82,78, 72,81,85,70	76	9.3
		overall	14	-	78	7.4

* One recovery sample at each fortification level was excluded.

An independent laboratory validation (ILV) of the method 00816/M001 was conducted by Class (2005). The results are summarised in Table 35.

Table 35 ILV data for flubendiamide and flubendiamide-des-iodo in plants using method 00816/M001

Analyte	Substrate	Fortification level (mg/kg)	Number of replicates	Mean recovery (%)	RSD (%)
Flubendiamide *	Tomato	0.01	5	98	9
		0.10	5	104	3
		overall	10	101	7
	Wheat grain	0.01	5	82	2
		0.10	5	80	4
		overall	10	81	3
	Head cabbage	0.01	5	81	9
		0.10	5	83	6
		overall	10	82	7
	Cotton seed oil	0.01	5	101	2
		0.10	5	101	2
		overall	10	101	2

Analyte	Substrate	Fortification level (mg/kg)	Number of replicates	Mean recovery (%)	RSD (%)
Flubendiamide-des-iodo**	Tomato	0.01	5	86	9
		0.10	5	87	2
		overall	10	87	6
	Wheat grain	0.01	5	91	2
		0.10	5	81	5
		overall	10	86	7
	Head cabbage	0.01	5	83	6
		0.10	5	79	9
		overall	10	81	8
	Cotton seed oil	0.01	5	99	5
		0.10	5	101	3
		overall	10	100	4

* Only product Ions Q3 Mass (amu) of 274 m/z is presented;

** Only product Ions Q3 Mass (amu) of 148 m/z is presented

Method 00816/M002 is a modification of the method 00816/M001, and used internal –d6 -labelled standards for quantification (Billian, 2007). LOQ for flubendiamide and flubendiamide-des-iodo is 0.01 mg/kg for all substrates. The Limit of Detection (LOD) was estimated to be at least 0.001 mg/kg. Two MRM transitions for quantitation and confirmation were monitored for each analyte (flubendiamide: m/z 681→254 and m/z 681→274; flubendiamide-des-iodo: m/z 555→254 and m/z 555→274). The validation results are summarised in Table 36.

Table 36 Validation data for flubendiamide and flubendiamide-des-iodo in plant materials using the method 00816/M002

Analyte	Substrate	Fortification level (mg/kg)	Number of replicates	Recovery		
				Individual values (%)	Mean (%)	RSD (%)
flubendiamide *Quantitation* *m/z 681→254*	Citrus (fruit)	0.01	5	85, 80, 90, 86, 84	85	4.2
		0.10	5	87, 87, 81, 85, 87	85	3.1
		overall	10	-	85	3.5
	Head cabbage (head)	0.01	3	84, 83, 79	82	3.2
		0.10	3	87, 87, 85	86	1.3
		overall	6	-	84	3.6
	Cotton (oil)	0.01	5	98, 97, 96, 95, 94	96	1.6
		0.10	5	101, 100, 98, 92, 97	98	3.6
		overall	10	-	97	2.8
	Bean (with pod)	0.01	3	89, 72, 74	78	11.9
		0.10	3	77, 73, 84	78	7.1
		overall	6	-	78	8.8
	Tomato	0.01	5	78, 91, 88, 96, 89	88	7.5
		0.10	5	85, 90, 88, 89, 89	88	2.2
		overall	10	-	88	5.2
	Wheat (grain)	0.01	5	80, 83, 82, 74, 85	81	5.2
		0.10	5	86, 81, 82, 81, 79	82	3.2
		overall	10	-	81	4.1
flubendiamide - des-iodo *Quantitation* *m/z 555→254*	Citrus (fruit)	0.01	5	88, 92, 91, 87, 89	89	2.3
		0.10	5	87, 89, 86, 89, 87	88	1.7
		overall	10	-	89	2.6
	Head cabbage (head)	0.01	3	85, 87, 89	87	2.3
		0.10	3	93, 89, 91	91	2.2
		overall	6	-	89	3.2
	Cotton (oil)	0.01	5	101, 103, 101, 102, 97	101	2.3
		0.10	5	99, 100, 99, 99, 100	99	0.6
		overall	10	-	100	1.7
	Bean (with pod)	0.01	3	86, 84, 78	83	5.0
		0.10	3	89, 84, 90	88	3.7
		overall	6	-	85	5.1
	Tomato	0.01	5	79, 92, 92, 92, 91	89	6.4
		0.10	5	84, 89, 92, 88, 90	89	3.3
		overall	10	-	89	4.8

Analyte	Substrate	Fortification level (mg/kg)	Number of replicates	Recovery		
				Individual values (%)	Mean (%)	RSD (%)
	Wheat (grain)	0.01	5	84, 81, 85, 77, 86	83	4.4
		0.10	5	83, 76, 79, 85, 72	79	6.6
		overall	10	-	81	5.8

An independent laboratory validation (ILV) of the method 00816/M002 was conducted by Rotzoll (2007). The results are summarised in Table 37.

Table 37 ILV data for flubendiamide and flubendiamide -des-iodo using the method 00816/M002

Analyte	Substrate	Fortification level (mg/kg)	*n*	Recovery		
				Individual values (%)	Mean (%)	RSD (%)
Flubendiamide *Quantitation* *m/z 681→254*	Wheat (Grain)	0.01	5	96, 99, 98, 101, 95	98	2.4
		0.10	5	92, 87, 94, 95, 95	93	3.7
		overall	10	-	95	4.1
	Oilseed Rape (Seeds)	0.01	5	100, 99, 101, 94, 101	99	2.9
		0.10	5	1.01, 101, 97, 101, 98	100	1.9
		overall	10	-	99	2.4
	Orange (Fruit)	0.01	5	87, 89, 89, 89, 91	89	1.6
		0.10	5	98, 96, 91, 95, 95	95	2.6
		overall	10	-	92	4.0
	Tomato (Fruit)	0.01	5	91, 93, 93, 92, 94	93	1.2
		0.10	5	100, 100, 100, 102, 103	101	1.4
		overall	10	-	97	4.7
	Bean (Seeds)	0.01	5	90, 90, 90, 93, 93	91	1.8
		0.10	5	93, 94, 96, 98, 100	96	3.0
		overall	10	-	94	3.6
Flubendiamide *Confirmation* *m/z 681→274*	Wheat (Grain)	0.01	5	98, 99, 96, 100, 94	97	2.5
		0.10	5	95, 89, 94, 97 96	94	3.3
		overall	10	-	96	3.2
	Oilseed Rape (Seeds)	0.01	5	99, 99, 98, 96, 98	98	1.2
		0.10	5	101, 100, 99, 102, 98	100	1.6
		overall	10	-	99	1.7
	Orange (Fruit)	0.01	5	86, 87 87 89, 88	87	1.3
		0.10	5	97 94, 90, 96, 93	94	2.9
		overall	10	-	91	4.4
	Tomato (Fruit)	0.01	5	93, 92, 93, 93, 93	93	0.4
		0.10	5	100, 99, 99, 101, 104	101	2.1
		overall	10	-	97	4.4
	Bean (Seeds)	0.01	5	91, 89, 93, 94, 90	91	2.3
		0.10	5	91, 92, 93, 96, 98	94	3.1
		overall	10	-	93	3.0
flubendiamide -des-iodo *Quantitation* *m/z 555→254*	Wheat (Grain)	0.01	5	82, 87, 89, 88, 88	87	3.2
		0.10	5	89, 84, 85, 88, 89	87	2.6
		overall	10	-	87	2.8
	Oilseed Rape (Seeds)	0.01	5	99, 101, 101, 98, 100	100	1.3
		0.10	5	102, 103, 101, 102, 101	102	0.8
		overall	10	-	101	1.5
	Orange (Fruit)	0.01	5	86, 91, 89, 90, 89	89	2.1
		0.10	5	99, 95, 92, 96, 94	95	2.7
		overall	10	-	92	4.2
	Tomato (Fruit)	0.01	5	95, 96, 93, 94, 95	95	1.2
		0.10	5	101, 100, 100, 102, 104	101	1.7
		overall	10	-	98	3.9
	Bean (Seeds)	0.01	5	95, 90, 98, 94, 93	94	3.1
		0.10	5	95, 100, 98, 102, 103	100	3.2
		overall	10	-	97	4.2
flubendiamide -des-iodo *Confirmation* *m/z 555→274*	Wheat (Grain)	0.01	5	83, 88, 89, 88, 87	87	2.6
		0.10	5	88, 84, 84, 87, 91	87	3.3
		overall	10	-	87	2.9
	Oilseed Rape (Seeds)	0.01	5	97, 101, 102, 98, 100	100	2.1
		0.10	5	101, 101, 99, 100, 99	100	1.0
		overall	10	-	100	1.5

Analyte	Substrate	Fortification level (mg/kg)	*n*	Recovery Individual values (%)	Mean (%)	RSD (%)
	Orange (Fruit)	0.01 0.10 overall	5 5 10	90, 94, 89, 91, 89 97, 95, 93, 96, 94 -	91 95 93	2.3 1.7 3.1
	Tomato (Fruit)	0.01 0.10 overall	5 5 10	95, 96, 95, 92, 95 99, 101, 100, 102, 103 -	95 101 98	1.6 1.6 3.8
	Bean (Seeds)	0.01 0.10 overall	5 5 10	95, 87, 97, 96, 97 95, 100, 98, 103, 100 -	94 94 97	4.5 4.5 4.4

Method 00816/M003 (Ballesteros and Gateaud, 2005) is a modification of method 00816/M002 and does not include clean up step. Flubendiamide and the des-iodo metabolite are extracted from plant matrices with microwave, filtered, the extract diluted and residues are quantified by LC/MS/MS. LOQ for flubendiamide is 0.01 mg/kg for all substrates and LOD was estimated to be at least 0.001 mg/kg for all substrates. The quantification was done by an external standardisation in solvent using d6- flubendiamide and d6- flubendiamide -des-iodo. Validation results are shown on Table 38.

Table 38 Validation data for flubendiamide and flubendiamide -des-iodo in plant material using the method 00816/M003

Substrate	Level (mg/kg)	N	Recovery (%) flubendiamide Individual	Mean	RSD	flubendiamide -des-iodo Individual	Mean	RSD
Tomato	0.01 0.10 overall	5 5 10	102 99 105 100 111 101 101 106 103 101 -	103 102 **103**	4.7 2.1 **3.5**	103 109 97 88 97 104 109 101 101 101 -	99 103 101	7.9 3.4 6.1
Grape (bunch)	0.01 0.10 overall	5 5 10	90 103 95 92 93 100 93 102 99 103 -	95 99 97	5.3 3.9 5.1	93 91 89 93 95 104 103 98 101 97 -	92 101 96	2.5 3.0 5.3

Method ATM-0012 (Radunz, 2008) is another modification of method 00816/M002, where the use of microwave for extraction was replaced by a shaker. A comparison in extraction efficiency between microwave and shaker was evaluated from radiovalidation of method 00816/M002 with corn samples (forage and fodder) (Renier, 2006) and the metabolism study following spray application of [phthalic acid ring-UL-^{14}C]flubendiamide onto corn plants, when a blender was used in the extraction procedure (Krolski and Nguyen, 2005). Before extraction, corn samples were firstly processed with dry ice until finely pulverized. The extraction efficiency of the residues in both cases is presented in Table 39. The results are obtained with corn samples from the metabolism study stored during 20 months (619 days) at -18 °C or below.

Table 39 Extraction efficiency of the residue method 00816/M002 using corn samples (forage and fodder) from the metabolism study

	metabolism study (blending)		method 00816/M002 (microwave)	
	% TRR	mg/kg [a]	% TRR	mg/kg [a,c]
corn forage				
total radioactive residue (TRR)	100	0.288 [b]	100	0.379
flubendiamide	91	0.262 [e]	82.7	0.314
flubendiamide-des-iodo	4	0.012 [e]	1.8	0.007
sum of flubendiamide + des-iodo	95	0.274 [e]	84.5	0.320
extraction efficiency	100%		89% [f]	
corn fodder				
total radioactive residue (TRR)	100	0.476 [c]	100	0.457
flubendiamide	77	0.367 [e]	65.5	0.297
flubendiamide-des-iodo	8	0.038 [e]	5.5	0.025

	metabolism study (blending)		method 00816/M002 (microwave)	
	% TRR	mg/kg ^{a}	% TRR	mg/kg ^{a,c}
sum of flubendiamide + des-iodo	85	0.405 ^{e}	71.0	0.322
extraction efficiency	100%		83% ^{e}	

[a] mg/kg expressed as parent compound equivalents;

[b] determined by combustion; [c] mean results from two parallel extractions;

[e] considering the relative amount of the TRR of the extraction efficiency sample and of the metabolism study

The LOQ of flubendiamide and flubendiamide des-iodo for method ATM-0012 in all tested crops was set at 0.02 mg/kg for each component and 0.045 mg/kg expressed as total flubendiamide parent equivalent. The results of the validation are shown in Table 40.

Table 40 Validation data for flubendiamide and flubendiamide-des-iodo (A-1) in plant material using the method ATM-0012

Substrate	Analyte	Level (mg/kg)	Number of replicates	Recovery (%)			Report No Doc No
				Individual values	Mean	RSD	
Green bean (Pods)	flubendiamide	0.02 0.10	2 2	95–100 95–101	98 98	2 3	BCS-0212.01 M-306208-01-1
	des-iodo	0.02 0.10	2 2	93–96 93–99	94 96	1 3	
Green bean (Pods)	flubendiamide	0.02 0.10	2 2	104–106 101–104	105 103	1 1	BCS-0186.01 M-306326-01-1
	des-iodo	0.02 0.10	2 2	87–99 103–104	93 104	6 0	
Green bean (Stubble)	flubendiamide	0.02 0.10	2 2	102–106 101–105	104 104	2 2	
	des-iodo	0.02 0.10	2 2	89–100 90–101	94 95	5 6	
Green pea (Pods)	flubendiamide	0.02 0.10	2 2	88–99 90–93	93 92	5 1	BCS-0185.01 M-306322-01-1
	des-iodo	0.02 0.10	2 2	87–90 88–89	89 88	2 0	
Green pea (Stubble)	flubendiamide	0.02 0.10	2 2	88–99 83–99	93 91	4 7	
	des-iodo	0.02 0.10	2 2	89–92 80–89	90 84	2 5	
Head lettuce (head)	flubendiamide	0.02 0.10	2 2	92–103 99–102	99 101	5 1	BCS-0187.01 M-306189-01-1
	des-iodo	0.02 0.10	2 2	92–94 95–96	93 95	1 1	
	flubendiamide	0.02 0.10	2 2	87–94 95–99	90 97	3 2	BCS-0188.01 M-306195-01-1
	des-iodo	0.02 0.10	2 2	88–91 93–96	90 94	2 1	
Leafy lettuce (leaves)	flubendiamide	0.02 0.10	2 2	92–98 98–102	94 100	2 2	BCS-0189.01 M-306206-01-1
	des-iodo	0.02 0.10	2 2	88–94 92–97	91 95	2 2	
	flubendiamide	0.02 0.10	2 2	102–105 99–101	103 100	1 1	BCS-0189.01 M-306206-01-1
	des-iodo	0.02 0.10	2 2	91–95 89–94	92 91	2 2	
Head cabbage (head)	flubendiamide	0.02 0.10	2 2	82–98 89–98	90 93	7 4	BCS-0082.01 M-306221-01-1
	des-iodo	0.02 0.10	2 2	85–97 94–99	91 96	6 2	
	flubendiamide	0.02 0.10	2 2	81–95 92–94	89 93	7 1	BCS-0117.01 M-306243-01-1
	des-iodo	0.02	2	96–103	100	3	

Substrate	Analyte	Level (mg/kg)	Number of replicates	Recovery (%)			Report No Doc No
				Individual values	Mean	RSD	
		0.10	2	105–107	107	1	
Brussels sprouts (buttons)	flubendiamide	0.02	2	101–114	107	5	BCS-0081.01 M-306217-01-1
		0.10	2	102–105	104	1	
	des-iodo	0.02	2	92–99	96	3	
		0.10	2	97–99	98	1	
Broccoli (flower heads)	flubendiamide	0.02	2	84–95	89	5	BCS-0083.01 M-306227-01-1
		0.10	2	90–95	93	3	
	des-iodo	0.02	2	83–94	90	5	
		0.10	2	90–98	94	4	
	flubendiamide	0.02	2	94–113	103	11	BCS-0118.01 M-306246-01-1
		0.10	2	92–93	93	0	
	des-iodo	0.02	2	98–114	106	9	
		0.10	2	95–103	100	3	
Sweet Corn (cobs)	flubendiamide	0.02	2	98–103	100	2	BCS-0086.01 M-306237-01-1
		0.10	2	95–97	96	1	
	des-iodo	0.02	2	88–93	91	3	
		0.10	2	94–97	95	1	
	flubendiamide	0.02	2	95–105	101	4	BCS-0118.01 M-306246-01-1
		0.10	2	92–99	95	3	
	des-iodo	0.02	2	105–109	107	2	
		0.10	2	105–108	107	1	
Tomato	flubendiamide	0.02	2	88–92	90	2	BCS-0084.01 M-306271-01-1
		0.10	2	88–91	89	2	
	des-iodo	0.02	2	87–88	88	1	
		0.10	2	86–90	87	2	
	flubendiamide	0.02	2	79–81	79	1	BCS-0108.01 M-306282-01-1
		0.10	2	84–86	85	1	
	des-iodo	0.02	2	106–110	108	2	
		0.10	2	119–121	120	1	
	flubendiamide	0.02	2	91–95	93	2	BCS-0120.01 M-306306-01-1
		0.10	2	78–79	78	1	
	des-iodo	0.02	2	87–102	96	7	
		0.10	2	78–80	78	1	
Pepper	flubendiamide	0.02	2	87–100	93	8	BCS-0085.01 M-306277-01-1
		0.10	2	93–97	95	2	
	des-iodo	0.02	2	94–104	99	6	
		0.10	2	94–99	97	2	
	flubendiamide	0.02	2	90–94	92	2	BCS-0109.01 M-306287-01-1
		0.10	2	94–97	96	2	
	des-iodo	0.02	2	90–95	92	3	
		0.10	2	97–98	97	1	
	flubendiamide	0.02	2	95–103	99	4	BCS-0121.01 M-306312-01-1
		0.10	2	83–94	89	7	
	des-iodo	0.02	2	97–100	98	1	
		0.10	2	81–95	88	9	

A HPLC/UV analytical method was developed for analysing flubendiamide and the des-iodo and 3-OH metabolites in tea samples (Motoba, 2002; M-358559-01-1). The samples were homogenized with ACN/0.1N HCl, extract with n-Hexane/EtOAc and the extracted submitted to a sequential clean up step using graphite carbon, C18 and NH2 cartridge columns. To analyse the flubendiamide-3-OH metabolite, the eluate was dissolved in ACN/water (2/3) and analysed by HPLC/UV at 260 nm using a C18 column. To analyse flubendiamide and the des-iodo metabolite, an addition clean-up step using silica gel cartridge column was included before HPLC/UV. Table 41 shows recovery data of flubendiamide and its metabolites for tea and other crops at 0.1 mg/kg fortification level

Table 41 Flubendiamide and its metabolites in plant material using the Japanese method at 0.1 mg/kg (n = 3)

Substrate	Analyte	Recovery	
		Mean (%)	RSD (%)
Cabbage	Flubendiamide Des-iodo 3-OH	97 96 92	3.2 3.2 4.8
Apple	Flubendiamide Des-iodo 3-OH	98 93 87	1.6 1.2 3.5
Tea	Flubendiamide Des-iodo 3-OH	107 94 90	1.9 3.8 3.8
Rice	Flubendiamide Des-iodo 3-OH	88 93 94	8.1 8.4 6.6

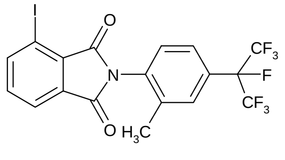
The method was validated for flubendiamide on tea during the residue supervised trial study at levels of 0.01 mg/kg (LOQ), as well as 0.2, 20 and 30 mg/kg (Table 42).

Table 42 Summary of recovery data for flubendiamide in/on tea

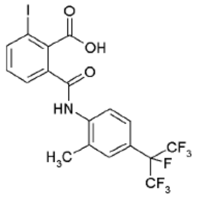
Spike level (mg/kg)	Sample size (n)	Recoveries (%)						Mean (%)	RSD (%)
0.01	18	96	92	88	97	95	91	95	5.8
		96	92	88	106	100	97		
		93	87	86	102	101	96		
0.2	3	88	87	87				87	0.7
20	3	96	96	95				96	0.6
30	12	82	78	76	92	84	83	87	7.6
		96	94	90	94	90	90		

Animal matrices

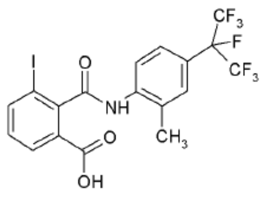
Method 00912 was developed for the determination of flubendiamide and the metabolite flubendiamide-iodophthalimide in muscle, liver, kidney, milk, fat and egg (Billian, 2005). The residues are extracted with acetonitrile/water and flubendiamide-iodophthalimide is completely converted to flubendiamide-des-alkylamino and its isomer under mild alkaline conditions.



flubendiamide-iodophthalimide



flubendiamide-des-alkylamino



flubendiamide-des-alkylaminoisomer

The residues of flubendiamide, flubendiamide-des-alkylamino and its isomer are cleaned-up using diatomaceous earth columns, and analysed by LC-MS/MS. The method uses matrix-matched standards for calibration or analysis using internal standards of flubendiamide-d_6, A-12-d_6, and A-37-d_6 with external standards of the analytes for calibration. In both cases, two ion transitions are monitored for each analyte. Residues of A-12/A-37 are converted to flubendiamide-iodophthalimide equivalents using a molecular weight conversion factor. LOQ for flubendiamide and its metabolite was 0.01 mg/kg. Expressed as parent equivalents (flubendiamide equivalents), the LOQ for flubendiamide-iodophthalimide in animal tissues was 0.013 mg/kg. LOD was estimated to be at least 0.0025 mg/kg for both compounds in all matrices. Two MRM transitions for quantitation and confirmation were monitored for each analyte. The validation data are shown on Table 43.

Table 43 Validation data for flubendiamide and flubendiamide-iodophthalimide in animal matrices using the method 00912 by Internal Standard Procedure

Analyte	Substrate	Fortification level (mg/kg)	*n*	Recovery Rates (%)		
				Individual values	Mean	RSD
Flubendiamide *Quantitation* *m/z 681→254*	Muscle	0.01	5	77, 73, 79, 85, 85	80	6.5
		0.10	5	90, 77, 83, 85, 86	84	5.7
		overall	10	-	82	6.4
	Liver	0.01	5	84, 79, 94, 89, 92	88	7.0
		0.10	5	95, 91, 89, 100, 102	95	5.9
		overall	10	-	91	7.5
	Kidney	0.01	5	75, 76, 84, 71, 77	77	6.2
		0.10	5	81, 80, 81, 81, 79	80	1.1
		overall	10	-	79	4.8
	Fat	0.01	5	81, 81, 85, 83, 81	82	2.2
		0.10	5	84, 94, 89, 99, 94	92	6.2
		overall	10	-	87	7.5
	Milk	0.01	5	79, 69, 83, 86, 76	79	8.5
		0.10	5	91, 83, 87, 90, 85	87	3.7
		overall	10	-	83	8.0
	Poultry egg	0.01	5	93, 98, 89, 91, 98	94	4.4
		0.10	5	90, 83, 80, 93, 83	86	6.4
		overall	10	-	90	6.9
Flubendiamide *Confirmation* *m/z 681 → 274*	Muscle	0.01	5	79, 81, 75, 105, 102	88	15.8
		0.10	5	96, 86, 91, 89, 90	90	4.0
		overall	10	-	89	10.8
	Liver	0.01	5	87, 95, 118, 126, 89	103	17.3
		0.10	5	95, 92, 95, 104, 99	97	4.8
		overall	10	-	100	12.7
	Kidney	0.01	5	73, 80, 82, 71, 78	77	6.1
		0.10	5	83, 81, 81, 86, 85	83	2.7
		overall	10	-	80	6.0
	Fat	0.01	5	73, 83, 86, 79, 86	81	6.8
		0.10	5	86, 92, 89, 100, 94	92	5.8
		overall	10	-	87	8.8
	Milk	0.01	5	80, 64, 92, 76, 90	81	14.1
		0.10	5	87, 83, 84, 94, 89	88	5.0
		overall	10	-	84	10.6
	Poultry egg	0.01	5	77, 79, 92, 91, 79	84	8.7
		0.10	5	85, 98, 87, 96, 87	91	6.6
		overall	10	-	87	8.3
flubendiamide-iodophthalimide * *Quantitation* *m/z 548 → 504*	Muscle	0.01**	5	87, 77, 78, 83, 76	80	5.8
		0.10***	5	88, 81, 85, 87, 85	85	3.1
		overall	10	-	83	5.4
	Liver	0.01**	5	86, 85, 87, 89, 86	87	1.8
		0.10***	5	96, 95, 97, 95, 95	96	0.9
		overall	10	-	91	5.4
	Kidney	0.01**	5	84, 80, 86, 83, 79	82	3.5
		0.10***	5	83, 87, 85, 88, 84	85	2.4
		overall	10	-	84	3.4
	Fat	0.01**	3	79, 82, 85, 89, 80	83	4.9
		0.10***	3	81, 86, 84, 88, 82	84	3.4
		overall	6	-	84	4.0
	Milk	0.01**	5	82, 78, 78, 83, 83	81	3.4
		0.10***	5	89, 87, 88, 89, 89	88	0.9
		overall	10	-	85	5.2
	Poultry egg	0.01**	6	81, 86, 89, 84, 77	83	5.5
		0.10***	6	89, 87, 90, 89, 84	88	2.7
		overall	12	-	86	4.9

Analyte	Substrate	Fortification level (mg/kg)	*n*	Recovery Rates (%)		
				Individual values	Mean	RSD
flubendiamide-iodophthalimide * *Confirmation* *m/z 548 → 127*	Muscle	0.01**	5	83, 76, 85, 83, 88	83	5.3
		0.10***	5	90, 83, 84, 85, 83	85	3.4
		overall	10	-	84	4.4
	Liver	0.01**	5	80, 85, 82, 87, 80	83	3.8
		0.10***	5	95, 96, 98, 95, 96	96	1.3
		overall	10	-	89	8.2
	Kidney	0.01**	5	78, 84, 91, 85, 89	85	5.9
		0.10***	5	77, 84, 88, 84, 82	83	4.8
		overall	10	-	84	5.3
	Fat	0.01**	5	75, 87, 79, 77, 82	80	5.9
		0.10***	5	80, 83, 88, 84, 87	84	3.8
		overall	10	-	82	5.4
	Milk	0.01**	5	83, 79, 80, 85, 90	84	5.1
		0.10***	5	91, 86, 86, 87, 87	87	2.2
		overall	10	-	86	4.3
	Poultry egg	0.01**	5	80, 91, 81, 87, 92	86	6.4
		0.10***	5	85, 82, 87, 86, 81	84	3.1
		overall	10	-	85	4.9

* Fortified as flubendiamide-iodophthalimide, determined as A-12 and A-37, calculated flubendiamide-iodophthalimide (sum of A-12 + A-14).

** Expressed as parent equivalents (flubendiamide equivalents): 0.013 mg/kg.

*** Expressed as parent equivalents: 0.13 mg/kg.

For enforcement purposes using quantification via external standard procedure, the high recovery values and the higher RSDs for determination of flubendiamide in/on liver should be considered during quantification of residues of flubendiamide in liver. The detailed results are shown in Table 44.

Table 44 Validation data for flubendiamide and flubendiamide-iodophthalimide in animal matrices using the method 00912 by External Standard Procedure

Analytes	Substrate	Fortification level (mg/kg)	n	Recovery Rates (%)		
				Individual values	Mean	RSD
Flubendiamide *Quantitation* *m/z 681→254*	Muscle	0.01	5	90, 84, 83, 86, 80	85	4.4
		0.10	5	101, 84, 83, 87, 87	88	8.2
		overall	10	-	87	6.7
	Liver [a]	0.01	5	95, 80, 104, 107, 103	98	11.1
		0.10	10	125, 123, 123, 117, 133 90, 98, 99, 98, 89	110	14.9
		overall	15	-	106	14.6
	Kidney	0.01	5	74, 77, 80, 69, 76	75	5.4
		0.10	5	103, 93, 85, 77, 79	87	12.3
		overall	10	-	81	12.3
	Fat	0.01	5	80, 83, 92, 87, 87	86	5.3
		0.10	5	95, 93, 89, 95, 98	94	3.5
		overall	10	-	90	6.4
	Milk	0.01	5	79, 63, 79, 79, 71	74	9.6
		0.10	5	91, 68, 69, 71, 62	72	15.3
		overall	10	-	73	12.1
	Poultry egg	0.01	5	87, 88, 86, 88, 94	89	3.5
		0.10	5	74, 68, 69, 76, 100	77	16.9
		overall	10	-	83	12.9
Flubendiamide *Confirmation* *m/z 681 → 274*	Muscle	0.01	5	81, 80, 69, 92, 83	81	10.1
		0.10	5	109, 94, 91, 92, 93	96	7.8
		overall	10	-	88	12.2
	Liver [a]	0.01	4 [b]	78, 77, 103, 80	85	14.7
		0.10	10	131, 129, 138, 127, 135 89, 96, 99, 111, 87	114	17.5
		overall	14	-	106	21.3

Analytes	Substrate	Fortification level (mg/kg)	n	Recovery Rates (%)		
				Individual values	Mean	RSD
	Kidney	0.01	5	73, 83, 80, 70, 79	77	6.9
		0.10	5	101, 89, 82, 79, 81	87	10.4
		overall	10	-	82	10.5
	Fat	0.01	5	70, 83, 89, 80, 88	82	9.3
		0.10	5	98, 91, 89, 97, 98	95	4.5
		overall	10	-	88	10.0
	Milk	0.01	4 **	79, 88, 70, 83	80	9.5
		0.10	5	93, 72, 72, 80, 70	77	12.3
		overall	9	-	79	10.6
	Poultry egg	0.01	5	81, 79, 100, 99, 86	89	11.2
		0.10	5	71, 82, 77, 80, 108	84	17.1
		overall	10	-	86	13.8
flubendiamide-iodophthalimide *Quantitation m/z 548 → 504*	Muscle	0.01	5	106, 94, 93, 96, 91	96	6.1
		0.10	5	100, 86, 91, 93, 89	92	5.7
		overall	10	-	94	6.1
	Liver	0.01*	5	92, 81, 87, 90, 88	88	4.7
		0.10**	10	117, 111, 107, 93, 113 97, 99, 97, 100, 100	103	7.8
		overall	15	-	98	10.5
	Kidney	0.01	5	90, 83, 89, 85, 87	87	3.3
		0.10	5	97, 102, 96, 96, 90	96	4.4
		overall	10	-	92	6.6
	Fat	0.01	5	87, 92, 96, 95, 90	92	4.0
		0.10	5	90, 92, 89, 97, 87	91	4.2
		overall	10	-	92	3.9
	Milk	0.01	5	93, 84, 90, 93, 92	90	4.2
		0.10	5	119, 93, 93, 106, 96	101	11.0
		overall	10	-	96	10.2
	Poultry egg	0.01	5	81, 85, 86, 82, 79	83	3.5
		0.10	5	80, 80, 81, 80, 100	84	10.5
		overall	10	-	83	7.5
flubendiamide-iodophthalimide *Confirmation m/z 548 → 127*	Muscle	0.01	5	92, 84, 93, 88, 98	91	5.8
		0.10	5	101, 89, 90, 90, 88	92	5.8
		overall	10	-	91	5.5
	Liver	0.01	5	92, 87, 87, 94, 86	89	4.0
		0.10	10	118, 114, 110, 95, 116 97, 101, 98, 100, 104	105	8.1
		overall	15	-	100	10.6
	Kidney	0.01	5	82, 86, 90, 86, 96	88	6.0
		0.10	5	92, 100, 100, 94, 89	95	5.2
		overall	10	-	92	6.6
	Fat	0.01	5	78, 91, 86, 78, 89	84	7.2
		0.10	5	88, 87, 92, 92, 91	90	2.6
		overall	10	-	87	6.0
	Milk	0.01	5	105, 99, 105, 108, 116	107	5.8
		0.10	5	122, 91, 90, 103, 94	100	13.3
		overall	10	-	103	10.1
	Poultry egg	0.01	5	77, 86, 75, 81, 90	82	7.6
		0.10	5	81, 80, 83, 81, 100	85	9.9
		overall	10	-	83	8.6

An independent laboratory validation (ILV) of the method 00 912 was conducted by Anspach (2005). The results are shown in Tables 45 and 46.

Table 45 ILV data for method 00912 for flubendiamide and flubendiamide-iodophthalimide in animal matrices using the Internal Standard Procedure

Substrate	Fortification level (mg/kg)	Number of replicates	Recovery (%)	RSD (%)
flubendiamide *quantitation: m/z 681 → 254*				
Muscle	0.01	5	94	5.1
	0.10	5	97	2.5
	overall	10	96	4.1
Fat	0.02	5	104	3.8
	0.20	5	102	3.1
	overall	10	103	3.4
Egg	0.01	5	107	3.4
	0.10	5	98	2.7
	overall	10	102	5.4
flubendiamide *quantitation: m/z 681 → 274*				
Muscle	0.01	5	93	5.6
	0.10	5	96	3.8
	overall	10	95	4.7
Fat	0.02	5	103	6.2
	0.20	5	103	3.3
	overall	10	103	4.7
Egg	0.01	5	107	3.1
	0.10	5	94	3.2
	overall	10	101	7.0
flubendiamide-iodophthalimide *quantitation: m/z 548 → 504*				
Muscle	0.01	5	99	5.4
	0.10	5	91	0.9
	overall	10	95	5.6
Fat	0.02	5	86	3.8
	0.20	5	80	3.8
	overall	10	83	5.2
Egg	0.01	5	97	4.2
	0.10	5	88	4.0
	overall	10	92	6.5
flubendiamide-iodophthalimide *quantitation: m/z 548 → 127*				
Muscle	0.01	5	96	2.6
	0.10	5	91	0.9
	overall	10	93	3.3
Fat	0.02	5	83	4.1
	0.20	5	81	3.2
	overall	10	82	3.8
Egg	0.01	5	94	5.0
	0.10	5	88	4.3
	overall	10	91	5.8

Table 46 ILV data for method 00912 for flubendiamide and flubendiamide-iodophthalimide in animal matrices using the External Standard Procedure

Substrate	Fortification level (mg/kg)	Number of replicates	Recovery (%)	RSD (%)
flubendiamide *quantitation: m/z 681 → 254*				
Muscle	0.01	5	90	2.9
	0.10	5	90	6.1
	overall	10	90	4.6
Fat	0.02	5	94	5.0
	0.20	5	92	3.6
	overall	10	93	4.3
Egg	0.01	5	80	7.0
	0.10	5	92	3.8
	overall	10	86	8.8
flubendiamide *quantitation: m/z 681 → 274*				
Muscle	0.01	5	87	7.2
	0.10	5	93	4.0
	overall	10	90	6.4

Substrate	Fortification level (mg/kg)	Number of replicates	Recovery (%)	RSD (%)
Fat	0.02 0.20 overall	5 5 10	88 91 89	5.7 2.9 4.4
Egg	0.01 0.10 overall	5 5 10	81 89 85	4.4 1.2 6.1
flubendiamide-iodophthalimide *quantitation: m/z 548 → 504*				
Muscle	0.01 0.10 overall	5 5 10	97 96 97	4.3 7.1 5.6
Fat	0.02 0.20 overall	5 5 10	89 87 88	4.4 4.6 4.4
Egg	0.01 0.10 overall	5 5 10	90 86 88	11 6.3 8.9
flubendiamide-iodophthalimide *quantitation: m/z 548 → 127*				
Muscle	0.01 0.10 overall	5 5 10	95 96 96	3.5 7.1 5.3
Fat	0.02 0.20 overall	5 5 10	89 88 88	4.9 4.9 4.7
Egg	0.01 0.10 overall	5 5 10	87 87 87	9.9 5.4 7.6

Stability of pesticide residues in stored analytical samples

Studies were conducted to evaluate the stability of flubendiamide and its des-iodo metabolite in various crops stored under frozen conditions up to 18 months (Billian, 2005a,b,c). Individual aliquots of the homogenised sample materials (5 g per aliquot) were fortified with the compounds at 0.10 mg/kg which corresponds to the 10 x LOQ of the residue analytical method (0.010 mg/kg). Tables 47 summarise the amount of flubendiamide and its metabolite remaining in the stored samples after the various storage intervals, as well as the procedural recoveries from samples freshly fortified at the respective sampling points.

Table 47 Stability of flubendiamide and its des-iodo metabolite in various crops fortified at 0.1 mg/kg and stored frozen for up to 18 months

Crop	Storage interval, months	Flubendiamide		Flubendiamide-des-iodo	
		% remaining [a]	Procedural recovery, %	% remaining	Procedural recovery, %
Tomato	0 1 3 6 12 18	87* 92 90 90 85 90	— 99 92 88 89 91	90* 96 76 90 86 83	— 92 96 98 96 95
Citrus	0 1 3 6 12 18	85* 92 93 88 86 80	— 94 94 93 88 86	82* 97 78 95 85 77	— 88 91 97 93 89
Head cabbage (head)	0 1 3 6 12 18	75* 84 88 78 80 90	— 81 88 84 79 91	83* 89 78 86 81 84	— 90 92 90 88 94

Crop	Storage interval, months	Flubendiamide		Flubendiamide-des-iodo	
		% remaining [a]	Procedural recovery, %	% remaining	Procedural recovery, %
Bean (with pod)	0	83*	—	89*	—
	1	92	92	87	93
	3	85	89	79	96
	6	76	87	90	91
	12	71	84	79	88
	18	81	89	86	95
Wheat (grain)	0	78*	—	75*	—
	1	80	86	61	72
	3	73	81	57	69
	6	85	94	65	68
	12	74	83	65	77
	18	76	77	73	79
Olive oil	0	87*	—	84*	—
	1	81	87	89	92
	3	83	84	79	92
	6	81	84	89	99
	12	78	79	81	93
	18	98	104	81	80
Grape (must)	0	87*	98	75*	96
	1	85	101	87	92
	3	93	118	92	97
	6	91	94	83	92
	12	94	97	89	99

[a] The results are the mean from three replicate samples

* The results are the mean from five replicate samples

An additional freezer storage stability study was conducted performed in the US by Murphy (2006). Samples of various crops were spiked separately at levels of 0.15 mg/kg with flubendiamide and its des-iodo metabolite and stored under deep freezer storage for period of 1 year (368 days). Samples were analysed using the method 00816/M002 with modifications. Table 48 summarises the results.

Table 48 Stability of flubendiamide and its des-iodo metabolite various crops fortified at 0.15 mg/kg and stored frozen for up to one year

Sample material	Flubendiamide			Flubendiamide des-iodo		
	Storage interval, (days)	Sample number	% remaining	Storage interval, (days)	Sample number	% remaining
Cotton seed	0	3	114	0	3	97
	28	2	107	28	2	100
	89	2	105	89	2	94
	181	2	114	181	2	109
	273	2	100	273	2	116
	369	2	96	369	2	102
Cotton gin trash	0	3	112	0	3	114
	28	2	109	28	2	104
	89	2	116	89	2	108
	181	2	107	181	2	115
	272	2	111	272	2	119
	368	2	108	368	2	104
Cotton meal	0	3	114	0	3	108
	28	2	112	28	2	108
	89	2	111	89	2	106
	181	2	109	181	2	112
	273	2	106	273	2	109
	368	2	107	368	2	104

Sample material	Flubendiamide			Flubendiamide des-iodo		
	Storage interval, (days)	Sample number	% remaining	Storage interval, (days)	Sample number	% remaining
Wheat forage	0	3	88	0	3	93
	28	2	93	28	2	91
	89	2	88	89	2	92
	183	2	93	183	2	95
	274	2	88	274	2	89
	369	2	104	369	2	97
Wheat straw	0	3	116	0	3	108
	28	2	115	28	2	114
	89	2	114	89	2	118
	181	2	107	181	2	107
	273	2	110	273	2	111
	369	2	105	369	2	107
Wheat flour	0	3	90	0	3	96
	28	2	95	28	2	94
	89	2	92	89	2	89
	181	2	100	181	2	107
	273	2	91	273	2	100
	369	2	101	369	2	96
Wheat germ	0	3	111	0	3	108
	28	2	106	28	2	99
	89	2	91	89	2	93
	181	2	102	181	2	91
	272	2	113	272	2	113
	368	2	95	368	2	100
Potato tuber	0	3	89	0	3	91
	28	2	89	28	2	89
	89	2	83	89	2	91
	183	2	80	183	2	82
	274	2	101	274	2	106
	369	2	94	369	2	93
Tomato paste	0	3	96	0	3	95
	28	2	98	28	2	89
	89	2	84	89	2	97
	183	3	87	183	3	86
	274	2	98	274	2	89
	369	2	98	369	2	94

USE PATTERN

Flubendiamide is used as SC (soluble concentrate) or WG (water dispersible granule) formulation in many countries as ground or aerial application. The use patterns for ground application that are relevant for this evaluation are summarised in Table 49.

Table 49 Use patterns of flubendiamide used as foliar appication

Crop	Country	Formulation	Application			PHI (days)	RTI (days)
			Rate (kg/ha)	Water rate (L/ha)	No.		
Brassica sp.	Australia	SC / 480 WG / 240	0.024-0.048	0.0036-0.0048 kg ai/hL	3	3	
Brassica sp.	United States		0.034	93.5	2	1	3
Corn, field	United States	SC / 480	0.105	93.5	4	28	
Cotton	United States	SC / 480	0.105	93.5	3	28	
Cucurbits	Australia	WG / 240	0.048–0.072		3	1	
Cucurbit vegetables	United States	WG / 240	0.033–0.05	93.5	3	1	7

Crop	Country	Formulation	Application			PHI (days)	RTI (days)
			Rate (kg/ha)	Water rate (L/ha)	No.		
Leafy vegetables	Australia	WG / 240	0.036–0.048		3	1	
Leafy vegetables, except brassica	United States	WG / 240	0.033–0.050	93.5	3	1	3
Lettuce	Australia	SC / 480	0.036–0.048		3	1	
Legume vegetables	Australia	WG / 240	0.048–0.072		3	1	
Legume vegetables, except soya beans – succulent and dry beans and peas	United States	SC 480	0.10	93.5	2	1 (succulent) 14 (dry) 3 (forage, hay, vines)	5
Fruiting vegetables	Australia	WG 240	0.048–0.072		3	1	
Fruiting vegetables, except cucurbis	United States	WG / 240	0.033–0.050	93.5	3	1	3
Grape	United States	SC / 480	0.140	93.5	3	7	5
Pepper	Australia	SC / 480	0.072		3	1	
Pepper	Greece	WG / 240*	0.096	0.006 kg ai/hL	2	1	
Pepper	Netherlands	WG 240		0.006 kg ai/hL	2	1	
Pome fruit	United States	SC / 480	0.14 -0.175	93.5	3	14	7
Rice	Bangladesh	WG / 240	0.048	500	2	-	
Rice	India	SC / 480	0.024	375–500	3	40	
Stone fruit	United States	SC / 480	0.140	93.5	3	7	7
Soya bean	United States		0.10	93.5	2	1 (green seed) 14 (dry seed) 3 (forage and hay	5
Sweet corn	Australia	SC / 480	0.036 -0.072	0.036 - 0.072 kg ai/hL	2	1	
Sweet corn	United States	SC / 480	0.105	93.5	4	1	
Tomato	Australia	SC / 480	0.072		3	1	
Tomato	Greece	WG / 240*	0.120	0.006 kg ai/hL	2	3	
Tomato	Netherlands	WG 240*		0.006 kg ai/hL	2	1	
Tree nut	United States	SC / 480	0.140	93.5	3	14	7
Tea	Japan	WG / 200	0.400	2000–4000	1	7	

* Greenhouse application

RESIDUES RESULTING FROM SUPERVISED TRIALS

Six hundred fourty seven supervised residue trials conducted with flubendiamide in Australia, European Countries, USA, Japan, Thailand and India using foliar application of EC or WG formulation were submitted, as summarised below.

Crop	Countries	Table
Apple, pear	Belgium, Germany, italy, Canada and USA	50
Cherry	Canada, UA	51
Peach	Canada, USA, France, Greece, Italy, Portugal, Spain	52
Plum	USA, France, Italy, Spain	53

Crop	Countries	Table
Table grape	USA	54
Broccoli/cauliflour	France, Germany, Italy, Spain, UK, USA	55
Cabbage	Australia, France, Germany, Italy, Spain, UK , USA	56
Brussels sprout	Australia, France, Germany, Italy, Spain, UK , USA	57
Cucumber/summershquash (field)	USA	58
Cucumber (glasshouse)	France, GermanyGreece, Italy, Portugal, Spain	59
Melon/watermelon (field)	USA	60
Melon (glasshouse)	France, Italy, Portugal, Spain	61
Pepper (field)	Australia, USA	62
Pepper (glasshouse)	France, Germany, Italy, Netherlands,	63
Tomato (field)	Australia, USA	64
Tomato (glasshouse)	France, Germany, Italy, Netherlands, Portugal, Spain,	65
Sweet corn	USA	66
Lettuce	Australia	67
Spinach	USA	68
Green bean/pea	Australia, USA	69
Soya bean	USA	70
Dry bean/pea	USA	71
Celery	USA	72
Field corn	Canada, USA	73
Rice	Thailand, India	74
Tree nuts	USA	75
Cotton	USA	76
Tea	Japan	77
Animal feed from soya bean	USA	78
Animal feed from legumes	USA	79
Animal feed from corn	Canada, USA	80
Animal feed from rice	Thailand, India	81
Almond hulls	USA	82
Cotton gintrash	USA	83

Results from the supervised trials are shown in Tables 50 to 83. Residues of flubendiamide and its des-iodo methabolite were determined by LC/MS/MS method in most cases, with a LOQ of 0.01 mg/kg. Unless stated otherwise, in all trials, untreated control plots gave residues < LOQ.

Residues of flubendiamide within 25% GAP are underlined and were considered for estimation maximum residue level, HR and STMR. When residues in samples harvested at a latter stage were higher than what was found at the critical PHI, they will be selected for the estimations.

Pome fruits

Flubendiamide is registered in USA in pome fruits with a GAP of 3 × 0.14–0.175 kg ai/ha (minimum of 93.4 L water/ha) and 14 days PHI. A total of 50 residue trials were conducted with apples and pears in Canada, Europe and USA in 2004. The results are shown in Table 50.

Table 50 Results of residue trials conducted with flubendiamide in apple and pears in Europe and USA in 2004

		Application					Residues (mg/kg)[a]		
Country Region	Crop Variety	F	No	kg ai/ha	L/ha	PHI	Flubendiamide	F-des-iodo	Report No. Trial No.
Apple									
Belgium Brabant	Jonagold	SC	3	0.18	1500	0* 0 7 14 21	0.14 0.27 0.17 0.17 0.17	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2301/04 R 2004 0317 8
Belgium Brabant	Jonagold	SC	3	0.18	1500	0* 0 7 14 21	0.17 0.29 0.15 0.21 0.21	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2301/04 R 2004 0970 2
Belgium Brabant	Jonagold	WG	3	0.18	1500	0* 0 7 14 21	0.11 0.26 0.12 0.18 0.11	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2325/04 R 2004 0986 9
Canada Ontario	MacIntosh	SC	3	0.166-0.182	578-664	14	0.23	< 0.01	RCAMY009 AM176-04H-A
Canada Ontario	MacIntosh	SC	3	0.172-0.173	2665-2922	14	0.20	< 0.01	RCAMY009 AM176-04H-B
France Rhone-Alpes	Golden	SC	3	0.144-0.147	1200-1225	0* 0 7 14 21	0.12 0.23 0.29 0.23 0.19	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2302/04 R 2004 0318 6
Germany Nordrhein-Westfalen	Goldparmäne	SC	3	0.12	1000	0 14	0.34 0.18	< 0.01 < 0.01	RA-2301/04 R 2004 0982 6
Germany Nordrhein-Westfalen	Rubinette	SC	3	0.12-0.132	1000	0 15	0.26 0.21	< 0.01 < 0.01	RA-2301/04 R 2004 0983 4
Germany Nordrhein-Westfalen	Goldparmäne	WG	3	0.12	1000	0* 0 7 14 20	0.13 0.20 0.14 0.10 0.10	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2325/04 R 2004 0987 7
Italy Emilia-Romagna	Jonathan	SC	3	0.150	1250	0 14	0.16 0.17	< 0.01 < 0.01	RA-2302/04 R 2004 0976
Spain Cataluña	Golden	SC	3	0.18	1500	0 14	0.41 0.27	< 0.01 < 0.01	RA-2302/04 R 2004 0978 8
USA Pennsylvania	Red delicious	SC	3	0.17-0.18	542-559	11	0.47	< 0.01	RCAMY009 AM172-04H-A
USA Pennsylvania	Red delicious	SC	3	0.174-0.175	2343-2347	11	0.36	< 0.01	RCAMY009 AM172-04H-B

Country Region	Crop Variety	Application				PHI	Residues (mg/kg)[a]		Report No. Trial No.
		F	No	kg ai/ha	L/ha		Flubendiamide	F-des-iodo	
USA New York	Idareds	SC	3	0.177-0.179	555-560	14	0.17	< 0.01	RCAMY009 AM173-04H-A
USA New York	Idared	SC	3	0.171-0.173	1841-1862	14	0.19	< 0.01	RCAMY009 AM173-04H-B
USA New York	Idareds	SC	3	0.175-0.177	658-706	14	0.23	< 0.01	RCAMY009 AM174-04H-A
USA New York	Idareds	SC	3	0.177-0.180	2329-2365	14	0.22	< 0.01	RCAMY009 AM174-04H-B
USA Georgia	Empire	SC	3	0.174-0.175	367-394	14	0.17	< 0.01	RCAMY009 AM175-04H-A
USA Georgia	Empire	SC	3	0.171-0.177	2261-2535	14	0.18	< 0.01	RCAMY009 AM175-04H-B
USA Michigan	Red delicious	SC	3	0.175-0.177	542-545	14	0.48	< 0.01	RCAMY009 AM177-04H-A
USA Michigan	Red delicious	SC	3	0.175-0.177	2607-2633	14	0.33	< 0.01	RCAMY009 AM177-04H-B
USA Colorado	Red delicious	SC	3	0.175	549-569	14	0.16	< 0.01	RCAMY009 AM178-04H-A
USA Colorado	Red delicious	SC	3	0.175-0.176	2378-2429	14	0.30	< 0.01	RCAMY009 AM178-04H-B
USA, California	Summerfield	SC	3	0.170-0.174	480-485	14	0.18	< 0.01	RCAMY009 AM179-04H-A
USA California	Summerfield	SC	3	0.172-0.177	3002-3013	14	0.15	< 0.01	RCAMY009 AM179-04H-B
USA Washington	Braeburn	SC	3	0.175	464-466	0 7 14 21 28	0.26 0.25 0.23 0.22 0.25	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RCAMY009 AM180-04D-A
USA Washington	Braeburn	SC	3	0.174	2806-2812	14	0.41	< 0.01	RCAMY009 AM180-04D-B
USA Idaho	Law Rome	SC	3	0.172-0.176	467-471	14	0.27	< 0.01	RCAMY009 AM181-04H-A
USA Idaho	Law Rome	SC	3	0.169-0.178	2315-2362	14	0.25	< 0.01	RCAMY009 AM181-04H-B
USA Oregon	Jonagold	SC	3	0.181-0.186	397-639	14	0.19	< 0.01	RCAMY009 AM182-04H-A
USA Oregon	Jonagold	SC	3	0.173-0.179	2318-2626	14	0.21	< 0.01	RCAMY009 AM182-04H-B
USA Oregon	Jonagold	SC	3	0.176	494-594	14	0.06	< 0.01	RCAMY009 AM183-04H-A
USA Oregon	Jonagold	SC	3	0.173-0.175	1979-2967	14	0.13	< 0.01	RCAMY009 AM183-04H-B

Country Region	Crop Variety	Application				PHI	Residues (mg/kg)[a]		Report No. Trial No.
		F	No	kg ai/ha	L/ha		Flubendiamide	F-des-iodo	
Pear									
France Rhone-Alpes	Passe crassane	SC	3	0.156	1300	0* 0 7 14 22	0.09 0.23 0.16 0.13 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2302/04 R 2004 0975 3
Germany Westfalen	Pear Alexander Lucas	SC	3	0.12	1000	0 14	0.30 0.12	< 0.01 < 0.01	RA-2301/04 R 2004 0984 2
Germany Westfalen	Pear Alexander Lucas	SC	3	0.12-0.126	1000-1050	0 15	0.26 0.21	< 0.01 < 0.01	RA-2301/04 R 2004 0985 0
Italy, Emilia-Romagna	Pear William	SC	3	0.15	1250	0 14	0.33 0.16	< 0.01 < 0.01	RA-2302/04 R 2004 0979 6
Spain Cataluña	Pear Conference	SC	3	0.18	1500	13	0.34	< 0.01	RA-2302/04 R 2004 0981 8
USA Pennsylvania	Pear Bartlett	SC	3	0.176-0.178	548-551	14	0.23	< 0.01	RCAMY009 AM184-04H-A
USA Pennsylvania	Pear Bartlett	SC	3	0.175-0.178	2726-2763	14	0.20	< 0.01	RCAMY009 AM184-04H-B
USA California	Pear Asian	SC	3	0.175-0.177	529-572	13	0.06	< 0.01	RCAMY009 AM185-04H-A
USA Califor-nia	Pear Asian	SC	3	0.172-0.176	2345-2397	13	0.09	< 0.01 (	RCAMY009 AM185-04H-B
USA California	Pear Bartlett	SC	3	0.174-0.176	467-509	0 7 14 21 28	0.45 0.32 0.30 0.35 0.19	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RCAMY009 AM186-04D-A
USA Califor-nia	Pear Bartlett	SC	3	0.174-0.177	1921-1991	14	0.37	< 0.01	RCAMY009 AM186-04D-B
USA Oregon	Pear Cascade	SC	3	0.171-0.180	395-2290	14	0.36	< 0.01	RCAMY009 AM187-04H-A
USA Oregon	Pear Cascade	SC	3	0.173-0.176	623-2629	14	0.32	< 0.01	RCAMY009 AM187-04H-B
USA Idaho	Pear Bartlett	SC	3	0.175-0.179	462-471	14	0.29	< 0.01	RCAMY009 AM188-04H-A
USA Idaho	Pear Bartlett	SC	3	0.171-0.174	2278-2315	14 14	0.33	< 0.01	RCAMY009 AM188-04H-B
USA Washington	Pear Concord	SC	3	0.174-0.175	463-464	14	0.37	< 0.01	RCAMY009 AM189-04H-A
USA Washington	Pear Concord	SC	3	0.175	2314-2380	14 14	0.59	< 0.01	RCAMY009 AM189-04H-B

[a] Residues from trials conducted in Canada and USA are the highest of two samples

Cherries

Flubendiamide is registered in USA in stone fruits with a GAP of 3 × 0.14 kg ai/ha (minimum of 93.4 L water/ha) and 7 days PHI. Sixteen supervised trials were conducted in cherries in Canada and USA in 2004 and 2007. The results are shown on Table 51.

Table 51 Results of residue trials conducted with flubendiamide SC480 in/on cherry, fruit depited

Country, State	Variety	Application				Residues (mg/kg)^a		Report No. Trial No., year
		No	kg ai/ha	L/ha	PHI	Flubendiamide	F-des-iodo	
Canada Ontario	sour Montmorency	3	0.138-0.140	564-592	7	0.89	< 0.01	RCAMY011 AM193-04H-C, 2004
Canada Ontraio	sour Montmorency	3	0.141-0.143	2211-2289	7	0.99	< 0.01	RCAMY011 AM193-04H-D, 2004
USA California	Sweet Brooks	3	0.140-0.143	608-623	7	0.53	< 0.01	RCAMY011 AM194-04H-C, 2004
USA California	Sweet Brooks	3	0.134-0.142	2228-2377	7 7	0.63	< 0.01	RCAMY011 AM194-04H-D, 2004
USA California	sweet Bing	3	0.139-0.141	448-545	7	0.11	< 0.01	RAAML003 AM058-07HC, 2007
USA California	sweet Bing	3	0.137-0.141	1993-2082	7	0.19	< 0.01	RAAML003 AM058-07HD, 2007
USA Pennsylvania	Sour Montmorency	3	0.141-0.142	473-475	0 3 7 10 12	0.81 0.95 1.0 0.73 0.81	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RCAMY011 AM191-04D-C, 2004
USA Pennsylvania	sour Montmorency	3	0.139-0.142	2321-2372	7	1.0	< 0.01	RCAMY011 AM191-04D-D, 2004
USA Michigan	sour Montmorency	3	0.140-0.141	559-573	7	0.58	< 0.01	RCAMY011 AM192-04H-C, 2004
USA Michigan	sour Montmorency	3	0.140-0.141	1952-2034	7	0.60	< 0.01	RCAMY011 AM192-04H-D, 2004
USA Oregon	sweet Sweet Hearts	3	0.137-0.142	449-467	7	0.16	< 0.01	RCAMY011 AM195-04H-C, 2004
USA Oregon	sweet Sweet Hearts	3	0.138-0.145	2530-2563	7	0.25	< 0.01	RCAMY011 AM195-04H-D, 2004
USA Oregon	sweet Bing	3	0.140-0.141	589-619	7	0.20	< 0.01	RCAMY011 AM196-04H-C, 2004
USA Oregon	sweet Bing	3	0.140-0.141	2090-2121	7	0.57	< 0.01	RCAMY011 AM196-04H-D, 2004
USA Oregon	sweet Montmorency	3	0.140-0.141	519-513	7	0.45	< 0.01	RAAML003 AM057-07HC, 2007
USA Oregon	sweet Montmorency	3	0.139-0.142	2313-2354	7	0.48	< 0.01	RAAML003 AM057-07HD, 2007

[a] Residues are the highest of two samples

Peaches and nectarines

Flubendiamide is registered in USA in stone fruits with a GAP of 3 × 0.14 kg ai/ha (minimum of 93.4 L water/ha) and 7 days PHI. A total of 57 residue trials were conducted with flubendiamide with peaches and nectarines in North America and Europe in 2003/2004. The results are shown on Table 52.

Table 52: Results of residue trials conducted with flubendiamide SC480 in/on peach and nectarines

Country, State	Variety	Application			Portion	PHI	Residues (mg/kg)^a		Report No. Trial No., year
		No	kg ai/ha	L/ha			Flubendiamide	F-des-iodo	
Canada Ontario	Peach, Canadian Harmony	3	0.138-0.141	490-537	fruit, depitted	7	0.20	< 0.01	RCAMY011 AM201-04H-C, 2004
Canada Ontario	Peach, Canadian Harmony	3	0.139-0.142	2155-2319	fruit, depitted	7	0.15	< 0.01	RCAMY011 AM201-04H-D, 2004

Country, State	Variety	Application			Portion	PHI	Residues (mg/kg)^{a}		Report No. Trial No., year
		No	kg ai/ha	L/ha			Flubendia-mide	F-des-iodo	
France Cote D'azur	Peach Spring White	3	0.132-0.138	1100-1150	fruit	0* 0 7 14 21	0.11 0.17 0.12 0.10 0.10	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2303/04 R 2004 0321 6, 2004
France Provence-Cote D'azur	Nectarine Big Top	3	0.135-0.138	1125-1150	fruit	0* 0 7 14 21	0.12 0.27 0.18 0.17 0.15	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2303/04 R 2004 0993 , 2004
Greece Tripotamus	Peach Hall	3	0.132	1100	fruit	0* 0 7 14 21	0.28 0.32 0.22 0.16 0.11	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2063/03 R 2003 0194 4, 2003
Italy Emilia - Romagna	Peach Red Haven	3	0.150	1250	fruit	0 7 14	0.38 0.12 0.09	< 0.01 < 0.01 < 0.01	RA-2303/04 R 2004 0995 8, 2004
Portugal Lamorosa	Peach Vistariche	3	0.120	1000	fruit	0 7 14	0.24 0.20 0.15	< 0.01 < 0.01 < 0.01	RA-2303/04 R 2004 0998 2, 2004
Portugal Alenquer	Peach Spring crest	3	0.120	1000	fruit	0* 0 7 14 21	0.19 0.33 0.22 0.12 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2063/03 R 2003 0771 3, 2003
Spain La Fortesa	Peach Royal Glory	3	0.144-0.150	1250	fruit	0* 0 6 15 21	0.24 0.37 0.25 0.14 0.12	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2063/03 R 2003 0770 5, 2003
Spain Cataluña	Nectarine Diptop	3	0.168	1400	fruit	0 8 15	0.30 0.24 0.22	< 0.01 < 0.01 < 0.01	RA-2303/04 R 2004 0996 6. 2004
Spain Cataluña	Peach Ruby Rich	3	0.150	1250	fruit	0 8 13	0.25 0.21 0.17	< 0.01 < 0.01 < 0.01	RA-2303/04 R 2004 0997 4, 2004
USA California	Peach OHenry	3	0.138-0.140	409-447	fruit, depitted	0 3 7 10 14	0.36 0.20 0.18 0.14 0.15	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RCAMY011 AM203-04D-C, 2004
USA California	Peach OHenry	3	0.139-0.142	1976-2022	fruit, depitted	7	0.35	< 0.01	RCAMY011 AM203-04D-D, 2004
USA California	Peach Ryans Sun	3	0.139-0.142	607-609	fruit, depitted	7	0.28	< 0.01	RCAMY011 AM204-04H-C, 2004
USA California	Peach Ryans Sun	3	0.138-0.140	2331-2342	fruit, depitted	7	0.39	< 0.01	RCAMY011 AM204-04H-D, 2004
USA California	Peach Starn	3	0.140-0.141	467-470	fruit, depitted	7	0.26	< 0.01	RCAMY011 AM205-04H-C, 2004
USA California	Peach Starn	3	0.138-0.140	1936-1958	fruit, depitted	7	0.40	< 0.01	RCAMY011 AM205-04H-D, 2004
USA Georgia	Peach Hawthorne	3	0.140	395-408	fruit, depitted	7	0.20	< 0.01	RCAMY011 AM199-04H-C, 2004
USA Georgia	Peach Hawthorne	3	0.140-0.141	2006-2099	fruit, depitted	7	0.20	< 0.01	RCAMY011 AM199-04H-D, 2004
USA Georgia	Peach Dixieland	3	0.140-0.144	470-496	fruit, depitted	7	0.16	< 0.01	RCAMY011 AM200-04H-C, 2004
USA Georgia	Peach Dixieland	3	0.139-0.141	2907-3182	fruit, depitted	7	0.30	< 0.01	RCAMY011 AM200-04H-D, 2004

Country, State	Variety	Application			Portion	PHI	Residues (mg/kg)[a]		Report No. Trial No., year
		No	kg ai/ha	L/ha			Flubendiamide	F-des-iodo	
USA Pennsylvania	Peach Glen Glo	3	0.141-0.142	508-594	fruit, depitted	7	0.20	< 0.01 < 0.01	RCAMY011 AM197-04H-C, 2004
USA Pennsylvania	Peach Glen Glo	3	0.140-0.142	3007-3035	fruit, depitted	7	0.24	< 0.01	RCAMY011 AM197-04H-D, 2004
USA Texas	Peach Texas Royal	3	0.139-0.140	532-552	fruit, depitted	7	0.22	< 0.01	RCAMY011 AM202-04H-C, 2004
USA Texas	Peach Texas Royal	3	0.140-0.142	2773-2851	fruit, depitted	7 7	0.32	< 0.01	RCAMY011 AM202-04H-D, 2004
USA Virginia	Peach Encore	3	0.141-0.142	593-752	fruit, depitted	7	0.23	< 0.01 < 0.01	RCAMY011 AM198-04HA-C, 2004
USA Virginia	Peach Encore	3	0.140	2704-2708	fruit, depitted	7	0.20	< 0.01	RCAMY011 AM198-04HA-D, 2004

[a]. Residues from trials conducted in Canada and USA are the higher of two samples

Plums

Flubendiamide is registered in USA in stone fruits with a GAP of 3 × 0.14 kg ai/ha (minimum of 93.4 L water/ha) and 7 days PHI. A total of 15 residue trials were conducted in Europe and USA with plums in 2004. The results are shown in Table 53.

Table 53 Results of residue trials conducted with flubendiamide SC480 in/on plum in 2004

Country, State	Variety	Application			Portion	PHI	Residues (mg/kg)a		Report No. Trial No.
		No	kg ai/ha	L/ha			Flubendiamide	F-des-iodo	
France Midi-Pyrenees	Bave	3	0.180	1500	fruit	0* 0 7 14 21	0.03 0.09 0.05 0.05 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2304/04 R 2004 0895 1
Italy Emilia - Romagna	Golden Pluma	3	0.180	1500	fruit	0* 0 7 14 21	0.02 0.06 0.05 0.04 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2304/04 R 2004 0403 4
Spain Comunidad Valenciana	Black Gold	3	0.108	900	fruit	0* 0 7 14 20	0.03 0.08 0.07 0.06 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2304/04 R 2004 0894 3
USA California	Autumn Beauty	3	0.140	493-499	fruit, depitted	0 3 7 10 14	0.03 0.04 0.02 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RCAMY011 AM207-04D-C
USA California	Autumn Beauty	3	0.138	2765-2808	fruit, depitted	7	0.03	< 0.01	RCAMY011 AM207-04D-D
USA California	Angelinas	3	0.138-0.144	522-614	fruit, depitted	7	0.03	< 0.01	RCAMY011 AM208-04HA-C
USA California	Angelinas	3	0.139-0.143	2444-2699	fruit, depitted	7	0.05	< 0.01	RCAMY011 AM208-04HA-D
USA California	Autumn Beauty	3	0.139-0.141	553-568	fruit, depitted	7	0.04	< 0.01	RCAMY011 AM209-04H-C

		Application					Residues (mg/kg)a		Report No. Trial No.
Country, State	Variety	No	kg ai/ha	L/ha	Portion	PHI	Flubendiamide	F-des-iodo	
USA California	Autumn Beauty	3	0.141	2425-2502	fruit, depitted	7 7	0.14	< 0.01	RCAMY011 AM209-04H-D
USA California	Howard Sun	3	0.137-0.140	494-498	fruit, depitted	7	0.02	< 0.01	RCAMY011 AM210-04H-C
USA California	Howard Sun	3	0.141-0.142	2828-2862	fruit, depitted	7	0.01	< 0.01	RCAMY011 AM210-04H-D
USA Michigan	Stanley	3	0.139	522-611	fruit, depitted	7	0.44	< 0.01	RCAMY011 AM206-04H-C
USA Michigan	Stanley	3	0.139-0.140	2700-3062	fruit, depitted	7	0.50	< 0.01	RCAMY011 AM206-04H-D
USA Oregon	Italian	3	0.138-0.143	477-509	fruit, depitted	7	0.09	< 0.01	RCAMY011 AM211-04H-C
USA Oregon	Italian	3	0.139-0.142	2067-2143	fruit, depitted	7	0.07	< 0.01	RCAMY011 AM211-04H-D

[a] Residues from trials conducted in USA and * are the highest of two samples

Grape

Flubendiamide is registered in USA in grape with a GAP of 3 × 0.14 kg ai/ha (minimum of 93.4 L water/ha) and 7 days PHI. Twelve trials were conducted in USA with flubendiamide in grape in 2004. The results are shown in Table 54.

Table 54 Results of residue trials conducted with flubendiamide SC480 in/on grape in USA in 2004 (Report No. RCAMY016)

		Application				Residues (mg/kg)[a]		Trial No.
State	Variety	No	kg ai/ha	L/ha	PHI	Flubendiamide	F-des-iodo	
California	Thompson Seedless	3	0.14	505-509	0 3 7 10 14	0.20 0.18 0.19 0.15 0.20	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	AM242-04D
California	Centurion	3	0.14	511-514	7	0.40	< 0.01	AM243-04H
California	Crimson	3	0.14	550-571	7	0.47	< 0.01	AM244-04H
California	Thompson Seedless	3	0.14	529-548	7	0.19	< 0.01	AM245-04H
California	Chardonnay	3	0.14	510-547	7	0.81	< 0.01	AM246-04H
California	Merlot	3	0.14	609-610	7	0.43	< 0.01	AM247-04H
California	Thompson Seedless	3	0.14	481-490	7	0.12	< 0.01	AM248-04H
California	Thompson Seedless	3	0.14	488-506	7	0.69	< 0.01	AM249-04H
Idaho	Concord	3	0.14	558-567	7	0.12	< 0.01	AM250-04H
New York	Vidal Blanc	3	0.14	567-572	7	0.68	< 0.01	AM241-04H
Pennsylvania	Concord	3	0.14	618-621	7	0.22	< 0.01	AM240-04H
Washington	Chardonay	3	0.14	562-565	7	0.67	< 0.01	AM251-04H

[a]. Residues are the highest of two samples

Broccoli and cauliflower

Flubendiamide is registered in brassica vegetables in Australia at a maximum rate of 3 × 0.048 kg ai/ha (0.0048 kg ai/hL) and 3 days PHI. GAP in USA for brassica is 2 x 0.034 kg ai/ha and 1 day PHI.

Twenty seven trials were conducted with flubendiamie in broccoli and nine in cauliflower in Australia, Europe and USA from 2004 to 2006. The results are shown on Table 55.

Table 55 Results of residue trials conducted with flubendiamide in/on broccoli and cauliflower

Country Region	Crop Variety	Application					PHI	Residues (mg/kg)[a]		Report No. Trial No., year
		F	No	kg ai/ha	kg ai/hL	L/ha		Flubendia-mide	F-des-iodo	
Broccoli										
AUS Victoria	Broccoli Viper	240 WG	3	0.048			0*	0.05	< 0.02	BCS-0083 VC29, 2006
							0	0.44	< 0.02	
							1	0.29	< 0.02	
							3	0.13	< 0.02	
							7	0.11	< 0.02	
AUS Victoria	Broccoli Viper	240 WG	3	0.072			0*	0.07	< 0.02	BCS-0083 VC29, 2006
							0	0.53	< 0.02	
							1	0.48	< 0.02	
							3	0.36	< 0.02	
							7	0.12	< 0.02	
AUS Victoria	Broccoli Viper	240 WG	3	0.108			0*	0.10	< 0.02	BCS-0083 VC29, 2006
							0	0.36	< 0.02	
							1	0.28	< 0.02	
							3	0.48	< 0.02	
							7	0.21	< 0.02	
AUS Victoria	Broccoli Viper	480 SC	3	0.048			0*	0.04	< 0.02	BCS-0118 B56, 2006
							0	0.45	< 0.02	
							3	0.25	< 0.02	
							7	0.05	< 0.02	
							14	< 0.02	< 0.02	
AUS Victoria	Broccoli Viper	480 SC	3	0.096			0*	0.37	< 0.02	BCS-0118 B56, 2006
							0	0.34	< 0.02	
							3	0.30	< 0.02	
							7	0.31	< 0.02	
							14	0.02	< 0.02	
AUS Victoria	Broccoli Viper	240 WG	3	0.096			0*	0.46	< 0.02	BCS-0118 B56, 2006
							0	0.63	< 0.02	
							3	0.17	< 0.02	
							7	0.24	< 0.02	
							14	0.07	< 0.02	
AUS Queensland	Broccoli Babylon	240 WG	3	0.048			0*	< 0.02	< 0.02	BCS-0083 Q1, 2006
							0	0.10	< 0.02	
							2	0.14	< 0.02	
							3	0.22	< 0.02	
							7	0.05	< 0.02	
AUS Queensland	Broccoli Babylon	240 WG	3	0.072			0*	0.07	< 0.02	BCS-0083 Q1, 2006
							0	0.21	< 0.02	
							2	0.27	< 0.02	
							3	0.26	< 0.02	
							7	0.09	< 0.02	
AUS Queensland	Broccoli Babylon	240 WG	3	0.108			0*	0.06	< 0.02	BCS-0083 Q1, 2006
							0	0.14	< 0.02	
							2	0.24	< 0.02	
							3	0.26	< 0.02	
							7	0.11	< 0.02	
France Haute-Normandie	Broccoli Monaco	480 SC	3	0.048	0.016		0*	< 0.01	< 0.01	RA-2317/04 R 2004 0950 8, 2004
							0	0.17	< 0.01	
							3	0.09	< 0.01	
							7	0.06	< 0.01	
							14	0.02	< 0.01	
Germany Langenfeld (Weitz)	Broccoli Marathon	480 SC	3	0.048	0.008		0*	0.04	< 0.01	RA-2317/04 R 2004 0949 4, 2004
							0	0.16	< 0.01	
							3	0.15	< 0.01	
							7	0.08	< 0.01	
							13	0.04	< 0.01	

Country Region	Crop Variety	Application					PHI	Residues (mg/kg)[a]		Report No. Trial No., year
		F	No	kg ai/ha	kg ai/hL	L/ha		Flubendiamide	F-des-iodo	
Italy Melfi (PZ) (Puglia)	Broccoli Maraton	480 SC	3	0.048	0.01		0* 0 3 7 14	0.04 0.21 0.13 0.15 0.10	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2318/04 R 2004 0408 5, 2004
Spain Cataluña	Broccoli Marathon	480 SC	3	0.048	0.005		0* 0 2 7 14	0.03 0.15 0.13 0.07 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2318/04 R 2004 0953 2 2004
Spain Gavá (Cataluña)	Broccoli Marathon	24 WG	3	0.048	0.005		0* 0 2 7 14	0.03 0.14 0.14 0.05 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2327/04 R 2004 0968 0 2004
USA Oklahoma	Broccoli Green Comet	24 WG	3	0.33- 0.34		106- 108	1	0.23	< 0.01	RCAMY001 AM067-04H, 2004
USA California	Broccoli Green Comet	24 WG	3	0.033- 0.034		173- 175	0 1 3 7 10	0.14 0.12 0.12 0.12 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RCAMY001 AM068-04D 2004
USA California	Broccoli Greenbelt	24 WG	3	0.034		142	1	0.16	< 0.01	RCAMY001 AM069-04H, 2004
United Kingdom Norfolk	Broccoli Marathon	480 SC	3	0.048	0.016		0* 0 3 7 14	< 0.01 0.38 0.21 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2317/04 R 2004 0407 7 2004
Cauliflower										
France Midi- Pyrenees	Cauliflower Flamenco	480 SC	3	0.048	0.01		0* 0 3 7 14	< 0.01 0.02 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2318/04 R 2004 0956 7, 2004
Germany Rhineland- Palatinate	Cauliflower Freedom	480 SC	3	0.048	0.006		0* 0 3 7 14	0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2317/04 R 2004 0951 6, 2004
Germany Langenfeld (Weitz)	Cauliflower Freedom	480 SC	3	0.048	0.008		0* 0 3 7 13	< 0.01 0.05 0.02 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2317/04 R 2004 0952 4, 2004
Italy Lazio	Cauliflower Balboa	480 SC	3	0.048	0.006		0* 0 3 7 14	< 0.01 0.03 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2318/04 R 2004 0954 0, 2004
Italy Lazio	Cauliflower Balboa	24 WG	3	0.048	0.006		0* 0 3 7 14	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2327/04 R 2004 0969 9, 2004
Spain Gava (Cataluña)	Cauliflower Casper	480 SC	4*	0.045- 0.048	0.008		0* 0 3 7 14	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2318/04 R 2004 0955 9 2004

		Application						Residues (mg/kg)[a]		Report No. Trial No., year
Country Region	Crop Variety	F	No	kg ai/ha	kg ai/hL	L/ha	PHI	Flubendia-mide	F-des-iodo	
USA California	Cauliflower Symphony	24 WG	3	0.033-0.034		130-132	1	< 0.01	< 0.01 < 0.01	RCAMY001 AM070-04H, 2004
USA California	Cauliflower Chieftan	24 WG	3	0.033-0.035		188-194	1	0.02	< 0.01	RCAMY001 AM071-04H, 2004
USA Oregon	Cauliflower Snowball	24 WG	3	0.034-0.036		169-175	1	0.03	< 0.01	RCAMY001 AM072-04H, 2004

[a]. Residues from trials conducted in USA and are the highest of two samples

Cabbage

Flubendiamide is registered in brassica vegetables in Australia at a maximum rate of 3 × 0.048 kg ai/ha (0.0048 kg ai/hL) and 3 days PHI. GAP in USA for brassica is 2 × 0.034 kg ai/ha and 1 day PHI. A total of 35 residue trials were conducted with flubendiamide in/on cabbage in Australia, Europe and USA from 2004 to 2007. The results are shown on Table 56.

Table 56 Results of residue trials conducted with flubendiamide in head cabbage

		Aplication						Residues (mg/kg)[a]		
Country Region	Crop Variety	F	No	kg ai/ha	kg ai/hL (L/ha)	Portion	PHI	Flubendia-mide	F-des-iodo	Report No. Trial No.
AUS Tasmania	Sombrero	240 WG	3	0.048		head	0* 0 3 7 14	0.09 0.13 0.19 0.04 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0082 C81 2007
AUS Tasmania	Sombrero	240 WG	3	0.072		head	0* 0 3 7 14	0.08 0.22 0.20 < 0.02 0.05	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0082 C81 2007
AUS Tasmania	Sombrero	240 WG	3	0.108		head	0* 0 3 7 14	0.17 0.19 0.24 < 0.02 0.07	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0082 C81 2007
AUS South Australia	Grand slam	240 WG	3	0.048		head	0* 0 3 7 14	0.02 0.38 0.04 0.04 0.20	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0082 C82 2007
AUS South Australia	Grand slam	240 WG	3	0.072		head	0* 0 3 7 14	0.10 0.72 0.38 0.15 0.20	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0082 C82 2007
AUS South Australia	Grand slam	240 WG	3	0.108		head	0* 0 3 7 14	0.03 1.1 0.11 0.44 0.18	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0082 C82 2007
AUS Queensland	Drum Head	240 WG	3	0.048		head	0* 0 3 7 14	0.15 0.37 0.30 0.43 0.23	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0082 C83 2007

		Aplication						Residues (mg/kg)[a]		
Country Region	Crop Variety	F	No	kg ai/ha	kg ai/hL (L/ha)	Portion	PHI	Flubendia-mide	F-des-iodo	Report No. Trial No.
AUS Queensland	Drum Head	240 WG	3	0.072		head	0*	0.33	< 0.02	BCS-0082 C83 2007
							0	1.0	< 0.02	
							3	0.29	< 0.02	
							7	0.11	< 0.02	
							14	0.03	< 0.02	
AUS Queensland	Drum Head	240 WG	3	0.108		head	0*	0.62	< 0.02	BCS-0082 C83 2007
							0	1.7	< 0.02	
							3	0.13	< 0.02	
							7	0.30	< 0.02	
							14	0.32	< 0.02	
AUS Victoria	Drum Head	480 SC	3	0.048		head	0*	0.05	< 0.02	BCS-0117 C136 2006
							0	0.27	< 0.02	
							3	0.27	< 0.02	
							7	0.09	< 0.02	
							14	0.04	< 0.02	
AUS Victoria	Drum Head	480 SC	3	0.096		head	0*	0.40	< 0.02	BCS-0117 C136 2006
							0	0.48	< 0.02	
							3	0.29	< 0.02	
							7	0.13	< 0.02	
							14	0.18	< 0.02	
AUS Victoria	Drum Head	240WG	3	0.096		head	0*	0.09	< 0.02	BCS-0117 C136 2006
							0	0.43	< 0.02	
							3	0.61	< 0.02	
							7	0.24	< 0.02	
							14	0.35	< 0.02	
AUS Queensland	Drum Head	480 SC	3	0.048		head	0*	1.2	< 0.02	BCS-0117 C137 2006
							0	0.40	< 0.02	
							3	0.76	< 0.02	
							7	0.92	< 0.02	
							14	0.18	< 0.02	
AUS Queensland	Drum Head	480 SC	3	0.096		head	0*	1.6	< 0.02	BCS-0117 C137 2006
							0	0.94	< 0.02	
							3	1.3	< 0.02	
							7	1.1	< 0.02	
							14	01.3	< 0.02	
AUS Queensland	Drum Head	240WG	3	0.096		head	0*	0.56	< 0.02	BCS-0117 C137 2006
							0	0.44	< 0.02	
							3	1.8	< 0.02	
							7	1.6	< 0.02	
							14	0.34	< 0.02	
AUS Queensland	Drum Head	480 SC	3	0.048		head	0*	0.80	< 0.02	BCS-0117 C138 2006
							0	4.6	0.03	
							3	2.7	< 0.02	
							7	0.97	< 0.02	
							14	0.26	< 0.02	
AUS Queensland	Drum Head	480 SC	3	0.096		head	0*	1.8	< 0.02	BCS-0117 C138 2006
							0	7.7	0.03	
							3	4.1	< 0.02	
							7	2.0	< 0.02	
							14	0.14	< 0.02	
AUS Queensland	Drum Head	240WG	3	0.096	0.083	head	0*	0.35	< 0.02	BCS-0117 C138 2006
							0	4.7	0.03	
							3	1.1	< 0.02	
							7	< 0.02	< 0.02	
							14	0.08	< 0.02	
France Alsace	Cabbage, white Iglesias	480 SC	3	0.048	(400)	head	0*	< 0.01	< 0.01	RA-2314/04 R 2004 0405 0 2004
							0	< 0.01	< 0.01	
							3	0.01	< 0.01	
							7	< 0.01	< 0.01	
							14	< 0.01	< 0.01	

Country Region	Crop Variety	Aplication				Portion	PHI	Residues (mg/kg)[a]		Report No. Trial No.
		F	No	kg ai/ha	kg ai/hL (L/ha)			Flubendia-mide	F-des-iodo	
France Rhone-Alpes	Cabbage, white shelton	480 SC	3	0.048	(500)	head	0* 0 2 8 14	< 0.01 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2315/04 R 2004 0904 4 2004
Germany Nordrhein-Westfalen	Cabbage, red Autoro	480 SC	3	0.048	(400)	head	0* 0 2 6 15	0.02 0.04 0.03 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2314/04 R 2004 0957 5 2004
Germany Leichlingen	Cabbage, white Marcello	480 SC	3	0.048	(600)	head	0* 0 3 7 14	0.01 0.08 0.03 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2314/04 R 2004 0958 3 2004
Germany Leichlingen	Cabbage, white Marcello	480 SC	3	0.144	(600)	head	7	0.02	>0.01	RA-2314/04 R 2004 1043 3 2004
Italy Abruzzo	Cabbage, white Head start F1	480 SC	3	0.048	(800)	head	0* 0 3 7 14	< 0.01 0.03 < 0.01 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2315/04 R 2004 0406 9 2004
Italy ILazio	Cabbage, red Primero	480 SC	3	0.048	(600)	head	0* 0 3 7 14	< 0.01 0.04 0.02 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2315/04 R 2004 0903 6 2004
Spain Andalusia	Cabbage, red Redsky F1	480 SC	3	0.048	(400)	head	0* 0 3 7 14	0.07 0.10 0.08 0.05 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2315/04 R 2004 0905 2 2005
Spain Cataluña	Cabbage, red Sombrero	480 SC	3	0.048	(700)	head	0* 0 3 7 14	< 0.01 0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2315/04 R 2004 0906 0 2005
UK Lincolnshire	Cabbage, red Rococo	480 SC	3	0.048	(600)	head	0* 0 3 6 14	0.03 0.06 0.04 0.05 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2314/04 R 2004 0959 1 2004
UK Lincolnshire	Cabbage, white Impulse	480 SC	3	0.048	(600)	head	0* 0 3 6 14	< 0.01 0.02 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2314/04 R 2004 0960 5 2004
						head, inner	1	< 0.01	< 0.01	
USA California	Cabbage Gazelle	24 WG	3	0.033-0.034	(170-173)	head	1	0.25	< 0.01	RCAMY001 AM078-04H 2004
						head, inner	1	0.01	< 0.01	
USA Florida	Cabbage Bravo	24 WG	3	0.033-0.034	(102-135)	head	1	0.39	< 0.01	RCAMY001 AM075-04H 2004
						head, inner	1	0.02	< 0.01	
USA New York	Blue thunder	24 WG	3	0.035	(159-161)	head	1	0.18		RCAMY001 AM073-04H
USA Georgia	Cabbage Bravo	24 WG	3	0.034	170-174	head	1	0.25	< 0.01	RCAMY001 AM074-04H 2004
						head, inner	1	0.01	< 0.01	

		Aplication						Residues (mg/kg)[a]		
Country Region	Crop Variety	F	No	kg ai/ha	kg ai/hL (L/ha)	Portion	PHI	Flubendia-mide	F-des-iodo	Report No. Trial No.
USA Kansas	Cabbage Flat Head Dutch	24 WG	3	0.033-0.035	(132-138)	head	0	0.22	< 0.01	RCAMY001 AM076-04D 2004
							1	0.30	< 0.01	
							3	0.03	< 0.01	
							7	0.02	< 0.01	
							10	0.03	< 0.01	
USA Texas	Cabbage Blue Thunder	24 WG	3	0.034	(176-188)	head	1	0.12	< 0.01	RCAMY001 AM077-04H 2004
						head, inner	1	< 0.01	< 0.01	

[a]. Residues from trials conducted in USA are the highest of two samples

Brussels sprouts

Flubendiamide is registered in brassica vegetables in Australia at a maximum rate of 3 × 0.048 kg ai/ha (0.0048 kg ai/hL) and 3 days PHI. GAP in USA for brassica is 2 × 0.034 kg ai/ha and 1 day PHI. Seventeen trials were conduced with flubendiamide in Brussels sprouts in Australia and Europe in 2004 and 2006. The results are shown on Table 57.

Table 57 Results of residue trials conducted with flubendiamide in Brussels sprouts

		Aplication						Residues (mg/kg)		
Country Region	Variety	F	No	kg ai/ha	kg ai/hL	L/ha	PHI	Flubendia-mide	F-des-iodo	Report No. Trial No.
AUS Victoria	Oliver	240 WG	3	0.048			0*	< 0.02	< 0.02	BCS-0081 VC30 2006
							0	0.27	< 0.02	
							1	1.1	< 0.02	
							3	0.18	< 0.02	
							7	0.23	< 0.02	
AUS Victoria	Oliver	240 WG	3	0.072			0*	0.06	< 0.02	BCS-0081 VC30 2006
							0	0.09	< 0.02	
							1	0.87	< 0.02	
							3	1.1	< 0.02	
							7	0.62	< 0.02	
AUS Victoria	Oliver	240 WG	3	0.106			0*	0.27	< 0.02	BCS-0081 VC30 2006
							0	0.56	< 0.02	
							1	1.3	< 0.02	
							3	1.1	< 0.02	
							7	0.96	< 0.02	
AUS Victoria	Oliver	240 WG	3		0.0048		0*	0.09	< 0.02	BCS-0081 VC30 2006
							0	1.1	< 0.02	
							1	1.2	< 0.02	
							3	1.1	< 0.02	
							7	0.82	< 0.02	
AUS Victoria	Oliver	240 WG	3		0.0072		0*	0.07	< 0.02	BCS-0081 VC30 2006
							0	0.17	< 0.02	
							1	1.4	< 0.02	
							3	1.4	< 0.02	
							7	0.78	< 0.02	
AUS Victoria	Oliver	240 WG	3		0.0108		0*	0.88	< 0.02	BCS-0081 VC30 2006
							0	1.1	< 0.02	
							1	1.5	< 0.02	
							3	1.5	< 0.02	
							7	1.0	< 0.02	
AUS South Australia	Oliver	240 WG	3	0.048			0*	0.07	< 0.02	BCS-0081 SA24 2006
							0	0.12	< 0.02	
							1	0.08	< 0.02	
							3	0.05	< 0.02	
							7	0.08	< 0.02	

Country Region	Variety	Aplication					PHI	Residues (mg/kg)		Report No. Trial No.
		F	No	kg ai/ha	kg ai/hL	L/ha		Flubendiamide	F-des-iodo	
AUS South Australia	Oliver	240 WG	3	0.072			0*	0.10	< 0.02	BCS-0081 SA24 2006
							0	0.10	< 0.02	
							1	0.12	< 0.02	
							3	0.09	< 0.02	
							7	0.11	< 0.02	
AUS South Australia	Oliver	240 WG	3	0.106			0*	0.13	< 0.02	BCS-0081 SA24 2006
							0	0.22	< 0.02	
							1	0.11	< 0.02	
							3	0.23	< 0.02	
							7	0.15	< 0.02	
AUS South Australia	Oliver	240 WG	3		0.0048		0*	0.14	< 0.02	BCS-0081 SA24 2006
							0	0.49	< 0.02	
							1	0.87	< 0.02	
							3	0.50	< 0.02	
							7	0.41	< 0.02	
AUS South Australia	Oliver	240 WG	3		0.0072		0*	0.38	< 0.02	BCS-0081 SA24 2006
							0	0.70	< 0.02	
							1	1.1	< 0.02	
							3	1.1	< 0.02	
							7	0.66	< 0.02	
AUS South Australia	Oliver	240 WG	3		0.0108		0*	1.2	< 0.02	BCS-0081 SA24 2006
							0	1.8	< 0.02	
							1	1.6	< 0.02	
							3	0.81	< 0.02	
							7	1.6	< 0.02	
France Fontaine l’etalon (Picardie) EUN	Brussels sprouts Maximum	480 SC	3	0.048		400	0*	0.01	< 0.01	RA-2316/04 R 2004 0948 6 2004
							0	0.02	< 0.01	
							3	0.01	< 0.01	
							7	0.01	< 0.01	
							14	< 0.01	< 0.01	
France Haute-Normandie	Romulus	480 SC	3	0.048		300	0*	0.02	< 0.01	RA-2316/04 R 2004 0945 1 2004
							0	0.06	< 0.01	
							3	0.05	< 0.01	
							7	0.06	< 0.01	
							14	0.04	< 0.01	
Germany (Nordrhein-Westfalen	Brussels sprouts Igor F1	480 SC	3	0.048		500	0*	0.03	< 0.01	RA-2316/04 R 2004 0404 2 2004
							0	0.04	< 0.01	
							4	0.04	< 0.01	
							7	0.03	< 0.01	
							14	0.04	< 0.01	
Germany Nordrhein-Westfalen	Genius	480 SC	3	0.048		600	0*	0.05	< 0.01	RA-2316/04 R 2004 0944 3 2004
							0	0.05	< 0.01	
							3	0.04	< 0.01	
							7	0.10	< 0.01	
							14	0.07	< 0.01	
Germany Niedersachsen	Maximus	480 SC	3	0.048		300	0*	0.02	< 0.01	RA-2316/04 R 2004 0947 8 2004
							0	0.03	< 0.01	
							3	0.02	< 0.01	
							7	0.01	< 0.01	
							14	< 0.01	< 0.01	

Cucumber and summer squash

Flubendiamide is registered in USA for cucurbit vegetables at 3 × 0.05 kg ai/ha and 1 day PHI. Eleven residue field trials were conducted with flubendiamide in/on cucumber and summer squash in the USA (Table 58) and fifteen greenhouse trials were conducted in cucumber in Europe (Table 59).

Table 58 Results of residue field trials conducted with flubendiamide 24 WG in/on cucumber and summer squash in USA in 2004 (RCAMY015)

State	Variety	Application		PHI	Residues (mg/kg)[a]		Trial No.
		No	kg ai/ha		Flubendiamide	F-des-iodo	
Cucumber							
Georgia	Thunder	5	0.05	1	0.01	< 0.01	AM223-04H
N. Carolina	Poinsett 76	5	0.05	1	0.03	< 0.01	AM224-04H
Florida	Marketmore 76	5	0.05-0.06	1	< 0.01	< 0.01	AM225-04H
Kansas		5	0.05-0.06	0 1 3 7 12	0.03 0.03 0.03 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	AM226-04D
Illinois	SMR58	5	0.05-0.06	1	< 0.01	< 0.01	AM227-04H
Oklahoma	Straight 8	5	0.05	1	0.01	< 0.01	AM228-04H
Squash, summer							
New York	Zucchini Select	5	0.05	1	0.02	< 0.01	AM235-04H
Georgia	Destiny III	5	0.05	0 1 3 7 10	0.02 0.01 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	AM236-04D
Florida	Early Straightneck	5	0.05-0.06	1	< 0.01	< 0.01	AM237-04H
Nebraska	Zucchini Elite	5	0.05	1	0.01	< 0.01	AM238-04H
California	Green Eclipse	5	0.05	1	0.04	< 0.01	AM239-04H

[a] highest of two samples

Table 59 Results of residue trials conducted with flubendiamide WG in/on greenhouse grown cucumber in Europe

Country Year	Aplication			PHI	Residues (mg/kg)[a]		
	No	kg ai/ha	kg ai/hL (L/ha)		Flubendiamide	F-des-iodo	Report No. Trial No.
France 2005	3	0.1-0.12	(1275-1500)	0 1	0.07 0.07	< 0.01 < 0.01	RA-2514/05 R 2005 0268/0 M-277599-01-1
France 2006	2	0.12	0.008 (1500)	0* 0 1 2 7	0.02 0.07 0.08 0.07 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2635/06 R 2006 0675/3 M-285521-01-1
Germany 2009	2	0.091	0.006 (1500)	0* 0 1	0.02 0.07 0.07	< 0.01 < 0.01 < 0.01	08-2018 08-2018-06 M-349890-01-1
Germany 2006	2	0.072-0.090	0.008 (900-1125)	0* 0 1	0.01 0.13 0.07	< 0.01 < 0.01 < 0.01	RA-2635/06 R 2006 0676/1 M-285521-01-1
Germany 2005	3	0.07-0.11	(900-1350)	0 1 3 8	0.05 0.04 0.03 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	RA-2514/05 R 2005 0267/2 M-277599-01-1
Germany 2006	2	0.12	0.008 (1500)	0* 0 1	0.02 0.05 0.06	< 0.01 < 0.01 < 0.01	RA-2635/06 R 2006 0648/6 M-285521-01-1
Greece 2009	2	0.09	0.006 (1500)	0* 0 1	< 0.01 0.05 0.04	< 0.01 < 0.01 < 0.01	08-2018 08-2018-05 M-349890-01-1
Italy 2009	2	0.09	0.006 (1500)	0* 0 1	< 0.01 0.06 0.05	< 0.01 < 0.01 < 0.01	08-2018 08-2018-03 M-349890-01-1

	Aplication				Residues (mg/kg)[a]		
Country Year	No	kg ai/ha	kg ai/hL (L/ha)	PHI	Flubendiamide	F-des-iodo	Report No. Trial No.
Italy 2005	3	0.90- 0.120	(1125-1500)	0 1	0.07 0.07	< 0.01 < 0.01	RA-2514/05 R 2005 0213/3 M-277599-01-1
Italy 2006	2	0.120	0.008 (1500)	0* 0 1 3 7	0.01 0.04 0.04 0.03 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2635/06 R 2006 0647/8 M-285521-01-1
Portugal 2009	2	0.091- 0.098	0.006 (1500-1611)	0* 0 1	0.04 0.09 0.08	< 0.01 < 0.01 < 0.01	08-2018 08-2018-04 M-349890-01-1
Spain 2005	3	0.06-0.11	0.008 (750-1350)	0 1 2 7	0.05 0.04 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01	RA-2514/05 R 2005 0211/7 M-277599-01-1
Spain 2006	2	0.11-0.12	0.008 (1410-1500)	0* 0 1 3 7	< 0.01 0.02 0.03 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2635/06 R 2006 0674/5 M-285521-01-1
Spain 2009	2	0.09	0.006 (1500)	0* 0 1	< 0.01 0.05 0.02	< 0.01 < 0.01 < 0.01	08-2018 08-2018-01 M-349890-01-1
Spain 2009	2	0.091- 0.096	0.006 (1500-1578)	0* 0 1	0.04 0.05 0.02	< 0.01 < 0.01 < 0.01	08-2018 08-2018-02 M-349890-01-1

* before the last application

Melons and watermelon

Flubendiamide is registered in USA for cucurbit vegetables at 3 × 0.05 kg ai/ha and 1 day PHI. Six residue field trials were conducted with flubendiamide in/on muskmelon in United States (Table 60) and 11 greenhouse trials were conducted in melon and watermelon in Europe (61).

Table 60 Residue field trials conducted with flubendiamide WG in/on muskmelon in USA in 2004 (RCAMY015)

	Crop	Application			Residues (mg/kg)[a]		Trial No.
State	Variety	No	kg ai/ha	PHI	flubendiamide	F-des iodo	
California	Archer F1	5	0.05	1	0.07	< 0.01	AM232-04H
California	Canary yellow	5	0.05	1	0.02	< 0.01	AM233-04H
California	Hales best Jumbo	5	0.05	1	0.05	< 0.01	AM234-04H
Florida	Hales Best Jumbo	5	0.05-0.06	1	0.04	< 0.01	AM229-04H
Nebraska	French orange	5	0.05	0 1 3 7 10	0.02 0.02 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	AM230-04D
Texas	Imperial 45	5	0.05	1	0.09	< 0.01	AM231-04H

[a]. highest of two samples

Table 61 Results of residue trials conducted with flubendiamide WG in/on melon and watermelon in greenhouse in Europe

		Application					Residues (mg/kg)[a]		
Country Year	Crop	No	kg ai/ha	kg ai/hL	PHI	Portion	flubendiamide	F-des iodo	Report No. Trial No.
France 2005	Melon	3	0.060	0.006	0	fruit	< 0.01	< 0.01	RA-2515/05 R 2005 0221/4 M-277602-02-1
					3	fruit	0.03	< 0.01	
					3	peel	0.03	< 0.01	
					3	pulp	0.04	< 0.01	
Italy 2006	Melon	2	0.060	0.006	0*	fruit	< 0.01	< 0.01	RA-2634/06 R 2006 0645/1 M-285548-01-1
					0	fruit	0.01	< 0.01	
					1	fruit	< 0.01	< 0.01	
					3	fruit	< 0.01	< 0.01	
					5	fruit	0.01	< 0.01	
					3	peel	0.01	< 0.01	
					3	pulp	< 0.01	< 0.01	
Italy 2006	Watermelon	2	0.060	0.006	0*	fruit	< 0.01	< 0.01	RA-2634/06 R 2006 0673/7 M-285548-01-1
					0	fruit	0.02	< 0.01	
					1	fruit	0.02	< 0.01	
					3	fruit	0.02	< 0.01	
					7	fruit	0.02	< 0.01	
					3	peel	0.04	< 0.01	
					3	pulp	< 0.01	< 0.01	
Italy 2006	Melon	2	0.060	0.006	0*	fruit	0.02	< 0.01	RA-2634/06 R 2006 0670/2 M-285548-01-1
					0	fruit	0.04	< 0.01	
					1	fruit	0.04	< 0.01	
					3	fruit	0.04	< 0.01	
					7	fruit	0.03	< 0.01	
					3	peel	0.08	< 0.01	
					3	pulp	< 0.01	< 0.01	
Portugal 2006	Melon	2	0.060	0.006	0*	fruit	< 0.01	< 0.01	RA-2634/06 R 2006 0671/0 M-285548-01-1
					0	fruit	0.01	< 0.01	
					1	fruit	< 0.01	< 0.01	
					3	fruit	< 0.01	< 0.01	
					7	fruit	0.01	< 0.01	
					3	peel	0.02	< 0.01	
					3	pulp	< 0.01	< 0.01	
Portugal 2005	Melon	3	0.060	0.006	0	fruit	0.04	< 0.01	RA-2515/05 R 2005 0220/6 M-277602-02-1
					1	fruit	0.02	< 0.01	
					4	fruit	0.03	< 0.01	
					7	fruit	0.03	< 0.01	
					4	peel	0.12	< 0.01	
					4	pulp	< 0.01	< 0.01	
Spain 2005	Watermelon	3	0.060	0.006	0	fruit	< 0.01	< 0.01	RA-2515/05 R 2005 0218/4 M-277602-02-1
					1	fruit	< 0.01	< 0.01	
					3	fruit	< 0.01	< 0.01	
					8	fruit	< 0.01	< 0.01	
					3	peel	0.01	< 0.01	
					3	pulp	< 0.01	< 0.01	
Spain 2005	Watermelon	3	0.060	0.006	0	fruit	0.01	< 0.01	RA-2515/05 R 2005 0219/2 M-277602-02-1
					3	fruit	0.01	< 0.01	
					3	peel	0.03	< 0.01	
					3	pulp	< 0.01	< 0.01	
Spain 2006	Melon	2	0.060	0.006	0*	fruit	< 0.01	< 0.01	RA-2634/06 R 2006 0644/3 M-285548-01-1
					0	fruit	< 0.01	< 0.01	
					1	fruit	< 0.01	< 0.01	
					3	fruit	< 0.01	< 0.01	
					7	fruit	< 0.01	< 0.01	
					3	peel	< 0.01	< 0.01	
					3	pulp	< 0.01	< 0.01	

Country Year	Crop	Application			PHI	Portion	Residues (mg/kg)[a]		Report No. Trial No.
		No	kg ai/ha	kg ai/hL			flubendiamide	F-des iodo	
Spain 2006	Melon	2	0.060	0.006	0*	fruit	< 0.01	< 0.01	RA-2634/06 R 2006 0863/2 M-285548-01-1
					0	fruit	0.03	< 0.01	
					1	fruit	0.02	< 0.01	
					3	fruit	0.02	< 0.01	
					7	fruit	0.02	< 0.01	
					3	peel	0.05	< 0.01	
					3	pulp	< 0.01	< 0.01	
Spain 2006	Watermelon	2	0.060	0.006	0*	fruit	< 0.01	< 0.01	RA-2634/06 R 2006 0672/9 M-285548-01-1
					0	fruit	0.01	< 0.01	
					1	fruit	0.02	< 0.01	
					3	fruit	0.01	< 0.01	
					7	fruit	0.01	< 0.01	
					3	peel	0.02	< 0.01	
					3	pulp	< 0.01	< 0.01	

* prior to the last application

Peppers

Flubendiamide is registered in Australia in peppers at a maximum rate of 0.072 kg ai/ha (0.0072 kg ai/hL, in USA to be used in fruiting vegetables (except cucurbits) at a maximum rate of 3 × 0.05 kg ai/ha and in Greece and the Netherlands in greenhouse in pepper at 2 × 0.006 kg ai/hL (0.096 kg ai/ha) and 1 day PHI. Thirty five residue field trials were conducted in Australia and the USA with flubendiamide in/on peppers (Table 62) and fourteen greenhouse trials were conducted in Europe (Table 63).

Table 62 Results of residue field trials conducted with flubendiamide in pepper

Country Region	Variety	Aplication				PHI	Residues (mg/kg)[a]		Report No. Trial No.
		F	No	kg ai/ha	kg ai/hL (L/ha)		Flubendia-mide	F-des-iodo	
AUS Queensland	Capsicum Warlock	240 WG	3	0.081		0*	0.08	< 0.02	BCS-0085 Q1 2007
						0	0.12	< 0.02	
						1	0.04	< 0.02	
						3	0.06	< 0.02	
						7	0.02	< 0.02	
AUS Queensland	Capsicum Warlock	240 WG	3	0.049		0*	0.04	< 0.02	BCS-0085 Q1 2007
						0	0.10	< 0.02	
						1	0.06	< 0.02	
						3	< 0.02	< 0.02	
						7	0.05	< 0.02	
AUS Queensland	Capsicum Warlock	240 WG	3	0.12		0*	0.10	< 0.02	BCS-0085 Q1 2007
						0	0.37	< 0.02	
						1	0.37	< 0.02	
						3	0.04	< 0.02	
						7	0.04	< 0.02	
AUS Queensland	Capsicum Aries	240 WG	3	0.072		0*	< 0.02	< 0.02	BCS-0085 Q2 2007
						0	0.03	< 0.02	
						1	0.04	< 0.02	
						4	0.03	< 0.02	
						7	< 0.02	< 0.02	
AUS Queensland	Capsicum Aries	240 WG	3	0.040		0*	< 0.02	< 0.02	BCS-0085 Q2 2007
						0	0.02	< 0.02	
						1	0.03	< 0.02	
						4	0.02	< 0.02	
						7	< 0.02	< 0.02	

Country Region	Variety	Aplication				PHI	Residues (mg/kg)[a]		Report No. Trial No.
		F	No	kg ai/ha	kg ai/hL (L/ha)		Flubendia-mide	F-des-iodo	
AUS Queensland	Capsicum Aries	240 WG	3	0.11		0*	0.03	< 0.02	BCS-0085
						0	0.04	< 0.02	Q2
						1	0.15	< 0.02	2007
						4	0.05	< 0.02	
						7	0.09	< 0.02	
AUS Queensland	Capsicum El Charro	240 WG	3	0.048		0*	< 0.02	< 0.02	BCS-0109
						0	< 0.02	< 0.02	C109
						3	0.50	< 0.02	2007
						7	< 0.02	< 0.02	
						14	0.03	< 0.02	
AUS Queensland	Capsicum El Charro	240 WG	3	0.072		0*	0.03	< 0.02	BCS-0109
						0	0.05	< 0.02	C109
						3	0.05	< 0.02	2007
						7	0.04	< 0.02	
						14	0.06	< 0.02	
AUS Queensland	Capsicum El Charro	240 WG	3	0.11		0*	0.11	< 0.02	BCS-0109
						0	0.04	< 0.02	C109
						3	0.10	< 0.02	2007
						7	0.07	< 0.02	
						14	0.03	< 0.02	
AUS Queensland	Capsicum Hercules	480 SC	3	0.048		0*	0.02	< 0.02	BCS-0121
						0	0.06	< 0.02	C192
						1	0.04	< 0.02	2007
						3	0.03	< 0.02	
						7	0.07	< 0.02	
						14	0.22	< 0.02	
AUS Queensland	Capsicum Hercules	480 SC	3		0.005	0*	0.02	< 0.02	BCS-0121
						0	0.06	< 0.02	C192
						1	0.03	< 0.02	2007
						3	0.04	< 0.02	
						7	0.70	< 0.02	
						14	0.17	< 0.02	
AUS Queensland	Capsicum Hercules	480 SC	3		0.009	0*	0.12	< 0.02	BCS-0121
						0	0.08	< 0.02	C192
						1	0.11	< 0.02	2007
						3	0.02	< 0.02	
						7	0.16	< 0.02	
						14	0.07	< 0.02	
AUS Queensland	Capsicum Hercules	480 SC	3		0.014	0*	0.07	< 0.02	BCS-0121
						0	0.09	< 0.02	C192
						1	0.11	< 0.02	2007
						3	0.12	< 0.02	
						7	0.24	< 0.02	
						14	0.04	< 0.02	
AUS Queensland	Capsicum Hercules	24 WG	3		0.009	0*	0.05	< 0.02	BCS-0121
						0	0.05	< 0.02	C192
						1	0.06	< 0.02	2007
						3	0.09	< 0.02	
						7	0.05	< 0.02	
						14	0.07	< 0.02	
AUS Queensland	Capsicum Hercules	480 SC	3	0.048		0*	< 0.02	< 0.02	BCS-0121
						0	0.19	< 0.02	C193
						1	0.08	< 0.02	2007
						3	0.07	< 0.02	
						7	0.14	< 0.02	
						14	0.12	< 0.02	
AUS Queensland	Capsicum Hercules	480 SC	3		0.005	0*	< 0.02	< 0.02	BCS-0121
						0	0.10	< 0.02	C193
						1	0.05	< 0.02	2007
						3	0.09	< 0.02	
						7	0.09	< 0.02	
						14	0.08	< 0.02	

Country Region	Variety	Aplication				PHI	Residues (mg/kg)[a]		Report No. Trial No.
		F	No	kg ai/ha	kg ai/hL (L/ha)		Flubendiamide	F-des-iodo	
AUS Queensland	Capsicum Ingot	480 SC	3		0.009	0*	0.03	< 0.02	BCS-0121 C193 2007
						0	0.08	< 0.02	
						1	0.12	< 0.02	
						3	0.21	< 0.02	
						7	0.12	< 0.02	
						14	0.19	< 0.02	
AUS Queensland	Capsicum Hercules	480 SC	3		0.014	0*	0.08	< 0.02	BCS-0121 C193 2007
						0	0.44	< 0.02	
						1	0.23	< 0.02	
						3	0.17	< 0.02	
						7	0.12	< 0.02	
						14	0.14	< 0.02	
AUS Queensland	Capsicum Hercules	24 WG	3		0.009	0*	0.07	< 0.02	BCS-0121 C193 2007
						0	0.16	< 0.02	
						1	0.18	< 0.02	
						3	0.27	< 0.02	
						7	0.37	< 0.02	
						14	0.16	< 0.02	
AUS Queensland	Capsicum Tycoon	480 SC	3	0.048		0*	< 0.02	< 0.02	BCS-0121 C194 2007
						0	0.02	< 0.02	
						1	< 0.02	< 0.02	
						3	< 0.02	< 0.02	
						7	< 0.02	< 0.02	
						14	0.03	< 0.02	
AUS Queensland	Capsicum Tycoon	480 SC	3		0.005	0*	0.06	< 0.02	BCS-0121 C194 2007
						0	0.03	< 0.02	
						1	0.04	< 0.02	
						3	0.04	< 0.02	
						7	0.05	< 0.02	
						14	0.03	< 0.02	
AUS Queensland	Capsicum Tycoon	480 SC	3		0.010	0*	0.04	< 0.02	BCS-0121 C194 2007
						0	0.05	< 0.02	
						1	0.05	< 0.02	
						3	0.09	< 0.02	
						7	0.03	< 0.02	
						14	0.02	< 0.02	
AUS Queensland	Capsicum Tycoon	480 SC	3		0.014	0*	0.09	< 0.02	BCS-0121 C194 2007
						0	0.15	< 0.02	
						1	0.09	< 0.02	
						3	0.12	< 0.02	
						7	0.12	< 0.02	
						14	0.06	< 0.02	
AUS Queensland	Capsicum Tycoon	24 WG	3		0.01	0*	0.04	< 0.02	BCS-0121 C194 2007
						0	0.06	< 0.02	
						1	0.05	< 0.02	
						3	0.07	< 0.02	
						7	0.05	< 0.02	
						14	0.04	< 0.02	
USA Georgia	Pepper, sweet Capestrano	24 WG	5	0.050		0	0.05	< 0.01	RCAMY006 AM138-04D 2004
						1	0.04	< 0.01	
						3	0.04	< 0.01	
						7	0.04	< 0.01	
						10	0.02	< 0.01	
USA Florida	Pepper, sweet Olympus	24 WG	5	0.05	(125-136)	1	0.04	< 0.01	RCAMY006 AM139-04H 2004
USA Nebraska	Pepper, sweet Big Bertha	24 WG	5	0.05	(133)	1	0.05	< 0.01	RCAMY006 AM140-04H 2004
USA Oklahoma	Pepper, sweet Jupiter	24 WG	5	0.05	(103-133)	1	0.010	< 0.01	RCAMY006 AM141-04H 2004

Country Region	Variety	Aplication				PHI	Residues (mg/kg)[a]		Report No. Trial No.
		F	No	kg ai/ha	kg ai/hL (L/ha)		Flubendia-mide	F-des-iodo	
USA California	Pepper, sweet Affinity	24 WG	5	0.05	(168-175)	1	0.09	< 0.01	RCAMY006 AM142-04H 2004
USA California	Pepper, sweet Baron	24 WG	5	0.05	(142)	1	0.12	< 0.01	RCAMY006 AM143-04H 2004
USA North Carolina	Pepper, sweet Capistrano	24 WG	5	0.05	(147)	1	< 0.01	< 0.01	RAAML002 AM059-07HA 2007
USA California	Pepper, sweet Crusader	24 WG	5	0.05	(138-149)	1	< 0.01	< 0.01	RAAML002 AM060-07HA 2007
USA North Carolina	Pepper, cayenne-	24 WG	5	0.05	(142-14)	1	0.02	< 0.01	RCAMY006 AM144-04H 2004
USA Florida	Pepper, cayenne-	24 WG	5	0.05	(139-131)	1 1	0.10	< 0.01	RCAMY006 AM145-04H 2004
USA California	Pepper, cayenne-	24 WG	5	0.05	(174)	1	0.14	< 0.01	RCAMY006 AM146-04H 2004

[a]. Residues from trials conducted in USA are the highest of two samples

Table 63 Results of residue trials conducted with flubendiamide in/on pepper in greenhouse in Europe

Country Region	Aplication				PHI	Residues (mg/kg)[a]		Report No. Trial No.
	F	No	kg ai/ha	kg ai/hL (L/ha)		Flubendia-mide	F-des-iodo	
France 2003	SC 480	3	0.05	0.006 (900)	0* 0 0** 1 3 5	0.11 0.17 0.11 0.11 0.06 0.07	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2070/03 R 2003 0831/0 M-248645-01-1
France 2004	SC 480	3	0.05-0.06	0.006 (750-1000)	0 0** 1 3	0.11 0.16 0.08 0.06	< 0.01 < 0.01 < 0.01 < 0.01	RA-2311/04 R 2003 0902/8 M-252025-01-1
Germany 2003	SC 480	3	0.04	0.006 (700)	0* 0 0** 1 3 5	0.07 0.13 0.16 0.13 0.11 0.11	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2070/03 R 2003 0198/7 M-248645-01-1
Germany 2004	SC 480	3	0.04-0.05	0.006 (525-600)	0 0** 1 3	0.18 0.14 0.10 0.10	< 0.01 < 0.01 < 0.01 < 0.01	RA-2311/04 R 2003 0314/3 M-252025-01-1
Italy 2003	SC 480	3	0.06	0.006 (1000)	0* 0 0** 1 3 5	0.10 0.16 0.09 0.13 0.09 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2070/03 R 2003 0833/7 M-248645-01-1
Netherlands 2004	SC 480	3	0.11-0.12	0.006 (1800-1900)	0 0** 1 3	0.10 0.08 0.08 0.07	< 0.01 < 0.01 < 0.01 < 0.01	RA-2311/04 R 2003 0899/4 M-252025-01-1

	Aplication					Residues (mg/kg)[a]		
Country Region	F	No	kg ai/ha	kg ai/hL (L/ha)	PHI	Flubendia-mide	F-des-iodo	Report No. Trial No.
Netherlands 2003	SC 480	3	0.09- 0.10	0.006 (1500-1700)	0* 0 0** 1 3 5	0.03 0.05 0.04 0.04 0.04 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2070/03 R 2003 0832/9 M-248645-01-1
Netherlands 2006	WG 24	2	0.12	0.008 (1500)	0* 0 1 3 7	0.03 0.11 0.06 0.05 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2656/06 R 2006 0891/8 M-285884-01-1
Netherlands 2006	WG 24	2	0.12	0.008 (1500)	0* 0 1 3 7	0.03 0.08 0.07 0.04 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2656/06 R 2006 0892/6 M-285884-01-1
Portugal 2003	SC 480	3	0.06- 0.08	0.008 (750-975)	0* 0 0** 1 3 5	0.09 0.14 0.15 0.11 0.09 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2070/03 R 2003 0835/3 M-248645-01-1
Portugal 2006	WG 24	2	0.60	0.008 (750)	0* 0 1 3 7	0.02 0.07 0.03 0.05 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2656/06 R 2006 0893/4 M-285884-01-1
Spain 2003	SC 480	3	0.07	0.006 (1150-1250)	0* 0 0** 1 3 5	0.15 0.16 0.12 0.10 0.09 0.09	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2070/03 R 2003 0834/5 M-248645-01-1
Spain 2004	SC 480	3	0.08- 0.10	0.006 (1300-1600)	0 1 3	0.09 0.09 0.05	< 0.01 < 0.01 < 0.01	RA-2311/04 R 2003 0900/1 M-252025-01-1
Spain 2006	WG 24	2	0.120	0.008 (1500)	0* 0 1 3 7	0.06 0.12 0.09 0.11 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2656/06 R 2006 0889/6 M-285884-01-1

* Before the last application;

** sampling on day 0.5 after last treatment;

Tomatoes

Flubendiamide is registered in Australia for use in tomatoes at a maximum rate of 0.072 kg ai/ha (0.0072 kg ai/hL, and in the USA for use in fruiting vegetables (except cucurbits) at a maximum rate of 3 × 0.05 kg ai/ha and in Greece and the Netherlands in greenhouse grown tomatoes at 2 × 0.006 kg ai/hL (0.096 kg ai/ha) and 3 and 1 day PHI, respectively. Fourty two field trials were conducted with flubendiamide in tomatoes in Australia and USA (Table 64) and fifteen greenhouse trials were conducted in Europe (Table 65).

Table 64 Results of residue trials conducted with flubendiamide SC480 in/on tomato

Country Region	Variety	Aplication					PHI	Residues (mg/kg)^{a}		Report No. Trial No.
		F	No	kg ai/ha	kg ai/hL	L/ha		Flubendia-mide	F-des-iodo	
AUS Rochester Victoria	Heinz 599	24 WG	3	0.048			0* 0 1 3 8	0.15 0.18 0.12 0.17 0.06	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0084 VC31 2007
AUS Rochester Victoria	Heinz 599	24 WG	3	0.072			0* 0 1 3 8	0.12 0.34 0.24 0.35 0.28	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0084 VC31 2007
AUS Rochester Victoria	Heinz 599	24 WG	3	0.106			0* 0 1 3 8	0.18 0.23 0.19 0.55 0.66	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0084 VC31 2007
AUS Rochester Victoria	Heinz 599	24 WG	3		0.0048		0* 0 1 3 8	0.10 0.33 0.28 0.18 0.19	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0084 VC31 2007
AUS Rochester Victoria	Heinz 599	24 WG	3		0.0072		0* 0 1 3 8	0.27 0.22 0.22 0.35 0.28	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0084 VC31 2007
AUS Rochester Victoria	Heinz 599	24 WG	3		0.0108		0* 0 1 3 8	0.23 1.7 0.97 0.60 0.55	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0084 VC31 2007
AUS Flagstone Creek, Queens-land	840	24 WG	3	0.048			0* 0 1 3 7	0.04 0.08 0.08 0.02 0.03	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0108 C108 2007
AUS Flagstone Creek, Queens-land	840	24 WG	3	0.072			0* 0 1 3 7	0.03 0.03 0.03 0.04 0.03	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0108 C108 2007
AUS Flagstone Creek, Queens-land	840	24 WG	3	0.108			0* 0 1 3 7	0.07 0.05 0.04 0.05 0.04	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0108 C108 2007
AUS Flagstone Creek, Queens-land	840	24 WG	3		0.0048		0* 0 1 3 7	0.05 0.04 < 0.02 0.03 0.03	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0108 C108 2007
AUS Flagstone Creek, Queens-land	840	24 WG	3		0.0072		0* 0 1 3 7	0.04 0.03 0.07 0.04 0.03	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0108 C108 2007
AUS Flagstone Creek, Queens-land	840	24 WG	3		0.0108		0* 0 1 3 7	0.09 0.13 0.10 0.07 0.24	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0108 C108 2007

		Aplication						Residues (mg/kg)[a]		Report No. Trial No.
Country Region	Variety	F	No	kg ai/ha	kg ai/hL	L/ha	PHI	Flubendiamide	F-des-iodo	
AUS Bowen Queens-land	Comanche	480 SC	3	0.096			0*	0.10	< 0.02	BCS-0120 C188 2007
							0	0.07	< 0.02	
							1	0.03	< 0.02	
							3	0.18	< 0.02	
							7	0.04	< 0.02	
							14	0.06	< 0.02	
AUS Bowen Queens-land	Comanche	480 SC	3		0.0048		0*	0.04	< 0.02	BCS-0120 C188 2007
							0	< 0.02	< 0.02	
							1	< 0.02	< 0.02	
							3	0.05	< 0.02	
							7	0.11	< 0.02	
							14	0.06	< 0.02	
AUS Bowen Queens-land	Comanche	480 SC	3		0.096		0*	0.12	< 0.02	BCS-0120 C188 2007
							0	0.12	< 0.02	
							1	0.05	< 0.02	
							3	0.11	< 0.02	
							7	0.10	< 0.02	
							14	0.15	< 0.02	
AUS Bowen Queens-land	Comanche	480 SC	3		0.014		0*	0.12	< 0.02	BCS-0120 C188 2007
							0	0.13	< 0.02	
							1	0.05	< 0.02	
							3	0.10	< 0.02	
							7	0.05	< 0.02	
							14	0.17	< 0.02	
AUS Bowen Queens-land	Comanche	240 WG	3		0.096		0*	0.04	< 0.02	BCS-0120 C188 2007
							0	0.03	< 0.02	
							1	0.09	< 0.02	
							3	0.06	< 0.02	
							7	0.08	< 0.02	
							14	0.16	< 0.02	
AUS Bowen Queens-land	Pinnacle	480 SC	3	0.096			0*	0.17	< 0.02	BCS-0120 C189 2007
							0	0.21	< 0.02	
							1	0.19	< 0.02	
							3	0.10	< 0.02	
							7	0.23	< 0.02	
							14	0.27	< 0.02	
AUS Bowen Queens-land	Pinnacle	480 SC	3		0.0048		0*	0.03	< 0.02	BCS-0120 C189 2007
							0	0.08	< 0.02	
							1	0.06	< 0.02	
							3	0.07	< 0.02	
							7	0.04	< 0.02	
							14	0.10	< 0.02	
AUS Bowen Queens-land	Pinnacle	480 SC	3		0.0096		0*	0.10	< 0.02	BCS-0120 C189 2007
							0	0.14	< 0.02	
							1	0.07	< 0.02	
							3	0.15	< 0.02	
							7	0.07	< 0.02	
							14	0.17	< 0.02	
AUS Bowen Queens-land	Pinnacle	480 SC	3		0.014		0*	0.12	< 0.02	BCS-0120 C189 2007
							0	0.16	< 0.02	
							1	0.19	< 0.02	
							3	0.14	< 0.02	
							7	0.10	< 0.02	
							14	0.15	< 0.02	
AUS Bowen Queens-land	Pinnacle	240 WG	3		0.0096		0*	0.09	< 0.02	BCS-0120 C189 2007
							0	0.07	< 0.02	
							1	0.08	< 0.02	
							3	0.11	< 0.02	
							7	0.05	< 0.02	
							14	0.27	< 0.02	

		Aplication						Residues (mg/kg)[a]		Report No. Trial No.
Country Region	Variety	F	No	kg ai/ha	kg ai/hL	L/ha	PHI	Flubendiamide	F-des-iodo	
AUS Bowen Queensland	Guardian	480 SC	3	0.096			0* 0 1 3 7 14	< 0.02 0.02 0.03 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0120 C191 2007
AUS Bowen Queensland	Guardian	480 SC	3		0.0048		0* 0 1 3 7 14	0.02 0.03 < 0.02 < 0.02 < 0.02 0.03	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0120 C191 2007
AUS Bowen Queensland	Guardian	480 SC	3		0.0096		0* 0 1 3 7 14	0.16 0.05 0.03 0.05 0.04 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0120 C191 2007
AUS Bowen Queensland	Guardian	480 SC	3		0.014		0* 0 1 3 7 14	0.23 0.06 0.08 0.04 0.05 0.04	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0120 C191 2007
AUS Bowen Queensland	Guardian	240 WG	3		0.0096		0* 0 1 3 7 14	0.02 0.04 < 0.02 0.02 0.03 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0120 C191 2007
Aus Carpendale Queensland		240 WG	3		0.0048		0* 0 1 3 7	0.05 0.88 0.15 0.12 0.13	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0120 Q1 2007
Aus Carpendale Queensland		240 WG	3		0.0072		0* 0 1 3 7	0.39 0.32 0.21 0.52 0.63	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0120 Q1 2007
Aus Carpendale Queensland		240 WG	3		0.0108		0* 0 1 3 7	0.85 0.86 0.28 0.83 1.4	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0120 Q1 2007
USA Pensilvania	Celebrity	24G	5	0.05			1	0.09	< 0.01	RCAMY006 AM126-04H 2004
USA Georgia	Homestead	24G	5	0.05			1	0.05	< 0.01	RCAMY006 AM127-04H 2004
USA Florida	FL 47	24G	5	0.05			1	0.04	< 0.01	RCAMY006 AM128-04H 2004
USA Florida	FL 47	24G	5	0.05			1	0.04	< 0.01	RCAMY006 AM129-04H 2004
USA Kansas	Rutgers	24 WG	5	0.05		127-146	0 1 3 7 10	0.04 0.03 0.03 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RCAMY006 AM130-04D 2004

Country Region	Variety	Aplication					PHI	Residues (mg/kg)[a]		Report No. Trial No.
		F	No	kg ai/ha	kg ai/hL	L/ha		Flubendia-mide	F-des-iodo	
USA California	Peto Hypeel 303	24 WG	5	0.05		170-174	1	0.18	< 0.01	RCAMY006 AM131-04H 2004
USA Glenn, California	H9557	24 WG	5	0.05		139-141	1	0.14	< 0.01	RCAMY006 AM132-04H 2004
USA Maxwell, California	H9888	24 WG	5	0.05		140-141	1	0.09	< 0.01	RCAMY006 AM133-04H 2004
USA Porterville, California	Early Girl	24 WG	5	0.05		172-182	1	0.08	< 0.01	RCAMY006 AM134-04H 2004
USA Visalia, California	Qualit-21	24 WG	5	0.05		130-135	1	0.07	< 0.01	RCAMY006 AM135-04H 2004
USA Fresno, California	Roma	24 WG	5	0.05		115-119	1	0.07	< 0.01	RCAMY006 AM136-04H 2004
USA Paso Robles, California	Roma	24 WG	5	0.05		137-146	1	0.01	< 0.01	RCAMY006 AM137-04H 2004

* Before the last application; a. Residues from trials conducted in USA are the highest of two samples

Table 65 Results of residue trials conducted with flubendiamide in/on tomato in greenhouse in Europe

Country Region year	Aplication				PHI	Residues (mg/kg)[a]		Report No. Trial No
	F	No	kg ai/ha	kg ai/hL (L/ha)		Flubendia-mide	F-des-iodo	
France 2004	SC 480	3	0.12	0.008 (1500)	0* 0 1 3	0.14 0.12 0.13 0.07	< 0.01 < 0.01 < 0.01 < 0.01	RA-2312/04 R 2004 0937/0 M-255741-01-1
Germany 2003	SC 480	3	0.12	0.008 (1500)	0* 0 0^{**} 1 3 5	0.05 0.13 0.04 0.02 0.02 0.03	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2071/03 R 2003 0199/5 M-246583-01-1
Germany 2005	WG)	3	0.12	0.012 (1000)	0 1 3	0.06 0.04 0.03	< 0.01 < 0.01 < 0.01	RA-2517/05 R2005 0224/9 M-276592-01-1
Germany 2008	SC 480	2	0.12	0.006 (2000)	0* 0 1 3 7	0.03 0.10 0.09 0.08 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	08-2213 08-2213-04 M-344411-01-1
Italy 2003	SC 480	3	0.11	0.0077 (1425)	0* 0 0** 1 3 5	0.11 0.14 0.11 0.11 0.11 0.12	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2071/03 R 2003 0199/5 M-246583-01-1
Italy 2004	SC 480	3	0.12	0.0075 (1600)	0* 0 1 3	0.06 0.07 0.07 0.04	< 0.01 < 0.01 < 0.01 < 0.01	RA-2312/04 R2004 0936/2 M-255741-01-1
Italy 2005	WG 24	3	0.12	0.08 (1500)	0 1 3	0.06 0.12 0.09	< 0.01 < 0.01 < 0.01	RA-2517/05 R2005 0269/9 M-276592-01-1

Country Region year	Aplication				PHI	Residues (mg/kg)[a]		Report No. Trial No
	F	No	kg ai/ha	kg ai/hL (L/ha)		Flubendia-mide	F-des-iodo	
Italy 2004	SC 480	3	0.12	0.075 (1600)	0* 0 1 3	0.06 0.07 0.07 0.04	< 0.01 < 0.01 < 0.01 < 0.01	RA-2312/04 R 2004 0936/2 M-255741-01-1
Italy* 2004	WG 24	3	0.12	0.075 (1600)	0* 0 1 3	0.08 0.06 0.07 0.11	< 0.01 < 0.01 < 0.01 < 0.01	RA-2326/04 R2004 0966/4 M-255744-01-1
Netherlands 2003	SC 480	3	0.12	0.006 (2000)	0* 0 0** 1 3 5	0.04 0.06 0.03 0.04 0.02 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2071/03 R 2003 0836/1 M-246583-01-1
Netherlands 2003	SC 480	3	0.12	0.008 (1500)	0* 0 0** 1 3 5	0.05 0.08 0.05 0.05 0.06 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2071/03 R 2003 0838/8 M-246583-01-1
Netherlands 2004	SC 480	3	0.12	0.006 (2000)	0 0[a] 1 3	0.07 0.09 0.06 0.06	< 0.01 < 0.01 < 0.01 < 0.01	RA-2312/04 R 2004 0313/5 M-255741-01-1
Portugal 2003	SC 480	3	0.12	0.007 (1600- 1800)	0* 0 0[d] 1 3 5	0.07 0.11 0.10 0.10 0.11 0.10	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RA-2071/03 R 2003 0842/6 M-246583-01-1
Portugal 2008	SC 480	2	0.12	0.006 (2000)	0* 0 1 3 7	0.09 0.07 0.13 0.10 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	08-2213 08-2213-03 M-344411-01-1
Spain 2004	SC 480	3	0.102	0.006 (1700)	0* 0 1 2	0.07 0.10 0.09 0.10	< 0.01 < 0.01 < 0.01 < 0.01	RA-2312/04 R2004 0935/4 M-255741-01-1
Spain* 2004	WG 24	3	0.12	0.006 (1700)	0* 0 1 2	0.08 0.09 0.08 0.09	< 0.01 < 0.01 < 0.01 < 0.01	RA-2326/04 R2004 0967/2 M-255744-01-1
Spain 2008	SC 480	2	0.12	0.006 (2000)	0* 0 1 3 6	0.03 0.06 0.05 0.04 0.06	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	08-2213 08-2213-01 M-344411-01-1
Spain 2008	SC 480	2	0.12	0.006 (2000)	0* 0 1 3 7	0.03 0.05 0.04 0.06 0.05	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	08-2213 08-2213-02 M-344411-01-1

* Before the last application; ** sampling on day 0.5 after last treatment.

Sweet corn

Flubendiamide is registered in USA in sweet corn at a maximum rate of 4 × 0.10 kg ai/ha and 1 day PHI. Eleven residue trials were conducted with sweet corn in USA in 2004/2005, with samples of corn-on-the-cob analysed. The results are on Table 66.

Table 66 Results of residue trials conducted with flubendiamide SC480 in/on sweet corn (corn-on-the-cob) in USA (RCAMY004)

Country Year	Crop	Application			PHI	Residues (mg/kg)^{a}		Trial No.
		No	kg ai/ha	L/ha water		flubendiamide	F-des iodo	
New York	Garst 8948RR	4	0.11	155-158	1	< 0.01	< 0.01	AM100-04H-B
Virginia	Silver Queen	4	0.10-0.11	130-134	1	< 0.01	< 0.01	AM101-04H
Georgia	RR31N26	4	0.10-0.11	141-148	1	< 0.01	< 0.01	AM102-04H-B
Florida	Incredible				0	< 0.01	< 0.01	
					1	< 0.01	< 0.01	
					3	< 0.01	< 0.01	
					7	0.01	< 0.01	
					10	0.01	< 0.01	
Kansas	Garst 8451RR	4	0.10-0.11	125-146	2	< 0.01	< 0.01	AM104-04D-B
Illinois	Garst 8550 Bt	4	0.11	130-134	1	< 0.01	< 0.01	AM105-04H-B
Nebraska	NK67T4	4	0.10	131-133	1	< 0.01	< 0.01	AM106-04D-B
Minnesota	GH0937	4	0.11	106-117	1	< 0.01	< 0.01	AM107-04H-B
California	Silver Queen	4	0.10-0.11	134-106	1	< 0.01	< 0.01	AM122-04HA
Oregon	Pronto	4	0.10-0.11	0.13-0.14	1	< 0.01	< 0.01	AM123-04H
Oregon	Super Sweet Jubilee	4	0.10-0.11	78-81	1	< 0.01	< 0.01	AM124-04H

Lettuce

Flubendiamide is registered in Australia for leafy vegetables, including leafy and head lettuce, at a maximum rate of 3 × 0.048 kg ai/ha and 1 day PHI. Thirthy six residue field trials were conducted with flubendiamide in/on head and leafy lettuce in Australia in 2006 (Table 67).

Table 67 Results of residue trials conducted with flubendiamide SC in/on head and leafy lettuce in Australia

Region	Variety	Application			PHI	Residues (mg/kg)		Trial No.
		No	kg ai/ha	L/ha water		flubendiamide	F-des iodo	
Head lettuce								
West Victoria	Lulu	3	0.046	903	0*	0.08	< 0.02	BCS-0187 B71 ** 2006
					0	1.1	< 0.02	
					1	0.32	< 0.02	
					3	0.16	< 0.02	
					7	< 0.02	< 0.02	
					10	< 0.02	< 0.02	
			0.066	903	0*	0.11	< 0.02	
					0	4.0	< 0.02	
					1	1.6	< 0.02	
					3	0.24	< 0.02	
					7	< 0.02	< 0.02	
					10	< 0.02	< 0.02	
			0.091	903	0*	0.07	< 0.02	
					0	3.0	< 0.02	
					1	0.77	< 0.02	
					3	0.12	< 0.02	
					7	< 0.02	< 0.02	
					10	< 0.02	< 0.02	
Queensland	Patagonia	3	0.048-0.052	353	0*	0.14	< 0.02	BCS-0187 B72 2006
					0	0.48	< 0.02	
					1	0.86	< 0.02	
					3	0.68	< 0.02	
					7	0.97	< 0.02	
					10	0.49	< 0.02	

		Application				Residues (mg/kg)		Trial No.
Region	Variety	No	kg ai/ha	L/ha water	PHI	flubendiamide	F-des iodo	
			0.072-0.079	353	0*	0.25	< 0.02	
					0	2.0	< 0.02	
					1	2.6	< 0.02	
					3	0.64	< 0.02	
					7	0.82	< 0.02	
					10	0.68	< 0.02	
			0.096-0.105	353	0*	1.4	< 0.02	
					0	1.4	< 0.02	
					1	1.5	< 0.02	
					3	2.2	< 0.02	
					7	3.9	< 0.02	
					10	1.6	< 0.02	
Victoria	Ice	3	0.049-0.051	500	0*	1.0	< 0.02	BCS-0187 B73 2006
					0	2.2	< 0.02	
					1	2.2	< 0.02	
					3	1.5	< 0.02	
					7	0.26	< 0.02	
					10	0.12	< 0.02	
			0.071-0.074	500	0*	1.3	< 0.02	
					0	2.2	< 0.02	
					1	3.1	< 0.02	
					3	2.0	< 0.02	
					7	0.78	< 0.02	
					10	0.25	< 0.02	
			0.094-0.097	500	0*	1.5	< 0.02	
					0	5.4	< 0.02	
					1	4.7	< 0.02	
					3	3.6	< 0.02	
					7	1.0	< 0.02	
					10	0.19	< 0.02	
West Victoria	Lulu	3	0.046	903	0*	< 0.02	< 0.02	BCS-0188 B99 ** 2006
					0	0.44	< 0.02	
					1	0.11	< 0.02	
					3	0.16	< 0.02	
					7	< 0.02	< 0.02	
					10	0.05	< 0.02	
			0.068	903	0*	0.03	< 0.02	
					0	0.44	< 0.02	
					1	0.17	< 0.02	
					3	0.85	< 0.02	
					7	0.65	< 0.02	
					10	0.02	< 0.02	
			0.091	903	0*	0.11	< 0.02	
					0	0.20	< 0.02	
					1	0.18	< 0.02	
					3	0.06	< 0.02	
					7	0.04	< 0.02	
					10	0.13	< 0.02	
Queensland	Desert Sun	3	0.046	318	0*	0.08	< 0.02	BCS-0188 B100 2006
					0	0.72	< 0.02	
					1	0.78	< 0.02	
					3	0.59	< 0.02	
					10	0.28	< 0.02	
			0.068	318	0*	0.08	< 0.02	
					0	2.1	< 0.02	
					1	0.73	< 0.02	
					3	0.84	< 0.02	
					10	0.32	< 0.02	
			0.091	318	0*	0.46	< 0.02	
					0	1.1	< 0.02	
					1	1.0	< 0.02	
					3	0.54	< 0.02	
					10	0.32	< 0.02	

Region	Variety	Application			PHI	Residues (mg/kg)		Trial No.
		No	kg ai/ha	L/ha water		flubendiamide	F-des iodo	
Victoria	Marksman	3	0.048	467	0*	0.28	< 0.02	BCS-0188 B101 2006
					0	1.8	< 0.02	
					1	1.0	< 0.02	
					3	1.0	< 0.02	
					7	0.42	< 0.02	
					10	0.12	< 0.02	
			0.07-0.074	467	0*	0.60	< 0.02	
					0	1.8	< 0.02	
					1	1.9	< 0.02	
					3	1.1	< 0.02	
					7	0.50	< 0.02	
					10	0.33	< 0.02	
			0.095-0.099	467	0*	0.71	< 0.02	
					0	4.3	< 0.02	
					1	1.1	< 0.02	
					3	1.7	< 0.02	
					7	0.31	< 0.02	
					10	0.12	< 0.02	
Leaf lettuce								
Victoria	Kos	3	0.045	903	0*	0.42	< 0.02	BCS-0182 B68 2006
					0	1.4	< 0.02	
					1	1.8	< 0.02	
					3	1.6	< 0.02	
					8	0.74	< 0.02	
					10	0.70	< 0.02	
			0.066	903	0*	0.74	< 0.02	
					0	4.0	< 0.02	
					1	2.8	< 0.02	
					3	2.1	< 0.02	
					8	1.3	< 0.02	
					10	0.82	< 0.02	
			0.091	903	0*	0.66	< 0.02	
					0	4.5	< 0.02	
					1	4.6	< 0.02	
					3	2.7	< 0.02	
					8	0.86	< 0.02	
					10	1.0	< 0.02	
Queensland	Cos	3	0.053	353	0*	1.0	< 0.02	BCS-0182 B69 2006
					0	4.4	< 0.02	
					1	1.2	< 0.02	
					3	1.6	< 0.02	
					7	1.0	< 0.02	
					10	0.53	< 0.02	
			0.072-0.079	353	0*	2.0	< 0.02	
					0	5.2	< 0.02	
					1	1.5	< 0.02	
					3	2.5	< 0.02	
					7	1.0	< 0.02	
					10	1.5	< 0.02	
			0.096-0.099	353	0*	3.0	< 0.02	
					0	5.5	< 0.02	
					1	2.3	< 0.02	
					3	3.5	< 0.02	
					7	3.3	< 0.02	
					10	1.3	< 0.02	
Victoria	Regal	3	0.047-0.049	500	0*	0.60	< 0.02	BCS-0182 B70 *** 2006
					0	3.2	< 0.02	
					1	2.7	< 0.02	
					3	1.3	< 0.02	
					7	0.40	< 0.02	
					10	0.12	< 0.02	

Region	Variety	Application			PHI	Residues (mg/kg)		Trial No.
		No	kg ai/ha	L/ha water		flubendiamide	F-des iodo	
			0.071-0.073	500	0*	0.99	< 0.02	
					0	5.2	< 0.02	
					1	4.4	< 0.02	
					3	2.0	< 0.02	
					7	0.54	< 0.02	
					10	0.15	< 0.02	
			0.094-0.099	500	0*	2.4	< 0.02	
					0	7.1	< 0.02	
					1	6.9	< 0.02	
					3	2.5	< 0.02	
					7	1.0	< 0.02	
					10	0.34	< 0.02	
Victoria	coral	3	0.046	903	0*	0.40	< 0.02	BCS-0189 B102 2006
					0	4.0	< 0.02	
					1	4.0	< 0.02	
					3	1.6	< 0.02	
					7	0.63	< 0.02	
					9	0.16	< 0.02	
			0.066	903	0*	0.71	< 0.02	
					0	5.2	< 0.02	
					1	6.9	< 0.02	
					3	3.5	< 0.02	
					7	1.2	< 0.02	
					9	0.32	< 0.02	
			0.091	903	0*	0.78	< 0.02	
					0	11.2	< 0.02	
					1	8.6	< 0.02	
					3	13.4	< 0.02	
					7	0.86	< 0.02	
					9	0.42	< 0.02	
Queensland	Shrek	3	0.048	318	0*	0.22	< 0.02	BCS-0189 B103 2006
					0	0.72	< 0.02	
					1	1.6	< 0.02	
					3	1.2	< 0.02	
					10	0.24	< 0.02	
		3	0.072	318	0*	0.42	< 0.02	
					0	1.3	< 0.02	
					1	1.5	< 0.02	
					3	1.3	< 0.02	
					10	0.56	< 0.02	
		3	0.096-0.113	318	0*	0.56	< 0.02	
					0	1.4	< 0.02	
					1	2.4	< 0.02	
					3	2.2	< 0.02	
					10	0.16	< 0.02	
Victoria	Regal	3	0.047-0.051	467	0*	0.46	< 0.02	BCS-0189 B104 2006
					0	1.5	< 0.02	
					1	0.95	< 0.02	
					3	0.43	< 0.02	
					7	0.42	< 0.02	
					10	0.19	< 0.02	
			0.070-0.075	467	0*	0.52	< 0.02	
					0	2.0	< 0.02	
					1	2.3	< 0.02	
					3	0.69	< 0.02	
					7	0.26	< 0.02	
					10	0.48	< 0.02	
			0.094-0.097	467	0*	0.69	< 0.02	
					0	2.2	< 0.02	
					1	2.7	< 0.02	
					3	0.79	< 0.02	
					7	0.41	< 0.02	
					10	0.44	< 0.02	

*** Residues of flubendiamide detected in untreated plots at 0.03 mg/kg

Spinach

Flubendiamide is registered in USA for leafy vegetables at 3 x 0.05 kg ai/ha and 1 day PHI. Five trials were conducted in USA in spinach (Table 68).

Table 68 Results of residue trials conducted with 5 applications of flubendiamide WG in/on spinach in USA in 2004 (RCAMY008)

		Application			Residues (mg/kg)[a]		
Country Year	Variety	kg ai/ha	L/ha water	PHI Days	flubendiamide	F-des iodo	Trial No.
New York	Melody	0.050	160-162	1	3.7	< 0.01	AM166-04D
Texas	Samich	0.050	141-145	1	6.7	< 0.01	AM168-04H
Idaho	Unipack 151	0.050	176-181	1	3.3	< 0.01	AM169-04H
California	Dolphin	0.050	145-151	1	3.1	< 0.01	AM170-04H
California	Shasta	0.050	140-143	1	5.9	< 0.01	AM165-04H

[a] highest residue of two samples

Green beans and peas

Flubendiamide is registered in Australia in legume vegetables at a maximum rate of 3 × 0.072 kg ai/ha and USA at 2 × 0.1 kg ai/ha. In both countries, PHI is 1 day. Fourty four residue field trials were conducted in grean bean and pea in Australia and the USA (Table 69).

Table 69 Results of residue trials conducted with flubendiamide SC in/on green bean and pea

		Application					Residues (mg/kg)[a]		
Country Year	Crop	No	kg ai/ha	L/ha water	Portion	PHI	flubendiamide	F-des iodo	Report No. Trial No. year
Australia Queensland	Green Bean, Field Excali-bur	3	0.046-0.051	372	pod	0*	0.04	< 0.02	BCS-0186 B84 2006
						0	0.16	< 0.02	
						1	0.11	< 0.02	
						3	0.10	< 0.02	
						7	0.15	< 0.02	
						10	0.18	< 0.02	
			0.072-0.074	372	pod	0*	0.04	< 0.02	
						0	0.24	< 0.02	
						1	0.18	< 0.02	
						3	0.16	< 0.02	
						7	0.22	< 0.02	
						10	0.21	< 0.02	
			0.096-0.102	372	pod	0*	0.05	< 0.02	
						0	0.28	< 0.02	
						1	0.25	< 0.02	
						3	0.24	< 0.02	
						7	0.26	< 0.02	
						10	0.30	< 0.02	
					stuble	0	14	0.07	
						1	14	0.07	
						3	15	0.08	
						7	15	0.09	
						10	9.9	0.06	
Australia Bulla, Queens-land	Green Bean, Field Jade	3	0.049-0.053	387-414	pod	0*	0.04	< 0.02	BCS-0186 B85 2006
						0	0.12	< 0.02	
						1	0.08	< 0.02	
						3	0.06	< 0.02	
						6	0.06	< 0.02	
						10	0.08	< 0.02	

		Application					Residues (mg/kg)[a]		
Country Year	Crop	No	kg ai/ha	L/ha water	Portion	PHI	flubendiamide	F-des iodo	Report No. Trial No. year
			0.077-0.082	387-414	pod	0* 0 1 3 6 10	0.09 0.20 0.20 0.16 0.15 0.14	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	
			0.100	387-414	pod	0* 0 1 3 6 10	0.10 0.30 0.30 0.19 0.22 0.81	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	
Australia Bulla, Queens-land	Green Bean, Field Excali-bur	3	0.044-0.050	761-789	pod	0* 0 1 3 8 10	0.06 0.17 0.10 0.18 0.08 0.10	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0186 B87 2007
			0.068-0.072	761-789	pod	0* 0 1 3 8 10	0.08 0.16 0.20 0.20 0.14 0.14	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	
			0.090-0.10	761-789	Pod	0* 0 1 3 8 10	0.08 0.26 0.26 0.21 0.12 0.19	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	
					stuble	0 1 3 8 10	11 12 11 8.6 8.7	0.05 0.05 0.08 0.05 0.05	
Australia Kindred, Tasmania	Green Bean, Field Classic	3	0.047-0.048	423	pod	0* 0 1 3 7 10	0.05 0.07 0.07 0.07 0.07 0.04	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0212 C234 2007
			0.070-0.071	436	pod	0* 0 1 3 7 10	0.09 0.09 0.08 0.11 0.11 0.08	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	
			0.094-0.096	429	pod	0* 0 1 3 7 10	0.11 0.17 0.12 0.12 0.13 0.13	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	
Australia Forth, Tasmania	Green Pea, Field Resal	3	0.047	248 – 254	pod	0* 0 1 3 7 10	0.18 0.33 0.37 0.27 0.23 0.21	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	BCS-0185 C199 2007

Country Year	Crop	Application			Portion	PHI	Residues (mg/kg)[a]		Report No. Trial No. year
		No	kg ai/ha	L/ha water			flubendiamide	F-des iodo	
					stubble	0*	1.2	< 0.02	
						0	2.8	< 0.02	
						1	3.2	< 0.02	
						3	2.9	< 0.02	
						7	3.3	< 0.02	
						10	2.9	< 0.02	
			0.071	248 – 254	pod	0*	0.30	< 0.02	
						0	0.73	< 0.02	
						1	0.45	< 0.02	
						3	0.42	< 0.02	
						7	0.40	< 0.02	
						10	0.26	< 0.02	
					stubble	0*	3.2	< 0.02	
						0	3.4	< 0.02	
						1	3.7	< 0.02	
						3	5.9	< 0.02	
						7	3.6	< 0.02	
						10	6.5	0.02	
			0.095	248 – 254	pod	0*	0.23	< 0.02	
						0	0.73	< 0.02	
						1	0.55	< 0.02	
						3	0.47	< 0.02	
						7	0.51	< 0.02	
						10	0.43	< 0.02	
					stubble	0*	3.5	< 0.02	
						0	6.2	0.02	
						1	7.4	0.02	
						3	6.7	< 0.02	
						7	7.1	0.02	
						10	6.1	< 0.02	
Australia Longford, Tasmania	Green Pea, Field Resal	3	0.047	390-398	pod	0*	< 0.02	< 0.02	BCS-0185 C200 2007
						0	0.31	< 0.02	
						1	0.26	< 0.02	
						3	0.18	< 0.02	
						7	0.10	< 0.02	
						9	0.07	< 0.02	
					stubble	0*	0.73	< 0.02	
						0	1.8	< 0.02	
						1	1.8	< 0.02	
						3	1.5	< 0.02	
						7	2.2	< 0.02	
						9	1.5	< 0.02	
			0.071	390-398	pod	0*	0.14	< 0.02	
						0	0.42	< 0.02	
						1	0.39	< 0.02	
						3	0.31	< 0.02	
						7	0.15	< 0.02	
						9	0.10	< 0.02	
					stubble	0*	1.2	< 0.02	
						0	3.3	< 0.02	
						1	2.6	< 0.02	
						3	2.9	< 0.02	
						7	2.6	< 0.02	
						9	2.3	< 0.02	
			0.095	390-398	pod	0*	0.06	< 0.02	
						0	0.44	< 0.02	
						1	0.40	< 0.02	
						3	0.22	< 0.02	
						7	0.17	< 0.02	
						9	0.08	< 0.02	

Country Year	Crop	Application			Portion	PHI	Residues (mg/kg)[a]		Report No. Trial No. year
		No	kg ai/ha	L/ha water			flubendiamide	F-des iodo	
					stubble	0*	1.9	< 0.02	
						0	3.0	< 0.02	
						1	3.6	< 0.02	
						3	2.6	< 0.02	
						7	2.4	< 0.02	
						9	2.3	< 0.02	
Australia Victoria	Green Pea, Field Princess	3	0.036	253-343	pod	0*	0.14	< 0.02	BCS-0185 C201 2007
						0	0.44	< 0.02	
						1	0.37	< 0.02	
						4	0.25	< 0.02	
						6	0.18	< 0.02	
						9	0.13	< 0.02	
					stubble	0*	2.2	< 0.02	
						0	4.6	< 0.02	
						1	4.6	< 0.02	
						4	3.1	< 0.02	
						6	3.7	0.03	
						9	2.0	< 0.02	
			0.054-0.071	253-343	pod	0*	0.14	< 0.02	
						0	0.41	< 0.02	
						1	0.38	< 0.02	
						4	0.24	< 0.02	
						6	0.22	< 0.02	
						9	0.19	< 0.02	
					stubble	0*	2.7	< 0.02	
						0	4.9	< 0.02	
						1	4.3	< 0.02	
						4	3.9	0.02	
						6	3.1	0.03	
						9	4.1	< 0.02	
			0.071-0.095	253-343	pod	0*	0.15	< 0.02	
						0	0.62	< 0.02	
						1	0.43	< 0.02	
						4	0.32	< 0.02	
						6	0.31	< 0.02	
						9	0.33	< 0.02	
					stubble	0*	3.1	< 0.02	
						0	6.9	0.02	
						1	6.0	0.02	
						4	7.6	0.03	
						6	5.9	0.04	
						9	5.0	0.03	
Australia Western	Green Pea, Field Massey	3	0.048	311-317	pod	0*	0.12	< 0.02	BCS-0185 C202 2007
						0	0.41	< 0.02	
						1	0.29	< 0.02	
						2	0.22	< 0.02	
						7	0.23	< 0.02	
						9	0.40	< 0.02	
					stubble	0*	0.77	< 0.02	
						0	3.6	< 0.02	
						1	2.8	< 0.02	
						2	2.1	< 0.02	
						7	1.9	< 0.02	
						9	3.8	0.03	
			0.073	311-317	pod	0*	0.28	< 0.02	
						0	0.70	< 0.02	
						1	0.41	< 0.02	
						2	0.51	< 0.02	
						7	0.57	< 0.02	
						9	0.90	< 0.02	

Country Year	Crop	Application			Portion	PHI	Residues (mg/kg)[a]		Report No. Trial No. year
		No	kg ai/ha	L/ha water			flubendiamide	F-des iodo	
					stubble	0*	2.0	< 0.02	
						0	5.2	0.02	
						1	5.8	0.02	
						2	5.2	< 0.02	
						7	4.5	0.02	
						9	7.4	0.04	
			0.097	311-317	pod	0*	0.27	< 0.02	
						0	0.70	< 0.02	
						1	0.60	< 0.02	
						2	0.63	< 0.02	
						7	0.44	< 0.02	
						9	1.1	< 0.02	
					stubble	0*	4.2	< 0.02	
						0	7.7	0.02	
						1	6.4	0.03	
						2	6.2	0.02	
						7	6.1	0.03	
						9	9.2	0.05	
USA Pensilvania	Bean, Kidney Savannah	2	0.110-0.111	184	pod	1	0.09	< 0.01	RAAMP014 AM022-07HA 2007
						3	0.06	< 0.01	
USA Georgia	Bean, Kidney Contender	2	0.104-0.105	91-111	pod	1	0.16	< 0.01	RAAMP014 AM023-07HA 2007
						3	0.17	< 0.01	
USA Florida	Bean, Kidney Provider	2	0.104	176	pod	1	0.09	< 0.01	RAAMP014 AM024-07HB 2007
						3	0.08	< 0.01	
USA Kansas	Bean, Kidney Bush Contender	2	0.104-0.105	129	pod	0	0.12	< 0.01	RAAMP014 AM025-07DA 2007
						1	0.14	< 0.01	
						3	0.12	< 0.01	
						7	0.06	< 0.01	
						10	0.04	< 0.01	
USA N Dakota	Bean, kidney Blue Lake 274	2	0.106-0.108	138	pod	1	0.03	< 0.01	RAAMP014 AM026-07HA 2007
						4	0.02	< 0.01	
USA Idaho	Bean, kidney Blue Lake Bush	2	0.109-0.110	98	pod	1	0.07	< 0.01	RAAMP014 AM027-07HA 2007
						3	0.04	< 0.01	
USA IA	Pea Sugar Sprint	2	0.105-0.106	138-143	pod	1	0.14	< 0.01	RAAMP014 AM028-07HA 2007
						3	0.14	< 0.01	
USA NE	Pea Oregon	2	0.104	130	pod	1	0.22	< 0.01	RAAMP014 AM029-07HA 2007
						3	0.13	< 0.01	
USA Idaho	Pea, Oregon sugar pod II	2	0.106-0.108	186-189	pod	1	0.21	< 0.01	RAAMP014 AM030-07HA 2007
						3	0.15	< 0.01	
USA Georgia	Bean, Lima Fordhook 242	2	0.105-0.106	136-163	bean, w/o pods	0	< 0.01	< 0.01	RAAMP015 AM031-07DA 2008
						1	< 0.01	< 0.01	
						3	< 0.01	< 0.01	
						7	< 0.01	< 0.01	
						10	< 0.01	< 0.01	
USA Virginia	Bean, Lima Thoro-green	2	0.107-0.108	125	bean, w/o pods	1	0.02	< 0.01	RAAMP015 AM032-07HA 2007
						3	0.03	< 0.01	
USA Georgia	Bean, Lima Jackson wonder	2	0.104-0.105	132-139	bean, w/o pods	1	< 0.01	< 0.01	RAAMP015 AM033-07HA 2007
						3	< 0.01	< 0.01	
USA N. Dakota	Bean, Lima Henderson Bush	2	0.107-0.110	145-159	bean, w/o pods	1	0.01	< 0.01	RAAMP015 AM034-07HA 2007
						3	< 0.01	< 0.01	
USA California	Bean, Lima Lee	2	0.104-0.105	140-141	bean, w/o pods	1	< 0.01	< 0.01	RAAMP015 AM035-07HA 2007
						3	< 0.01	< 0.01	
USA Idaho	Bean, Lima Henderson Bush	2	0.103-0.104	96-98	bean, w/o pods	1	< 0.01	< 0.01	RAAMP015 AM036-07HA 2007
						3	< 0.01	< 0.01	
USA Pensilvania	Pea, garden Wando	2	0.108-0.109	180-182	peas w/o pods	1	< 0.01	< 0.01	RAAMP015 AM037-07HA 2007
						3	< 0.01	< 0.01	

		Application					Residues (mg/kg)[a]		
Country Year	Crop	No	kg ai/ha	L/ha water	Portion	PHI	flubendiamide	F-des iodo	Report No. Trial No. year
USA Iowa	Pea, garden Laxton´s Progress #9	2	0.104-0.108	128-148	peas w/o pods	0 1 3 7 10	0.02 0.02 0.03 < 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	RAAMP015 AM038-07DA 2007
USA Kansas	Pea, garden Little Marvel	2	0.105-0.106	134-135	peas w/o pods	1 3	< 0.01 < 0.01	< 0.01 < 0.01	RAAMP015 AM039-07HA 2007
USA N. Dakota	Pea, garden Little Marvel	2	0.104	140	peas w/o pods	1 3	< 0.01 < 0.01	< 0.01 < 0.01	RAAMP015 AM040-07HA 2007
USA Idaho	Pea, garden Pendelton	2	0.106-0.109	170-174	peas w/o pods	1 3	0.01 < 0.01	< 0.01 < 0.01	RAAMP015 AM041-07HA 2007
USA Oregon	Pea, garden Sugar Snap	2	0.105-0.106	138-150	peas w/o pods	1 3	< 0.01 < 0.01	< 0.01 < 0.01	RAAMP015 AM042-07HA 2007

[a]. residues from USA are the highest of two samples; * prior to last treatment; ** residue in control;

Soya bean

Flubendiamide is registered in USA in soya beans at a GAP of 2 × 0.10 kg ai/ha, with 1 day PHI for green seed and 3 days PHI for dry seed. Twenty trials were conducted in soya bean seed in USA (Table 70).

Table 70 Results of residue trials conducted with flubendiamide SC480 in/on soya bean in USA in 2007 (RAAMP004)

		Application					Residues (mg/kg)[a]		
State	Variety	No	kg ai/ha	L/ha water	Portion	PHI	flubendiamide	F-des iodo	Trial No.
Georgia	S76-L9	2	0.10	168	seed, green	1 3	0.04 0.04	< 0.01 < 0.01	AM001-07HA
					seed	14 21	0.01 0.06	< 0.01 < 0.01	
North Carolina	Pioneer 95M50	2	0.10	153	seed, green	1 3	0.04 0.03	< 0.01 < 0.01	AM002-07HA
					seed	14 21	0.01 0.01	< 0.01 < 0.01	
Arkansas	AG4403RR	2	0.11	148	seed, green	1 3	0.18 0.21	< 0.01 < 0.01	AM003-07HA
					seed	14 21	0.25 0.15	< 0.01 < 0.01	
Louisiana	Dyna-gro 33B52	2	0.11	124	seed, green	1 3	0.03 0.01	< 0.01 < 0.01	AM004-07HA
					seed	14 21	0.01 0.01	< 0.01 < 0.01	
Arkansas	SG 4680 RR	2	0.10-0.11	188	seed, green	1 3	0.40 0.20	< 0.01 < 0.01	AM005-07HA
					seed	15 21	0.02 0.02	< 0.01 < 0.01	
Illinois	5N382RR	2	0.10-0.11	158-180	seed, green	0 1 3 5 7	0.07 0.04 0.02 0.02 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	AM006-07DA
					seed	10 14 21 28 35	0.05 0.05 0.06 0.14 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	

State	Variety	Application			Portion	PHI	Residues (mg/kg)^a		Trial No.
		No	kg ai/ha	L/ha water			flubendiamide	F-des iodo	
Iowa	Pioneer 93B82	2	0.10-0.11	121	seed, green	0	0.19	< 0.01	AM007-07DA
						1	0.15	< 0.01	
						3	0.22	< 0.01	
						5	0.07	< 0.01	
						7	0.07	< 0.01	
					seed	10	0.03	< 0.01	
						14	0.07	< 0.01	
						21	0.02	< 0.01	
						28	0.01	< 0.01	
						35	< 0.01	< 0.01	
Missouri	Hutcheson	2	0.104	133	seed, green	1	0.29	< 0.01	AM008-07HA
						3	0.16	< 0.01	
					seed	14	0.02	< 0.01	
						21	0.06	< 0.01	
Ohio	Shur Grow 354 RR	2	0.11	164	seed, green	1	0.01	< 0.01	AM009-07HA
						3	0.04	< 0.01	
					seed	14	0.03	< 0.01	
						22	< 0.01	< 0.01	
Iowa	93M11	2	0.11	177-179	seed, green	1	0.02	< 0.01	AM010-07HA
						3	0.02	< 0.01	
					seed	15	0.01	< 0.01	
						22	0.07	< 0.01	
North Dakota	Croplan	2	0.105	940	seed, green	1	0.05	< 0.01	AM011-07HA
						3	0.04	< 0.01	
					seed	14	0.02	< 0.01	
						24	0.01	< 0.01	
North Dakota	4238491	2	0.10-0.11	142-149	seed, green	1	0.09	< 0.01	AM012-07HA
						3	0.09	< 0.01	
					seed	15	< 0.01	< 0.01	
						21	0.01	< 0.01	
Iowa	Asgrow 3802	2	0.10-0.11	156-163	seed, green	1	0.20	< 0.01	AM013-07HA
						3	0.18	< 0.01	
					seed	14	0.09	< 0.01	
						21	0.11	< 0.01	
Nebraska	NKS32G5	2	0.11	143-146	seed, green	1	0.07	< 0.01	AM014-07HA
						3	0.04	< 0.01	
					seed	13	0.03	< 0.01	
						19	0.03	< 0.01	
Kansas	NK S39-K6	2	0.11	140	seed, green	1	0.07	< 0.01	AM015-07HA
						3	0.02	< 0.01	
					seed	14	0.03	< 0.01	
						20	0.04	< 0.01	
Minnesota	Pioneer 91M70	2	0.10-0.11	161-166	seed, green	1	0.07	< 0.01	AM016-07HA
						3	0.08	< 0.01	
					seed	14	0.03	< 0.01	
						21	0.03	< 0.01	
Indiana	Pioneer 92m91	2	0.10-0.11	119-133	seed, green	1	0.10	< 0.01	AM017-07HA
						3	0.12	< 0.01	
					seed	14	0.27	< 0.01	
						21	0.30	< 0.01	
Nebraska	NC+ 3A61RR	2	0.10	185	seed, green	1	0.20	< 0.01	AM018-07HA
						3	0.16	< 0.01	
					seed	14	0.03	< 0.01	
						21	0.01	< 0.01	
Ohio	Crows 3817R	2	0.11	144-150	seed, green	1	0.09	< 0.01	AM019-07HA
						3	0.10	< 0.01	
					seed	15	0.03	< 0.01	
						19	0.02	< 0.01	
Minnesota	DynaGro 33T06	2	0.10	140	seed, green	1	0.03	< 0.01	AM020-07HA
						3	0.03	< 0.01	
					seed	14	0.01	< 0.01	
						20	0.01	< 0.01	

[a]. highest residue of two samples

Dry peas

Flubendiamide is registered in USA for dry beans and peas at 2 × 0.1 kg ai/ha and 14 days PHI. Fourteen residue field trials were conducted with flubendiamide in dry bean in the country (Table 71).

Table 71 Results of residue trials conducted with flubendiamide SC480 in/on dry pea and cowpea in USA 2007 (RAAMP016)

		Application				Residues (mg/kg)[a]		
Country Year	Crop variety	No	kg ai/ha	L/ha water	PHI	flubendiamide	F-des iodo	Trial No.
Kansas	Cowpea Pinkeye Purplehull	2	0.107	140	7 14 21 28 35	0.02 < 0.01/0.02* < 0.01 0.01 < 0.01	< 0.01 < 0.01/0.01** < 0.01 < 0.01 < 0.01	AM043-07DA
Michigan	Cowpea Vista Navy	2	0.10-0.11	140-143	14 21	< 0.01/0.02** 0.01	< 0.01/0.01** < 0.01	AM044-07HA
Iowa	Cowpea, Clifornia Blackeye	2	0.10	161-167	15 22	0.04/0.02** 0.03	< 0.01/0.01** < 0.01	AM045-07HA
Ilinois	Cowpea California Blackeye	2	0.11	164	14 21	0.20/0.02** 0.02	< 0.01/0.01** < 0.01	AM046-07HA
N Dakota	Cowpea Naviagator	2	0.11	180	14 21	0.04 0.02	< 0.01 < 0.01	AM047-07HA
Kansas	Cowpea, Pinto Field Bean	2	0.10	165-170	13 20	0.06/0.01** 0.03	< 0.01 < 0.01	AM048-07HA
Idaho	Cowpea Othello Pinto	2	0.10-0.11	173	14 21	0.01/0.01** 0.01	< 0.01/0.01** < 0.01	AM049-07HA
California	Cowpea Canario 707	2	0.105	142	14 21	0.01/0.02** 0.02	< 0.01/0.01** < 0.01	AM050-07HA
Idaho	Cowpea Small Reds 63	2	0.10-0.11	155	14 21	0.04/0.02** 0.02	< 0.01/0.01** < 0.01	AM051-07HA
Washington	Pea Tonic	2	0.105-0.106	178	7 14 21 28 35	0.04 0.04/0.02** 0.05 0.03 0.08	< 0.01 < 0.01 < 0.01/0.01** < 0.01 < 0.01	AM052-07DA
Oregon	Pea, Green Arrow	2	0.106	0.0635-0.0639	14 21	0.10/0.02** 0.18	0.03/0.01** 0.03	AM053-07HA
Oregon	Pea Ariel	2	0.10-0.11	170	14 21	0.07/0.02** 0.11	0.03/0.01** 0.03	AM054-07HA
Idaho	Pea Pendelton	2	0.105-0.107	170	14 21	0.59/0.02** 0.04	< 0.01/0.01** < 0.01	AM055-07HA
Idaho	Pea, Austrian Winter Peas	2	0.105	184	14 21	0.12/0.01** 0.18	0.03/0.02** 0.03	AM056-07HA

* [a] highest residue of two samples; ** residue in control.

Celery

Flubendiamide is registered in USA in leafy vegetables at a maximum rate of 3 × 0.05 kg ai/ha and 1 day PHI Five trials were conducted in USA in celery (Table 72).

Table 72 Results of residue trials conducted with 5 applications of flubendiamide WG in/on celery in USA in 2004 (RCAMY008)

		Application				Residues (mg/kg)[a]		
Country Year	Variety	kg ai/ha	L/ha water		PHI	flubendiamide	F-des iodo	Trial No.

Country Year	Variety	Application			PHI	Residues (mg/kg)[a]		
		kg ai/ha	L/ha water			flubendiamide	F-des iodo	Trial No.
Nebraska	unknown	0.050-0.051	131-135	Stalk*	0 1 3 7 10	2.5 1.2 0.65 0.91 0.95	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	AM161-04D
Florida	M9	0.049-0.051	94-104	stalk*	1	0.81	< 0.01	AM160-04H
California	Sonora	0.050-0.052	187-191	stalk*	1	2.3	< 0.01	AM162-04H
California	Challenger	0.050-0.051	177-184	stalk*	1	2.1	< 0.01	AM163-04H
California	Conquistador	0.049-0.051	148-155	stalk*	1	1.3	< 0.01	AM164-04H
California	Big C	0.050-0.051	136-138	stalk*	1	2.6	< 0.01	AM165-04H

* untrimmed leaf stalks; [a] highest residue of two samples

Corn

Flubendiamide is registered in USA in field corn at a maximum rate of 4 × 0.10 kg ai/ha and 28 day PHI. Twenty trials were conducted in Canada and USA (Table 73).

Table 73 Results of residue trials conducted with 4 applications of flubendiamide SC480 in/on field corn kernel in 2004 (RCAMY004)

Country, Region	Crop	Application		PHI	Residues (mg/kg)[a]		Trial No.
		kg ai/ha	L/ha water		Flubendiamide	F-des iodo	
Canadá, Ontario	P39M79	0.10	87-98	32	< 0.01	< 0.01	AM108-04H-A
Canadá, Ontario	38P04	0.10	89-102	34	< 0.01	< 0.01	AM109-04H
Canada, Ontario	Pioneer 39T70	0.10	84-98	34	< 0.01	< 0.01	AM110-04H
USA, New York	Garst 8948RR	0.10	155-158	28	< 0.01	< 0.01	AM100-04H-A
USA, Georgia	Pioneer RR 31N26	0.10	141-148	28	< 0.01	< 0.01	AM102-04H-A
USA Kansas	Garst 8451RR	0.10	125-146	7 14 21 27 35	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	AM104-04D-A
USA Nebraska	NK 67T4	0.10	131-133	7 14 20 27 34	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	AM106-04D-A
USA, Illinois	Garst 8550 Bt	0.10	130-134	28	< 0.01	< 0.01	AM105-04H-A
USA, Minnesota	NK N27-M3	0.10	106-117	35	0.01	< 0.01	AM107-04H-A
USA, Iowa	33P34	0.11	148-202	28	< 0.01	< 0.01	AM111-04H
USA, N. Dakota	2789RRY (Sabre)	0.10	163-171	27	< 0.01	< 0.01	AM112-04H
USA, N. Dakota	0083098 (Dekalb)	0.10	157-171	27	< 0.01	< 0.01	AM113-04H
USA, Ohio	AgriGold 6395	0.10	138-140	27	< 0.01	< 0.01	AM114-04H
USA, Illinois	Buurus 569	0.10	91-102	28	< 0.01	< 0.01	AM115-04H
USA, Illinois	NK N50-P5	0.10	128-134	48	< 0.01	< 0.01	AM116-04H
USA, N. Dakota	LR9781RR (Legend)	0.10	165-170	27	< 0.01	< 0.01	AM117-04H
USA, Iowa	GH H9247BT	0.10	127-158	27	< 0.01	< 0.01	AM118-04H
USA, Minnesota	Cropland 212RR/BT	0.10	185-211	26	< 0.01	< 0.01	AM119-04H
USA, Missouri	LG 2540	0.10	158-174	28	0.01	< 0.01	AM120-04H
USA, Texas	EXP 804 RR/YGRW	0.10	94-110	27	< 0.01	< 0.01	AM121-04H

[a] mean of two samples

Rice

Flubendiamide is registered in India in rice at 3 × 0.024 kg ai/ha (375 to 500 L/ha) and 40 days PHI. Twelve trials were conducted in rice in Thailand and India (Table 74).

Table 74 Results of residue trials conducted with 3 applications of flubendiamide WDG in/on rice in Asia in 2008 (20074104/AS1-FPRI)

Country Year	Crop	Application		Portion	PHI	Residues (mg/kg)[a]		Trial No.
		kg ai/ha	L/ha water			Flubendiamide	F-des iodo	
Thailand, Parthunthani	Suphan Buri 1	0.025	250-265	Grain[a]	30	< 0.01	< 0.01	TH07W008R
		0.05-0.06	246-292	Brown	30	< 0.01	< 0.01	
				Polished	30	< 0.01	< 0.01	
Thailand	Suphan Buri 1	0.02	230-256	Grain[a]	27	0.04	< 0.01	TH07W009R
Thailand	RD29	0.026	246-270	Grain[a]	28	0.11	< 0.01	TH07W010R
Thailand	Phitsanulok 2	0.021-0.027	240-268	Grain[a]	28	< 0.01	< 0.01	TH07W011R
Thailand	Pathum Thani .1	0.024-0.025	235-278	Grain[a]	28	0.04	< 0.01	TH07W012R
Thailand	Chai Nat 1	0.024-0.027	238-254	Grain[a]	13	0.30	< 0.01	TH07W013R
Thailand	Pathum Thani 1	0.024-0.026	240-256	Grain[a]	28	0.03	< 0.01	TH07W014R
Thailand	Pathum Thani 80	0.024-0.026	246-264	Grain[a]	28	0.05	< 0.01	TH07W015R
Thailand	Pathum Thani	0.020-0.024	240-290	Grain[a]	28	0.02	< 0.01	TH07W016R
Thailand	Suphan buri	0.023-0.029	226-290	Grain[a]	28	< 0.01	< 0.01	TH07W017R
India, Mandya	Jaya	0.025		Grain	28	0.20	< 0.01	G5077, F1
India, Shimoga	Jyothi	0.025		Grain	28	0.06	< 0.01	G5077, F2

[a] highest residue of two samples

Tree nuts

Flubendiamide is registered in USA in tree nuts at 3 × 0.14 kg ai/ha and 14 days PHI Twenty trials were conducted in almonds and pecan nut in USA (Table 75).

Table 75 Results of residue trials conducted with flubendiamide SC in/on tree nut in USA in 2004. Residues in nut without shell (RCAMY014)

Country Year	Crop	Application			PHI	Residues (mg/kg)[a]		Trial No.
		No	kg ai/ha	L/ha water		flubendiamide	F-des iodo	
California	Almond, Non-Pareil	3	0.14	598-617	14	< 0.01	< 0.01	AM214-04H-C
California	Almond Wood Colony, Butte	3	0.14	411-415	0 7 14 21 28	0.07 0.05 0.03 0.04 0.04	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	AM213-04D-C
California	Almond, Mission	3	0.14	570-614	14	< 0.01	< 0.01	AM215-04H-C
California	Almond, Carmel	3	0.14	524-568	14	0.01	< 0.01	AM216-04H-C
California	Almond, Price	3	0.14	488-494	14	0.02	< 0.01	AM217-04H-C
California	Almond, Wood Colony	3	0.140	2340	14	0.05	< 0.01	AM213-04D-D
California	Almond, Non-Pareil	3	0.14	2143-2149	14	< 0.01	< 0.01	AM214-04H-D
California	Almond, Mission	3	0.14	2530-2641	14	< 0.01	< 0.01	AM215-04H-D
California	Almond, Carmel	3	0.14	2217-2414	14	0.02	< 0.01	AM216-04H-D
California	Almond, Price	3	0.14	2774-2834	14	0.02	< 0.01	AM217-04H-D
Georgia	Pecan nut, Stewart	3	0.14	545-564	13	< 0.01	< 0.01	AM218-04H-C
Georgia	Pecan nut, Sumner	3	0.14	457-466	13	< 0.01	< 0.01	AM219-04H-C
Texas	Pecan nut, Stuart	3	0.14	422-452	14	< 0.01	< 0.01	AM221-04H-C
Oklahoma	Pecan nut, Natives	3	0.14	532-572	12	0.03	< 0.01	AM222-04H-C
Louisiana	Pecan nut, Oconee	3	0.14	377-600	0 7 14 21 28	0.02 0.01 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	AM220-04D-C
Georgia	Pecan nut, Stewart	3	0.14	0.007	13	< 0.01	< 0.01	AM218-04H-D
Georgia	Pecan nut, Sumner	3	0.14	0.006-0.007	13	< 0.01	< 0.01	AM219-04H- D

Country Year	Crop	Application			PHI	Residues (mg/kg)[a]		
		No	kg ai/ha	L/ha water		flubendiamide	F-des iodo	Trial No.
Louisiana	Pecan nut, Oconee	3	0.14	0.005-0.007	14	0.01	< 0.01	AM220-04D- D
Texas	Pecan nut, Stuart	3	0.14	0.007	14	< 0.01	< 0.01	AM221-04H- D
Oklahoma	Pecan nut, Natives	3	0.14	0.007	12	0.02	< 0.01	AM222-04H- D

[a] highest residues of two samples

Cotton

Flubendiamide is registered in USA in cotton at 3 × 0.10 kg ai/ha and 28 days PHI. Twelve trials were conducted with flubendiamide in/on cotton seed in the country (Table 76).

Table 76 Results of residue trials conducted with 3 applications of flubendiamida SC480 in/on cotton seed in USA 2004/2005 (RCAMY002)

State	Variety	Application		PHI	Residues (mg/kg)[a]		
		kg ai/ha	L/ha water		flubendiamide	F-des iodo	Trial No.
Georgia	RR Delta Pine	0.104-0.105	97-101	26	0.19	< 0.01	AM087-04HA
Mississippi	FM 960 BR	0.105-0.107	153-173	0 10 22 26 35	0.45 0.41 0.21 0.18 0.13	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	AM088-04D
Arkansas	PM 1218 BG RR	0.105	94	28	0.11	< 0.01	AM089-04HA
Arkansas	ST4793R	0.10-0.11	139	28	0.03	< 0.01	AM090-04H
Texas	FiberMax 989 LL	0.10-0.11	129-140	26	0.37	< 0.01	AM091-04H
Texas	Fibermax 958	0.10-0.11	174-177	27	1.0	< 0.01	AM092-04HA
Oklahoma	PM 2280	0.10	112-117	28	0.12	< 0.01	AM093-04HA
Texas	FM 989 BR	0.10-0.11	134-144	28	< 0.01	< 0.01	AM094-04H
Oklahoma	Paymaster 2280	0.11	133-135	27	0.02	< 0.01	AM095-04H
California	Sierra RR	0.10	166-173	28	0.25	< 0.01	AM096-04H
California	Acala Riata	0.10	170-173	28	0.12	< 0.01	AM097-04H
California	Acala Maxxa	0.10-0.11	135-137	28	0.28	< 0.01	AM098-04H

[a]. highest residues of two samples

Tea

Flubendiamide is registered in Japan in tea at 1 × 0.40 kg ai/ha and 7 days PHI. Six trials were conducted with flubendiamide in/on tea in Japan (Table 77).

Table 77 Results of residue trials conducted with flubendiamide in/on green tea in Japan in 2008 (08R001)

Region	Variety	Application			PHI	Residues (mg/kg) flubendiamide
		No	kg ai/ha	kg ai/hL		
Saitama	Fukumidori	1	0.4	10	7 14 21	24 12 3.8
Kagoshima	Okumidori	1	0.4	10	7 14 21	22 8.6 1.3
Kyoto	Yabukita	1	0.4	10	7	17
J Mie	Yabukita	1	0.4	10	7	28
Kouchi	Yabukita	1	0.4	10	7	29
J Miyazaki	Fuushun	1	0.4	10	7	11

Animal feed

In the supervised trials conducted in dry pea, corn, rice, cotton, soya bean and almonds crops parts of the plants used as animal feed were also sampled. Residues of flumendiamide in these samples are shown on Tables 78 to 83.

Table 78 Results of residue trials conducted with flubendiamide SC480 in/on soya bean in USA in 2007 (RAAMP004

		Application					Residues (mg/kg)[a]		
State	Variety	No	kg ai/ha	kg ai/hL	Portion	PHI	flubendiamide	Flubendiamide des iodo	Trial No.
Georgia	S76-L9	2	0.105-0.106	150	forage	3	10	0.04	AM001-07HA
						7	8.9	0.05	
					hay	3	26	0.09	
						7	22	0.09	
North Carolina	Pioneer 95M50	2	0.104-0.108	157-164	forage	3	11	0.07	AM002-07HA
						7	11	0.06	
					hay	3	25	0.17	
						7	24	0.14	
Arkansas	AG4403RR	2	0.105-0.106	133	forage	3	9.1	0.04	AM003-07HA
						7	7.2	0.03	
					hay	3	25	0.08	
						7	30	0.12	
Louisiana	Dyna-gro 33B52	2	0.106	148	forage	3	4.3	0.04	AM004-07HA
						7	2.9	0.04	
					hay	3	14	0.14	
						7	8.1	0.11	
Arkansas	SG 4680 RR	2	0.105	187	forage	3	8.0	0.05	AM005-07HA
						7	6.2	0.06	
					hay	3	35	0.27	
						7	30	0.27	
Illinois	5N382RR	2	0.104-0.105	170-178	forage	0	15	0.05	AM006-07DA
						3	11	0.06	
						7	8.7	0.06	
						10	5.4	0.02	
						14	4.0	0.03	
					hay	0	32	0.12	
						3	22	0.12	
						7	15	0.10	
						10	15	0.08	
						14	14	0.10	
Iowa	Pioneer 93B82	2	0.105-0.106	158-180	forage	0	14	0.04	RAAMP004 AM007-07DA 2007
						3	10	0.06	
						7	7.4	0.05	
						10	3.4	0.03	
						14	8.9	0.03	
					hay	0	80	0.23	
						3	39	0.21	
						7	25	0.26	
						10	13	0.11	
						14	6.7	0.07	
Missouri	Hutcheson	2	0.107	133	forage	3	2.8	< 0.01	AM008-07HA
						7	6.7	0.03	
					hay	3	17	0.05	
						7	9.8	0.04	
Ohio	Shur Grow 354 RR	2	0.105-0.106	163	forage	3	4.6	0.05	AM009-07HA
						7	6.0	0.04	
					hay	3	8.6	0.10	
						7	12	0.13	
Iowa	93M11	2	0.102-0.107	168-178	forage	3	6.1	0.03	AM010-07HA
						7	4.4	0.04	
					hay	3	25	0.16	
						7	16	0.14	

State	Variety	Application No	kg ai/ha	kg ai/hL	Portion	PHI	Residues (mg/kg)[a] flubendiamide	Flubendiamide des iodo	Trial No.
North Dakota	Croplan	2	0.106	141	forage	5	7.1	0.03	AM011-07HA
						7	3.7	0.01	
					hay	5	13	0.06	
						7	8.8	0.05	
North Dakota	4238491	2	0.106-0.108	144	forage	3	7.2	0.04	RAAMP004 AM012-07HA
						7	4.5	0.03	
					hay	3	32	0.21	
						7	20	0.14	
Iowa	Asgrow 3802	2	0.104	162-178	forage	3	13	0.06	RAAMP004 AM013-07HA
						7	5.9	0.05	
					hay	3	33	0.18	
						7	26	0.26	
Nebraska	NKS32G5	2	0.105	126	forage	3	9.9	0.06	RAAMP004 AM014-07HA
						7	7.2	0.02	
					hay	3	41	0.23	
						7	19	0.07	
Kansas	NK S39-K6	2	0.104-0.107	143-146	forage	3	7.6	0.03	RAAMP004 AM015-07HA
						7	6.5	0.02	
					hay	3	29	0.18	
						9	24	0.09	
Minnesota	Pioneer 91M70	2	0.105-0.107	143-146	forage	3	6.8	0.05	RAAMP004 AM016-07HA
						7	2.9	0.01	
					hay	3	15	0.08	
						7	10	0.04	
Indiana	Pioneer 92m91	2	0.104	146-153	forage	3	15	0.04	RAAMP004 AM017-07HA
						7	7.3	0.02	
					hay	3	34	0.10	
						7	18	0.06	
Nebraska	NC+ 3A61RR	2	0.104-0.105	113	forage	3	7.9	0.06	RAAMP004 AM018-07HA
						7	5.0	0.03	
					hay	3	41	0.41	
						7	11	0.16	
Ohio	Crows 3817R	2	0.106-0.107	151	forage	3	7.7	0.04	RAAMP004 AM019-07HA
						7	6.3	0.02	
					hay	3	29	0.14	
						7	25	0.12	
Minnesota	DynaGro 33T06	2	0.105-0.106	144-150	forage	3	10	0.06	RAAMP004 AM020-07HA
						7	4.1	0.05	
					hay	3	23	0.20	
						7	21	0.22	

[a]. highest residue of two samples

Table 79 Results of residue trials conducted with flubendiamide SC480 in/on dry pea in USA in 2007 (RAAMP016)

Region	Crop	Application No	kg ai/ha	L/ha water	Portion	PHI	Residues (mg/kg)[a] flubendiamide	F-des iodo	Trial No.
Gardner	Cowpea Pinkeye Purplehull	2	0.11	139	forage	0	11	0.04	AM043-07DA
						3	6.6	0.04	
						7	4.1	0.03	
						10	1.7	0.01	
						14	0.80	< 0.01	
			0.10-0.11	140-143	hay	0	25	0.05	
						3	25/0.02**	0.09/0.01**	
						7	14	0.11	
						9	15	0.07	
						14	14	0.15	
Saginaw	Cowpea Vista	2	0.10-0.11	161-167	forage	3	11/0.01**	0.03	RAAMP016 AM044-07HA
						7	14	0.04	

Region	Crop	Application			Portion	PHI	Residues (mg/kg)[a]		Trial No.
		No	kg ai/ha	L/ha water			flubendiamide	F-des iodo	
	Navy		0.10-0.11	169	hay	3 6	13/0.02** 26	0.03/0.01** 0.05	
Bagley	Cowpea California Blackeye	2	0.10	170-174	forage	4 7	3.4 4.2	0.02 0.02	RAAMP016 AM045-07HA
			0.10-0.11	170-179	hay	3 7	24/21** 12	0.05/0.10** 0.04	
Carlyle	Cowpea California Blackeye	2	0.11	121-130	forage	3 7	5.5/0.01** 3.9	0.02/0.01** 0.02	RAAMP016 AM046-07HA
			0.10-0.11	150-166	hay	3 7	10/0.02** 16	0.04/0.01** 0.07	
Eldridge	Cowpea California Blackeye	2	0.10-0.11	133	forage	3 7	3.9 3.4	0.01 0.01	RAAMP016 AM047-07HA
				115-137	hay	3 7	7.3/0.01** 8.3	0.02 0.03	
Rupert	Cowpea Small Reds 63	2	0.10-0.11	153-157	forage	3 7	9.0 2.6	0.04 0.02	RAAMP016 AM051-07HA
			0.11	98	hay	3 7	15/0.02** 9.0	0.06/0.01** 0.04	
Ephrata	Pea Tonic	2	0.10-0.11	179	vines	0 3 7 10 14	3.4 2.4/0.01** 1.4 0.91 0.78	0.01 0.01/0.01** 0.01 < 0.01 0.02	RAAMP016 AM052-07DA
					hay	0 3 7 10 14	6.2 4.0/0.02** 4.2 2.4 2.8	0.02 0.03/0.01** 0.02 0.02 0.02	
Parkdale	Pea Green Arrow	2	0.106	167	vines	3 7	3.4/0.01** 3.6	0.02/0.01** 0.02	RAAMP016 AM053-07HA
					hay	3 7	8.0/0.02** 12	0.04/0.01** 0.07	
Madras	Pea Ariel	2	0.10-0.11	169	vines	3 7	3.1/0.01** 2.1	0.02/0.01** 0.02	RAAMP016 AM054-07HA
					hay	3 7	7.6/0.01** 9.1	0.04/0.01** 0.06	
Jerome	Pea Pendelton	2	0.10-0.11	167-173	vines	3 7	2.4/0.01** 1.5	0.01/0.01** 0.01	RAAMP016 AM055-07HA
					hay	3 7	9.9/0.01** 9.2	0.04/0.01** 0.05	
Payette	Pea Austrian Winter	2	0.10	183-185	vines	3 7	5.5/0.01** 5.0	0.02/0.02** 0.02	RAAMP016 AM056-07HA
					hay	3 7	19/0.01** 20	0.06/0.01** 0.07	

[a]. highest residue of two samples, ** residue in control

Table 80 Results of residue trials conducted with flubendiamide SC480 in/on corn (field and sweet) in 2004/2005 (RCAMY004)

Country Year	Crop	Application			Portion	PHI	Residues (mg/kg)[a]		Trial No.
		No	kg ai/ha	L/ha water			flubendiamide	F-des iodo	
Canada Ontario	Corn P39M79	4	0.10-0.11	87-98	forage	1	3.7	0.02	AM108-04H-A
					fodder	32	5.9	0.02	
Canada Ontario	Corn 38P04	4	0.10-0.11	89-102	forage	1	3.9	0.02	AM109-04H
					fodder	34	9.6	0.03	
Canada Ontario	Corn Pioneer39T70	4	0.10-0.11	84-97	forage	1	5.0	0.02	AM110-04H
					fodder	34	4.3	0.02	
Canada Ontario	Corn, sweet P39M79	4	0.10-0.11	87-95	forage	1	3.5	0.01	AM108-04H-B

		Application					Residues (mg/kg)[a]		
Country Year	Crop	No	kg ai/ha	L/ha water	Portion	PHI	flubendiamide	F-des iodo	Trial No.
USA New York	Corn Garst 8948RR	4	0.11	155-162	forage	1	1.0	< 0.01	AM100-04H-A
					fodder	28	2.9	0.01	
USA Georgia	Corn, Pioneer RR 31N26	4	0.10	149	forage	1	5.6	0.02	AM102-04H
					fodder	28	9.7	0.04	
USA Stilwell, Kansas	Corn Garst 8451RR	4	0.10	138-144	forage	0 1 2 7 10	3.3 3.8 3.8 3.4 2.6	0.01 0.01 0.01 0.02 0.01	AM104-04D-A
					fodder	7 14 21 27 35	6.1 3.8 3.5 5.3 2.6	0.02 0.02 0.02 0.02 0.01	
USA Springfield, Nebraska	Corn NK 67T4	4	0.10-0.11	135-138	forage	0 1 3 7 10	2.4 3.1 3.4 2.4 2.5	0.01 0.02 0.02 0.02 0.01	AM106-04D-A
					fodder	7 14 20 27 34	6.0 4.8 4.8 4.2 7.4	0.02 0.02 0.02 0.02 0.03	
USA Illinois	Corn Garst 8550 Bt	4	0.10-0.11	143-150	forage	1	4.2	0.02	AM105-04H-A
					fodder	28	3.4	0.02	
USA Minnesota	Corn NK N27-M3	4	0.11	106-118	forage	1	1.7	< 0.01	AM107-04H-A
					fodder	35	4.4	0.02	
USA Iowa	Corn 33P34	4	0.11	148-202	forage	1	3.8	0.02	AM111-04H
					fodder	28	8.0	0.04	
USA Dakota	Corn, 789RRY (Sabre)	4	0.10-0.11	163-171	forage	1	3.7	< 0.01	AM112-04H
					fodder	27	3.8	0.02	
USA Dakota	Corn, 0083098 (Dekalb)	4	0.10-0.11	157-171	forage	1	3.6	0.01	AM113-04H
					fodder	27	5.0	0.02	
USA Ohio	Corn AgriGold 6395	4	0.10	138-140	forage	1	5.3	0.03	AM114-04H
					fodder	27	14	0.05	
USA Illinois	Corn Buurus 569	4	0.10-0.11	91-102	forage	1	1.8	< 0.01	AM115-04H
					fodder	28	5.9	0.04	
USA Illinois	Corn NK N50-P5	4	0.10-0.11	128-134	forage	1	4.8	0.02	AM116-04H
					fodder	48	6.3	0.02	
USA N. Dakota	CornLR9781RR (Legend)	4	0.10-0.11	165-170	forage	1	3.6	0.01	AM117-04H
					fodder	27	7.3	0.02	
USA Iowa	Corn GH H9247BT	4	0.10	158-134	forage	1	1.8	< 0.01	AM118-04H
					fodder	27	2.7	0.02	
USA Minnesota	Corn Cropland 212RR/BT	4	0.10-0.11	185-211	forage	1	4.6	0.02	AM119-04H
					fodder	26	10	0.03	
	Corn LG 2540	4	0.10-0.11	158-174	forage	0	8.4	0.02	AM120-04H
					fodder	28	12	0.08	
USA Texas	Corn EXP 804 RR/YGRW	4	0.10-0.11	84-110	forage	1	3.6	0.02	AM121-04H
					fodder	27	4.3	0.04	
USA New York	Corn, sweet Garst 8948RR	4	0.11	155-158	forage	1	2.0	< 0.01	AM100-04H-B
USA Virginia	Corn, sweet Silver Queen	4	0.10-0.11	130-134	forage	1	6.7	0.02	AM101-04H
					fodder	28	9.3	0.04	
USA Georgia	Sweet Pioneer RR31N26	4	0.10-0.11	141-148	forage	1	3.8	0.01	AM102-04H-B

		Application					Residues (mg/kg)[a]		
Country Year	Crop	No	kg ai/ha	L/ha water	Portion	PHI	flubendiamide	F-des iodo	Trial No.
USA Florida	Corn, sweet Incredible	4	0.10-0.11	124-129	forage	0	5.1	0.02	AM103-04DA
						1	4.4	0.02	
						3	5.8	0.02	
						7	5.6	0.02	
						10	5.1	0.03	
					fodder	7	7.27	0.04	
						12	4.8	0.02	
						19	2.8	0.01	
						27	2.1	0.01	
						33	1.7	0.01	
USA Kansas	Corn, sweet Garst 8451RR	4	0.10-0.11	125-146	forage	2	2.4	0.01	AM104-04D-B
USA Illinois	Corn, sweet Garst 8550 Bt	4	0.11	130-134	forage	1	3.9	0.01	AM105-04H-B
USA Nebraska	Corn, sweet NK67T4	4	0.10	131-133	forage	1	2.5	0.02	AM106-04D-B
USA Minnesota	Corn, sweet GH0937	4	0.11	106-117	forage	1	2.2	< 0.01	AM107-04H-B
USA California	Corn, sweet Silver Queen	4	0.10-0.11	134-106	forage	1	4.6	0.02	AM122-04HA
					fodder	28	9.8	0.06	
USA Oregon	Corn, sweet Pronto	4	0.10-0.11	0.13-0.14	forage	1	3.9	0.01	AM123-04H
					fodder	55	17	0.05	
USA Oregon	Corn, sweet Super Sweet Jubilee	4	0.10-0.11	78-81	forage	1	5.5	0.02	AM124-04H
					fodder	69	14	0.04	

[a] highest residue of two samples

Table 81 Results of residue trials conducted with flubendiamide WDG 20 in/on rice in Asia in 2008 (20074104/AS1-FPRI)

	Application					Residues (mg/kg)[a]		
Country Year	No	kg ai/ha	L/ha water		PHI	flubendiamide	Flubendiamide des iodo	Trial No.
Thailand Pathumthani	3	0.025-0.026	250-265	Plant	0	0.62	< 0.01	TH07W008R
				Panicle	0	2.3	0.02	
		0.049-0.058	246-292	Hulls	30	0.19	< 0.01	
				Bran	30	0.03	< 0.01	
Thailand Prachinburi	3	0.023-0.026	230-256	Plant	0	0.86	< 0.01	TH07W009R
				Panicle	0	0.99	< 0.01	
Thailand Phitsanulok	3	0.025-0.027	246-270	Plant	0	0.82	< 0.01	TH07W010R
				Panicle	0	1.6	< 0.01	
Thailand Chachoengsao	3	0.021-0.027	240-268	Plant	0	0.52	< 0.01	TH07W011R 2
				Panicle	0	1.2	< 0.01	
Thailand Lopburi	3	0.024-0.025	235-278	Plant	0	1.3	< 0.01	TH07W012R
				Panicle	0	1.6	< 0.01	
Thailand Ratchaburi	3	0.024-0.027	238-254	Plant	0	1.0	0.02	TH07W013R
				Panicle	0	1.7	0.02	
Thailand Chainat	3	0.024-0.026	240-256	Plant	0	0.79	< 0.01	TH07W014R
				Panicle	0	0.95	< 0.01	
Thailand Chainat	3	0.024-0.026	246-264	Plant	0	0.82	< 0.01	TH07W015R
				Panicle	0	0.90	< 0.01	
Thailand Suphanburi	3	0.024-0.026	240-290	Plant	0	1.4	< 0.01	TH07W016R
				Panicle	0	2.6	0.02	
Thailand Suphanburi	3	0.023-0.029	226-290	Plant	0	1.4	< 0.01	TH07W017R
				Panicle	0	3.0	0.02	
India, Mandya	3	0.025		Shoot	0	0.62	< 0.01	G5077, F1

	Application					Residues (mg/kg)[a]		
Country Year	No	kg ai/ha	L/ha water		PHI	flubendiamide	Flubendiamide des iodo	Trial No.
India, Shimoga	3	0.025		Shoot	0	1.2	0.03	G5077, F2

[a]. highest residue of two samples

Table 82 Results of residue trials conducted with 3 applications of flubendiamide SC480 in/on almond hull from tree nut in USA (California) in 2004 (RCAMY014)

	Application				Residues (mg/kg)[a]		
Variety	No	kg ai/ha	L/ha water	PHI	flubendiamide	F-des iodo	Trial No.
Non-Pareil	3	0.14	598-617	14	3.3	< 0.01	AM214-04H-C
Wood Colony, Butte	3	0.14	411-415	0 7 14 21 28	5.3 5.5 5.2 3.9 4.2	0.02 0.02 0.02 0.02 0.02	AM213-04D-C
Mission	3	0.14	570-614	14	0.98	< 0.01	AM215-04H-C
Carmel	3	0.14	534-568	14	1.4	< 0.01	AM216-04H-C
Price	3	0.14	488-494	14	2.4	0.01	AM217-04H-C
Wood Colony	3	0.140	2340	14	4.7	0.03	AM213-04D-D
Non-Pareil	3	0.14	2143-2149	14	2.1	< 0.01	AM214-04H-D
Mission	3	0.14	2530-2641	14	1.4	< 0.01	AM215-04H-D
Carmel	3	0.14	2217-2414	14	2.5	0.01	AM216-04H-D
Price	3	0.14	2774-2834	14	2.9	0.01	AM217-04H-D

[a]. highest residue of two samples

Table 83 Residues in cotton gintrash from trials conducted with flubendiamide SC in USA in 2004/2005 (RCAMY002)

		Application				Residues (mg/kg)[a]		
State	Variety	No	kg ai/ha	L/ha water	PHI	flubendiamide	F-des iodo	Trial No.
Georgia	RR Delta Pine	3	0.10	97-101	26	25	0.12	AM087-4HA
Mississippi	FM 960 BR	3	0.10	153-173	0 10 26 35	26 21 6.8 3.2	0.08 0.09 0.04 0.03	AM088-04D
Texas	FiberMax 989	3	0.10	129-140	26	3.5	0.05	AM091-04H
Texas	Fibermax 958	3	0.10	174-177	27	8.1	0.03	AM092-4HA
Oklahoma	PM 2280	3	0.10	112-117	28	2.3	< 0.01	AM093-4HA
California	Sierra RR	3	0.10	160-173	28	25	0.11	AM096-04H

[a] highest residue of two samples

FATE OF RESIDUES IN PROCESSING

Effects on the nature of residues

A hydrolysis study under conditions representative for core processing procedures was conducted (Justus and Brueckner, 2005; MEF 04/411). [Phthalic-acid ring-UL-^{14}C]flubendiamide (0.2 mg ai/L water containing 1% acetonitrile) was incubated in buffered drinking water at three representative sets of conditions: pasteurization at 90 °C at pH 4 for 20 min; baking, brewing, boiling at 100 °C at pH 5 for 60 min; sterilization (autoclave) at 120 °C at pH 6 for 20 min. Two samples of each test solution were closed with a septum and crimp top and placed in a water bath (at 90 °C and 100 °C) or in an autoclave (120 °C). At zero time and at test termination, samples were analysed by HPLC and TLC. The content of radioactivity was determined by liquid scintillation counting (LSC). HPLC/MS was used for confirmation of the identy of the test compound. Radioactivity balances were in a range of 99.8 to 101.0% of applied radioactivity. In all three processing scenarios, no degradates were observed in any of the samples, and flubendiamide was the only compound in all HPLC profiles.

Processing studies

Processing studies were conducted on apple, peach, plum, grape, tomato, cucurbits, flowering and head brassica, lettuce, cotton, soya bean, corn and rice. In all studies, residues of flubendiamide and its metabolite flubendiamide-des-iodo were determined by HPLC-MS/MS.

Apple

Two trials were conducted in Germany in apple treated three times with flubendiamide at 0.120 to 0.132 kg ai/ha and PHI of 14 days (Ballesteros *et al*., 2005; RA-3301/04). In each trial, the apple sample was analysed and two separate portions were submitted to the processing procedure. The washing simulated houselhold processing whereas the apple sauce processing simulated the industrial practice at a laboratory scale. A processing study was conducted in USA with apples treated at 3 x 0.88 kg ai/ha, sample collected at 14 days PHI and processed using procedures which simulated commercial processing practices (Helfrich and Mackie, 2005; RCAMY010). Residues of flubendiamide and the processing factor (PF) in the processed commodities obtained in Germany and USA are shown on Table 84. The des-iodo metabolite was not detected in any sample analysed.

Table 84 Results of a processing trial conducted with flubendiamide in/on apple in Germany and the USA

Processed commodity	Germany, 0982/6 Fruit = 0.18 mg/kg		Germany, 0983/4 Fruit = 0.21 mg/kg		USA Fruit = 0.78 mg/kg		PF, mean or best estimate
	Residues, mg/kg	PF	Residues, mg/kg	PF	Residues, mg/kg	PF	
fruit, washed A/B	0.15/0.20	0.83/1.1	0.18/0.17	0.9/0.8	0.93	1.2	0.97
peeled fruit	-	-	-	-	< 0.01	< 0.01	< 0.01
dried fruit	-	-	-	-	0.40	0.51	0.51
juice	-	-	-	-	0.05	0.06	0.06
sauce A/B	0.08/0.09	0.4/0.5	0.08/0.07	0.4/0.3	0.021	0.02	0.36
pomace, wet A/B	1.8/1.7	10/9.4	1.9/1.2	9.0/5.7	2.8	3.6	7.5
raw stewed fruit A/B	0.11/0.12	0.6/0.7	0.11/0.11	0.5/0.5	-	-	0.6

Peach

Two peach processing trials were conducted in Spain and Portugal with flubendiamide 480 SC in 2004. The product was applied three times with an application rate of 0.12–0.15 kg/ha and peach sampled at 7 days PHI (Ballesteros *et al*., 2005; RA-2303/04). The processing of washed fruit, depitted fruit and peeled fruit simulated household practice and of peach preserve simulated the industrial practice at a laboratory scale. The des-iodo metabolite was not detected in any sample analysed. Residues of flubendiamide and the processing factor (PF) in the processed commodities are shown on Table 85.

Table 85 Results of residue trial conducted with flubendiamide SC480 in/on peach in Europe

Processed commodity	Spain, R 2004 0997 4		Portugal, R 2004 0998 2		PF, mean or best estimate
	Residues, mg/kg	PF	Residues, mg/kg	PF	
fruit	0.21	-	0.20	-	-
whole fruit, washed	0.10	0.48	0.16	0.76	0.6
peel	0.84	4	1.2	5.7	4.9
fruit, peeled	< 0.01	< 0.05	0.02	0.09	0.07
fruit, depitted	0.17	0.81	0.13	0.62	0.72
preserve	0.02	0.09	0.02	0.09	0.9

Plum

A plum processing trial was conducted inUSA with flubendiamide 480 SC applied three times at a target rate of 0.702 kg ai/ha, five times the total maximum proposed label (Helfrich & Mackie, 2006; RCAMY012). The plums were harvested at PHI of 7 days and processed using procedures which simulated commercial processing practices. The des-iodo metabolite was not detected in any sample analysed. Residues of flubendiamide in plum, washed plum and prunes were 0.09, 0.05 and 0.10 mg/kg, respectively. Processing factors for washed plum and prunes were 0.55 and 0.9.

Grapes

Seven processing trials were performed with flubendiamide SC480 on grape in Europe in 2003/2004. The processing procedures simulated the industrial practice at a laboratory scale. In 4 trials conducted on wine grape (RA-3066/03, RA-3067/03, RA-3308-04) and one on the product was applied at 4 × 0.1 kg ai/ha and samples harvested at 14 days PHI. In two table grape trials (RA-3068/03) at the same rate, samples were harveste at 7 days PHI. No residues of flubendiamide-des-iodo were found in grapes from any trial, but it was detected in grape pomace and pomace dried (0.02 and 0.03 mg/kg) and in raisin waste (0.13 and 0.15 mg/kg). The results of all trials are shown in Table 86.

Table 86 Processing trial conducted with flubendiamide SC480 in/on grape in Europe and USA

Processed commodity	RA-3066/03 Germany 0216/9 217/7		RA-3067/03, Spain 218/5 Greece 219/3		RA-3308/04 France		RA-3068/03 Portugal 223/1 A/B		RA-3068/03 Greece 779/9 A/B		RCAMY017 USA		PF, mean or best estimate
	mg/kg	PF	mg/kg	PF	mg/kg	PF	mg/kg	PF	mg/kg	PF	mg/kg	PF	
grape	0.75 0.90	-	0.06 0.18	-	0.18	-	0.40	-	0.08	-	1.9	-	-
washed	-	-	-	-	-	-	-	-	-	-	0.86	0.45	0.45
Juice	0.04 0.03	0.05 .04	< 0.01 < 0.01	0.2 0.06	< 0.01	0.06	-	-	-	-	0.15	0.07	0.13
jelly	-	-	-	-	-	-	-	-	-	-	0.08	0.04	0.04
raisin	-	-	-	-	-	-	0.83 0.51	2.1 1.3	0.23 0.14	2.9 1.8	0.57	0.3	1.7
wine at bottling	0.09 0.04	0.1 0.04	0.02 0.03	0.3 0.2	0.01	0.06	-	-	-	-	-	-	0.19
Pomace	1.8 1.9	2.4 2.1	-	-	1.0	5.6	-	-	-	-	-	-	3.4
pomace, wet	-	-	0.17 0.61	2.8 3.4	0.44	2.4	-	-	-	-	-	-	2.9
pomace, dry	-	-	0.33 1.2	5.5 6.7	0.99	5.5	-	-	-	-	-	-	5.9
raisin waste	-	-	-	-	-	-	13 15	33 38	0.66 0.62	8 8	-	-	2.2
Must	0.14 0.20	0.2 0.2	0.01 0.03	0.2 0.2	0.03	0.2	-	-	-	-	-	-	0.2

Tomato

In two trials conducted in Europe in 2003, flubendiamide was sprayed three times at 0.060 kg ai/ha to tomato plants and samples were harvested 14 days PHI. In each trial, the apple sample was analysed and two separate portions were submitted to the processing procedure. Washed and peeled tomatoes were prepared using household practices. Treated tomatoes were washed and processed into raw juice, juice, preserve, puree, paste and raw simulating industrial processing procedures. A tomato processing trial was conducted in USA in 2004 at exaggerated rate 5 × 0.263 kg ai/ha and samples harvested at 1 day PHI (Fischer, 2005). Processing was performed using procedures which simulated commercial processing practices. Flubendiamide des-iodo was not detected in any sample analysed. Residues of flubendiamide and the respective processing factors in processed commodities in the all trials are shown on Table 87.

Table 87 Results from processing study on tomato in Europe and USA

Processed commodity	Greece / R 2003 0201/0 tomato = 0.03 mg/kg		Portugal / R 2003 0826/4 tomato = 0.04 mg/kg		USA/RCAMY017 tomato = 0.20 mg/kg		PF, mean or best estimate
	Residues, mg/kg	PF	Residues, mg/kg	PF	Residues, mg/kg	PF	
Washed A/B	< 0.01/< 0.01	< 0.33/< 0.33	0.05/0.03	1.2/0.75	0.09	0.44	0.61
Juice A/B	< 0.01/< 0.01	< 0.33/< 0.33	0.02/0.02	0.5/0.5	0.07	0.78	0.49
Peeled A/B	< 0.01/< 0.01	< 0.33/< 0.33	< 0.01/< 0.01	< 0.25/< 0.25			0.29
Preserve/canned A/B	< 0.01/< 0.01	< 0.33/< 0.33	0.01/0.01	0.25/0.25	< 0.01	< 0.05	0.24
Pomace wet A/B	0.06/0.06	2/2	0.18/0.16	4.5/4			3.13
Paste A/B	0.13/0.09	4.3/3	0.27/0.13	6.8/3.2	0.5	2.5	4
Puree A/B	0.05/0.04	1.7/1.3	0.11/0.08	2.8/2	0.21	1.1	1.8
Cooked	-	-	-	-	0.12	0.61	0.61
Dried	-	-	-	-	1.0	5.1	5.1

Cucurbit

Samples of cucumber, muskmelon, and summer squash treated 5 × 0.05 kg ai/ha and collected at PHI 1 day (RCAMY015) were washed, peeled and or cooked. Residues of flubendiamide in the raw commodity were 0.02 and 0.06 mg/kg in cucumber and muskmelon, but were not detected in summer squash (< 0.01 mg/kg). No residues were detected in peeled and washed cucumber, peeled muskmelon and washed and cooked summer squash.

Cabbage and brocolli

Two head cabbage processing trials were conducted in Europe with flubendiamide 480 SC applied used three times at 0.048 kg (R 2004 0959/1) or once at 0.144 kg ai/ha (R 2004 1043/3) (Wolters and Billian, 2005; RA 3314/04). The processing of wash and cook frozen head simulated household practice. In one trials conducted in USA (3 treatments, 35 g ai/ha, PHI 1 day) in/on broccoli, cauliflower and on head cabbage, samples were processed according to normal household preparation and/or cooking procedures (Fischer, 2005; RCAMY001).The results of all trials are shown in Table 88.

Table 88 Results of processing trials with flubendiamide in cabbage and broccoli

Portion analysed	R 2004 0959/1UK		R 2004 1043/3 Germany		RCAMY001 USA		PF, mean or best estimate
	Residues, mg/kg	PF	Residues, mg/kg	PF	Residues, mg/kg	PF	
Cabbage head	0.05		0.02	-	0.22	-	
head, washed A/B	0.01/0.01	0.2/0.2	< 0.01/< 0.01	< 0.5/< 0.5	-	-	0.35
head, cooked A/B	0.01/< 0.01	0.2/< 0.2	< 0.01/< 0.01	< 0.5/0.5	< 0.01	0.04	-
Broccoli					0.10	-	
Washed					0.09	0.9	0.9
Cooked					0.07	0.7	0.7

Lettuce

Dacus and Harbin (2005; RCAMY008) evaluated the effect of removing the wrapper leaves and washing household procedures on the residues of flubendiamide on treated head lettuce (3 × 0.05 kg ai/ha). Flubendiamide-des-iodo was not detected in any samples analysed in the study. The results are shown in Table 89.

Table 89 Effect of processing on flubendiamide residues on head lettuce

Commodity	Residues, mg/kg	PF	Mean PF
Head lettuce	0.11, 0.69, 0.37, 0.66, 0.67, 0.97	-	
Head lettuce, without wrapper leaves	< 0.01, 0.02, 0.01, 0.10, 0.10, 0.01	< 0.09, 0.03, 0.03, 0.15, 0.15, 0.01	0.08
Head lettuce	1.5	-	

Commodity	Residues, mg/kg	PF	Mean PF
Head lettuce , washed	0.52	0.35	0.35

Soya bean

Soya bean was treated with flubendiamide 480 SC in USA at 2 × 0.52 kg ai/ha, and seed samples harvested at 12 days PHI were processed using procedures that simulated commercial processing practices (Dallstream and Krolski, 2008; RAAMP010). The soya bean seed samples and processed commodities were analysed flubendiamide and its metabolite (Table 90).

Table 90 Results of processing study in/on soya bean

	Flubendiamide		Des-iodo	
Portion analysed	Residues*, mg/kg	PF	Residues*, mg/kg	PF
seed	0.26	-	0.01	
aspirated grain fractions	93	358	0.46	46
meal	0.03	0.12	< 0.01	<1
hulls	0.70	2.7	< 0.01	<1
refined oil	0.01	0.04	< 0.01	<1
Defatted flour	0.02	0.08	< 0.01	<1
soymilk	< 0.01	< 0.04	< 0.01	<1

* each result is the average of triplicate samples from one bach of processed sample

Corn

A processing study was conducted with field corn treated at 4 × 0.526 kg ai/ha and samples were harvested 28 days after the last application (Lenz, 2005; RCAMY005)). The field corn grain was sub-sampled, aspirated grain fractions were generated, and the corn divided for further processing into wet and dry milled commodities. Wet milled commodities included starch and refined oil. Dry milled commodities included grits, meal, flour, and refined oil. The soya bean seed samples and processed commodities were analysed flubendiamide and its metabolite (Table 91). Flubendiamide des-iodo was only detected in aspired grain fractions (0.1 mg/kg).

Table 91 Results of flubendiamide in field corn processed commodities

Portion analysed	Residues, mg/kg	PF
kernel	0.15	-
starch	< 0.01	0.07
meal	0.14	0.93
flour	0.31	2.1
oil, refined	0.07	0.45
grits	0.04	0.24
oil, dry milled	0.02	0.12
aspirated grain fractions	48	318

Rice

A rice processing trial was conducted in Thailand with flubendiamide WDG 20 applied three times at 0.050 kg ai/ha and harvested 30 days after the last application (Balluff, 2008. 20074104). The day after harvest, samples of rice grain were dried before processing, grain humidity decreased from 28 to 14%. The samples were passed through a cleaning machine to separate out dirt and other seeds, leaving husked rice (rice with husks). In the next step the rice was passed through the dehusking machine to separate the husks from the grains resulting in huskes and brown rice (bran). In the last step the brown rice was treated with a polishing machine to obtain polished rice and bran. Residues of flubendiamide were 0.04 mg/kg in husked rice, < 0.01 mg/kg in brown and polished rice, 0.19 mg/kg in hulls and 0.03 mg/kg in bran. No residues of the des-iodo metabolite were found in the samples.

Cotton

In one study conducted in Greece (two trials), flubendiamide was applied four times at 0.072 kg ai/ha and seed samples harvested at 21 days PHI. Processed commodities were prepared simulating commertial practices (Billian and Wolters, 2005; RA3077/03). Residues of flubendiamide des-iodo were detected in samples of crude oil, at levels of 0.02 mg/kg. In one trial conducted in the USA, with an exaggerated rate of 0.525 kg ai/ha, residues of flubendiamide at 25 days PHI were 0.40 mg/kg in cotton seed, 0.03 mg/kg in hulls and < 0.01 mg/kg in meal and oil (Murphy, 2006; RCAMY003). The results of both studies are shown in Table 92.

Table 92 Results of flubendiamide from processing studies on cotton

Commodity	RA-3077/03 R 2003 0205/3 Seed = 0.15 mg/kg		RA-3077/03 R 2003 0855/8 Seed = 0.03 mg/kg		RCAMY003 Seed = 0.40 mg/kg		Mean PF
	Residues, mg/kg	PF	Residues, mg/kg	PF	Residues, mg/kg	PF	
extracted meal A/B	0.03/0.03	0.2/0.2	0.01/0.01	0.33/0.33	< 0.01	< 0.025	0.22
crude oil A/B	0.93/0.93	6.2/6.2	0.19/0.17	6.3/5.7	-	-	6.1
oil refined A/B	0.11/0.16	0.73/1.1	0.03/0.02	1/0.67	< 0.01	< 0.025	0.70

Residies in animal commodities

Poultry

Seventy-two laying hens (*Gallus gallus domesticus*; 12 hens/dosing and control group) were fed *ad libitum* for 28 consecutive days with feed containing flubendiamide at target dose rates of 0.02, 0.10, or 0.50 mg/kg (Billian and Eberhardt, 2005; MR-031/05). Eggs were collected daily during the dosing period and analysed for flubendiamide and flubendiamide-iodophthalimide. On day 29 after the first dose, the hens were sacrificed and liver, fat, muscle, and overlaying skin, together with any associated fat, were collected for analysis. Additionally, two groups of laying hens (12 hens/group) were fed at 0.5 mg/kg feed for 28 consecutive days in order to investigate the depuration of flubendiamide and its metabolite in eggs and tissues thereafter. Eggs were collected from both dosing groups on days 28–35 after the first dosing, and from the remaining hens additionally on days 36–42 after the first dose. On Day 36 and 43, respectively, the hens were sacrificed and liver, fat, muscle, and skin were collected for analysis.

Tissue and egg samples were analysed by HPLC-MS/MS according to method 00912 (LOQ of 0.01 mg/kg and LOD of 0.003 mg/kg). In order to express residues of flubendiamide-iodophthalimide as parent equivalents, the conversion factor of 1.285 was used (= ratio of molecular weights of 682.4 g/mol and 531.2 g/mol). All tissue and egg samples were analysed within 30 days of collection; freezer storage stability studies on poultry tissue and egg matrices were not conducted.

Table 93 shows the concentration of flubendiamide and its metabolites in eggs. Flubendiamide was detected in eggs at the second dosing level from day 13 (0.01 mg/kg) and at the highest level dose reached 0.06 mg/kg. No residues of flubendiamide iodo-phthalimide were detected in any egg sample at any dose. The depuration study has shown that flubendiamide was not present in egg samples 14 days after the last dose.

Table 93 Flubendiamide and its iodophthalimide metabolite (as parent) residues (mg/kg) in eggs

Day	0.02 mg/kg		0.1 mg/kg		0.5 mg/kg	
	Flubendiamide	Iodo-phthalimide	Flubendiamide	Iodo-phthalimide	Flubendiamide	Iodo-phthalimide
0	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003
1	< 0.003	< 0.003	< 0.003	< 0.003	0.02	< 0.003
5	< 0.003	< 0.003	< 0.003	< 0.003	0.04	< 0.003
7	< 0.003	< 0.003	< 0.003	< 0.003	0.05	< 0.003
9	< 0.003	< 0.003	< 0.003	< 0.003	0.06	< 0.003
13	< 0.003	< 0.003	0.01	< 0.003	0.05	< 0.003

Day	0.02 mg/kg Flubendiamide	 Iodo-phthalimide	0.1 mg/kg Flubendiamide	 Iodo-phthalimide	0.5 mg/kg Flubendiamide	 Iodo-phthalimide
14	< 0.003	< 0.003	0.01	< 0.003	0.06	< 0.003
19	< 0.003	< 0.003	0.01	< 0.003	0.05	< 0.003
21	< 0.003	< 0.003	0.01	< 0.003	0.06	< 0.003
23	< 0.003	< 0.003	0.01	< 0.003	0.06	< 0.003
26	< 0.003	< 0.003	0.01	< 0.003	0.06	< 0.003
27	< 0.003	< 0.003	0.01	< 0.003	0.06	< 0.003
28	< 0.003	< 0.003	0.01	< 0.003	0.02	< 0.003
Depuration study						
22	-	-	-	-	0.05	< 0.003
25	-	-	-	-	0.05	< 0.003
27	-	-	-	-	0.05	< 0.003
28	-	-	-	-	0.06	< 0.003
29	-	-	-	-	0.06	< 0.003
30	-	-	-	-	0.04	< 0.003
35	-	-	-	-	0.03	< 0.003
36	-	-	-	-	0.02	< 0.003
40	-	-	-	-	< 0.01	< 0.003
42	-	-	-	-	< 0.003	< 0.003

*Each value reported corresponds to the mean residue level from twelve animals.

The highest flubendiamide residues in tissues were observed for fat samples, showing evidence of a dose response (Table 94). Flubendiamide iodo-phthalimide was found only at the highest dose level in fat (0.02 mg/kg).

Table 94 Flubendiamide and its iodophthalimide metabolite (as parent) residues (mg/kg) in poultry tissues

Matrice	0.02 mg/kg Flubendiamide	 Iodo-phthalimide	0.1 mg/kg Flubendiamide	 Iodo-phthalimide	0.5 mg/kg Flubendiamide	 Iodo-phthalimide
Fat	0.01 (3)	< 0.003 (3)	0.07 (2), 0.06	< 0.01 (3)	0.27, 0.29, 0.25	0.02 (3)
Liver	< 0.003 (3)	< 0.003 (3)	0.02 (2), 0.01	< 0.003 (3)	0.07 (2), 0.06	< 0.003 (3)
Muscle	< 0.003 (3)	< 0.003 (3)	< 0.003 (3)	< 0.003 (3)	< 0.01, 0.01 (2)	< 0.003 (3)
Skin	< 0.01 (3)	< 0.003 (3)	0.03, 0.02 (2)	< 0.003 (3)	0.11 (3)	< 0.01 (3)

Tissue results of the depuration study at a 0.5 mg/kg feed level are shown in Table 95. Residues of flubendiamide decreased in fat from 0.27 mg/kg at the end of the dosing period to 0.04 mg/kg 7 days after the last dose to 0.01 mg/kg after 14 days (only one fat sample).

Table 95 Residue in tissues during the depuration phase of poultry treated at 0.5 mg/kg

Matrice	7 days after last dose Flubendiamide	 Iodo-phthalimide	14 days after last dose Flubendiamide	 Iodo-phthalimide
Fat	0.05, 0.04, 0.03	0.02, 0.01 (2)	< 0.01 (2), 0.01	< 0.01 (3)
Liver	< 0.01, 0.01 (2)	< 0.003 (3)	< 0.003 (3)	< 0.003 (3)
Muscle	< 0.003 (3)	< 0.003 (3)	< 0.003 (3)	< 0.003 (3)
Skin	0.02 (2), 0.01 (3)	< 0.01 (3)	< 0.003 (3)	< 0.01, < 0.003 (2)

Cattle

Thirteen lactating Holstein dairy cows (Bos taurus; three cows/treatment group and one control cow) were dosed orally, via capsule, for 29 consecutive days with flubendiamide at 2.5, 7.5, 30 or 50 mg/kg feed/day (Billian *et al*., 2005). Milk was collected twice daily during the dosing period. Additionally, a portion of the 25-day milk sample from the highest dose group was separated into milk fat and skim milk (whey), and each was analysed. On day 29 after the first application, the animals were sacrificed and liver, kidney, composite muscle, subcutaneous fat, omental fat, and perirenal fat were collected for analysis. Additionally, two lactating Holstein dairy cows were fed at 50 mg/kg for 29 consecutive days in order to investigate the depuration of residues in milk and tissues. Milk was collected from the first animal from day 29 to 35 after the first application, and from the second animal from day 29 to 49 after the first application. On day 36 and 50 after the first application, the animals were sacrificed and liver, kidney, composite muscle, subcutaneous fat, omental fat, and perirenal fat were collected for analysis. Tissue and milk samples were analysed for residue by HPLC-MS/MS according to method 00912. The tissue and milk samples in this study were analysed within 21 days of collection and freezer storage stability were not conducted.

Residues of flubendiamide and its iodophthalimide metabolite in milk are reported in Table 96. For the low and medium dose levels, residues in milk remained very low throughout the dosing period (up to 0.03 mg/kg). At higher dose levels (30 and 50 mg/kg), residues in milk reached a plateau level observed after 7-8 days of dosing). Milk originating from 26 days at the highest dose group separated into skim milk and milk fat. Flubendiamide residues were 0.02 mg/kg in milk whey and 1.5 mg/kg in milk fat. The iodophthalimide metabolite was only detected in milk fat, at 0.23 mg/kg.

Table 96 Summary of flubendiamide and its iodophthalimide metabolite residues in milka

Day	2.5 mg/kg		7.5 mg/kg		30 mg/kg		50 mg/kg	
	Flubendiamide	Iodophthalimide	Flubendiamide	Iodophthalimide	Flubendiamide	Iodophthalimide	Flubendiamide	Iodophthalimide
0	< 0.01	< 0.01	< 0.003	< 0.01	< 0.01	< 0.01	0.02	< 0.01
1	< 0.01	< 0.01	0.02	< 0.01	0.03	< 0.01	0.03	< 0.01
2-3	< 0.01	< 0.01	0.01	< 0.01	0.03	< 0.01	-	-
7-8	< 0.01	< 0.01	0.03	< 0.01	0.08	< 0.01	0.11	< 0.01
9-10	< 0.01	< 0.01	0.02	< 0.01	0.08	< 0.01	0.10	0.01
14	< 0.01	< 0.01	0.03	< 0.01	0.09	< 0.01	0.08	0.01
16-17	0.01	< 0.01	0.02	< 0.01	0.09	< 0.01	0.11	0.01
21	< 0.01	< 0.01	0.03	< 0.01	0.10	< 0.01	0.11	0.01
23-24	0.01	< 0.01	0.03	< 0.01	0.08	< 0.01	0.11	0.01
28	0.01	< 0.01	0.02	< 0.01	0.08	< 0.01	0.11	0.01
Depuration study								
21	-	-	-	-	-	-	0.14[b]	0.01[b]
23	-	-	-	-	-	-	0.14[b]	0.01[b]
28	-	-	-	-	-	-	0.16[b]	0.01[b]
29	-	-	-	-	-	-	0.15[b]	0.01[b]
31	-	-	-	-	-	-	0.10[b]	< 0.01[b]
32	-	-	-	-	-	-	0.10[b]	< 0.01[b]
33	-	-	-	-	-	-	0.09[b]	< 0.01[b]
34	-	-	-	-	-	-	0.06[b]	< 0.01[b]
35	-	-	-	-	-	-	0.06[b]	< 0.01[b]
36	-	-	-	-	-	-	0.08[c]	< 0.01[c]
40	-	-	-	-	-	-	0.06[c]	< 0.01[c]
41	-	-	-	-	-	-	0.05[c]	< 0.01[c]
42	-	-	-	-	-	-	0.04[c]	< 0.01[c]
46	-	-	-	-	-	-	0.02[c]	< 0.01[c]
47	-	-	-	-	-	-	0.01[c]	< 0.01[c]
49	-	-	-	-	-	-	0.02[c]	< 0.01[c]

[a] mean of three values;

[b] mean of two values;

[c] single value

The individual residue levels in animal tissue are summarised in Table 97. Residues of flubendiamide were observed in tissues of all animals in all dose groups, with the highest levels in fat. Flubendiamide iodophthalimide was detected at the lower dose level only in fat.

Table 97 Summary of flubendiamide and its iodophthalimide metabolite residues in beef tissue

Matrice	2.5 mg/kg		7.5 mg/kg		30 mg/kg		50 mg/kg	
	Flubendiamide	Iodo-phthalimide	Flubendiamide	Iodo-phthalimide	Flubendiamide	Iodo-phthalimide	Flubendiamide	Iodo-phthalimide
liver	0.03 0.06 0.04	< 0.01 < 0.01 < 0.01	0.10 0.23 0.11	< 0.01 < 0.01 < 0.01	0.52 0.36 0.26	< 0.01 < 0.01 < 0.01	0.58 0.33 0.47	< 0.01 < 0.01 < 0.01
kidney	0.03 0.06 0.05	< 0.01 < 0.01 < 0.01	0.10 0.20 0.13	< 0.01 < 0.01 < 0.01	0.54 0.39 0.28	0.01 0.01 0.01	0.57 0.33 0.42	0.02 0.01 0.01
muscle	< 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01	0.02 0.04 0.03	< 0.01 < 0.01 < 0.01	0.08 0.06 0.06	< 0.01 < 0.01 < 0.01	0.12 0.09 0.14	0.01 < 0.01 0.02
fat (1)*	0.05 0.08 0.06	< 0.01 < 0.01 < 0.01	0.13 0.15 0.16	0.02 0.02 0.01	0.66 0.51 0.53	0.07 0.07 0.04	0.82 0.36 0.77	0.08 0.06 0.13
fat (2)*	0.06 0.10 0.09	< 0.01 < 0.01 < 0.01	0.19 0.25 0.16	0.03 0.02 0.02	0.76 0.60 0.61	0.17 0.11 0.12	1.2 0.75 1.2	0.16 0.23 0.19
fat (3)*	0.06 0.10 0.10	< 0.01 0.01 < 0.01	0.22 0.27 0.16	0.02 0.03 0.03	0.63 0.63 0.67	0.15 0.11 0.14	1.1 0.76 1.2	0.21 0.17 0.27

* Subcutaneous Fat (1); Omental Fat (2); Perirenal Fat (3)

Flubendiamide and flubendiamide iodophthalimide from the depuration phase found in tissues are summarised in Table 98. During the depuration phase, residues in milk decline close to the LOQ. The results show that residue decline rapidly within one week post dosing. In the following weeks residues still declined but more slowly, especially for fat tissues.

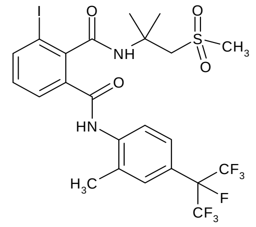
Table 98 Residue in tissues during the depuration phase of cow treated at 50 mg/kg

Matrice	7 days after last dose		14 days after last dose	
	Flubendiamide	Iodo-phthalimide	Flubendiamide	Iodo-phthalimide
Liver	0.15	< 0.01	0.11	< 0.01
Muscle	0.18	0.01	0.14	< 0.01
Skin	0.04	0.02	0.03	< 0.01
fat (1)*	0.21	0.13	0.19	0.06
fat (2)*	0.22	0.15	0.06	0.09
fat (3)*	0.23	0.17	0.09	0.08

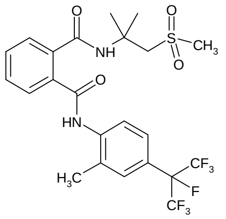
* Subcutaneous Fat (1); Omental Fat (2); Perirenal Fat (3)

APPRAISAL

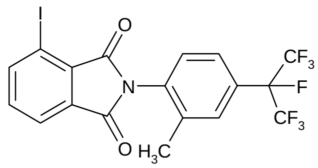
Flubendiamide is an insecticide for use in a broad number of annual and perennial crops against a wide range of lepidopteran pests. The compound is being evaluated by the 2010 JMPR as a new compound, for both residue and toxicological aspects. Data was provided on metabolism of flubendiamide in farm animals and plants, methods of analysis, GAP information, supervised residue trials on various crops, storage stability, processing and animal feeding studies. Below are the chemical structures of flubendiamide and its major metabolites in plant (des-iodo) and animals (iodophthalimide).



Flubendiamide



Flubendiamide-des-iodo

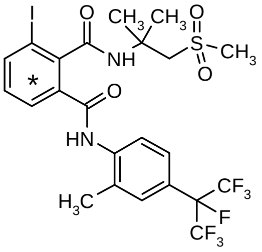


Flubendiamide-iodophthalimide

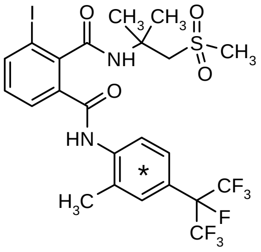
Metabolism in animals

The metabolism of flubendiamide in rats was evaluated by the WHO panel of the JMPR at the present Meeting.

The positions of the radiolabels are shown in the figures below.



[phthalic acid ring-UL-^{14}C] flubendiamide



[aniline ring-UL-^{14}C] flubendiamide

Two metabolism studies were conducted on laying hens using similar experimental designs. In each study, radio-labelled doses were orally administered to six birds for 14 days. In one study, the hens were dosed with [phthalic acid ring-UL-^{14}C]flubendiamide at 1.0 mg/kg bw/day (16.95 ppm in the diet) and in the second study with [aniline ring-UL-^{14}C] flubendiamide at 0.71 mg/kg bw/day (8.86 ppm in the diet). Eggs and excreta were collected once daily and hens were sacrificed 24 hours after the last dose.

About 91 and 98% of the administered cumulative dose of [phthalic acid ring] and [aniline ring-UL-^{14}C]flubendiamide, respectively, was recovered from organs, tissues, eggs, and excreta. The majority of the radioactivity (62–66%) was detected in the excreta, 24.4% in tissues and 5.1–7.7% in eggs. In tissues, residues concentrated in fat (18–12.2 mg/kg eq.), followed by liver (4.0–3.0 mg/kg eq.) and muscle (2.9–2.6 mg/kg eq.). Residues in eggs increased during the experiment, from 0.15–0.33 mg/kg eq. in the first 4 days to 2.9–2.6 mg/kg eq. towards the end of the dosing period. Flubendiamide accounted for 92–93% TRR in eggs, 95% TRR in muscle, 98–97% TRR in fat and 82% TRR in liver. The metabolite flubendiamide-benzyl alcohol was present in eggs and tissues, accounting for less than 10% TRR. Traces of flubendiamide-iodophtalimide was found in eggs and tissues from the [phthalic acid ring] dosing, and accounted for 1.6% TRR (0.20 mg/kg eq.) in fat from the [aniline acid ring] experiment.

In the goat metabolism studies, a single goat received daily for 4 days by gavage either [phthalic acid ring-UL-^{14}C]flubendiamide at a mean dose rate of 4.83 mg/kg bw/day (176 ppm in the diet) or [aniline ring UL-^{14}C] flubendiamide at a daily dose rate of 5 mg/kg bw/day (370 ppm in the diet). Each goat was milked in the morning immediately prior to each administration, 8, 24, 32, 48, 56, 72, and 77 hours (at sacrifice) after the first dose and excreta were collected in intervals of 24 hours and at sacrifice, when tissues were sampled.

Until sacrifice, 53.7% of the administered [phthalic acid ring-UL-^{14}C]flubendiamide was recovered, mostly in the faeces (44.2%). Tissues accounted for 8.7% of the dose and milk 0.5%. The highest residue levels were observed in fat (9.9 mg/kg eq.) and liver (10.1 mg/kg eq.), followed by kidney (2.4 mg/kg eq.), muscle (0.83 mg/kg eq.) and milk (0.70 mg/kg eq.). The parent compound accounted for 78.3–90.6% of TRR. Flubendiamide-iodophthalimide was detected in milk and tissues, the highest levels found in fat (1.0 mg/kg eq and 11% TRR) and liver (0.24 mg/kg eq. and 2.4% TRR). Liver contained six other metabolites (< 5%TRR) at levels ranging from 0.053 (F-iodo-alkylphthalimide) to 0.39 (F-hydroxy) mg/kg eq. About 25% of the totally administered dose of [aniline ring UL ^{14}C]flubendiamide, was excreted until sacrifice with 24% in the faeces. Milk accounted for 0.4% and tissues for 15% of the totally administered doses. The highest radioactivity was measured in fat (21 mg/kg eq.), followed by liver (13.5 mg/kg eq.), kidney (4.4 mg/kg eq.), muscle (1.5 mg/kg eq.) and milk (1.5 mg/kg eq.). The parent compound was the main residue component (72 to 93%TRR). The major metabolite, flubendiamide-iodophthalimide accounted for approx. 17% of the TRR in milk (0.24 mg/kg eq), 24% in fat (5 mg/kg eq) and 8.4% TRR in muscle (0.13 mg/kg eq). Minor identified metabolites accounted for less than 6% TRR each. The major metabolic pathway of flubendiamide in hens and goats was the oxidation of the methyl groups to form a primary alcohol (hydroxylation), further oxidation of the aliphatic alcohol to a carboxylic acid group followed by conjugation with glucuronic acid, which was exclusively found in the excreta and in the bile. A minor reaction was the cleavage of the respective amide bond of flubendiamide and the cyclisation to flubendiamide-iodophthalimide and flubendiamide-iodo-alkylphthalimide.

Metabolism in plants

All plant metabolism studies involved foliar application of flubendiamide to reflect the intended field use patterns. Additionally, the greenhouse metabolism studies for cabbages and tomatoes both made use of a quartz-ceiling greenhouse to make light irradiation conditions similar to field conditions: photolytic studies demonstrating a mean photolytic half-life of 5.5 days support this decision. A metabolism study was conducted on cabbages in a greenhouse using [phthalic acid ring-UL-14C]- and [aniline ring-UL-14C]-flubendiamide applied to immature plants at 300 µg/plant. Samples were collected 3 weeks and 6 weeks after application (maturity). Residues in cabbage heads represented < 0.1% of the applied radioactivity, AR. Flubendiamide was the main compound detected in the loose outer leaves (> 90% AR), and flubendiamide-des-iodo and flubendiamide-3-OH were the main metabolites, reaching up to 1.7% AR.

Cherry tomato plants were either treated in a glasshouse with [phthalic acid ring-UL-^{14}C] or [aniline ring-UL-^{14}C]-labelled flubendiamide at 125 µg/branch of fruits (25 µg/fruit) and 800 µg/branch of leaves. Samples were collected at day 0, and 1, 2 and 4 weeks after application. Total radioactivity decreased during the experiment, from about 3.3 to 1.4 mg/eq. in fruits (99–67% TRR) and 44–45 to 16.5–14.9 mg/kg eq. in leaves (100–67% TRR). The surface rinsate contained most of the radioactive residues. Analysis of untreated plant parts four weeks after treatment showed less than 0.5% of the AR. Flubendiamide was the main component detected in fruits and amounted to 1.27 and 1.43 mg/kg eq. after four weeks for the phthalic acid and aniline label, respectively (63.4 and 66.3% AR). Flubendiamide-des-iodo accounted for up to 0.3% TAR (up to 0.007 mg/kg eq.) and flubendiamide-3-hydroxy for up to 0.2% AR. Flubendiamide was also the main component found in leaves (over 80% TAR after 4 weeks).

The metabolism of flubendiamide in apples was studied by applying [phthalic acid ring-UL-^{14}C]- and [aniline ring-UL-^{14}C] flubendiamide as an EC formulation to two apple trees (one for each label) at 0.11 kg ai/ha. Samples of apples and leaves were collected at 0, 7, 14, 28, and 56 days after treatment. About 100% of TRR was recovered from the fruits, with residues below 0.05 mg/kg at each harvest date for each label, mostly present in the apple rinses (over 60% TRR at 14 days PHI). Residues in fruit pellets were < 0.005 mg/kg eq. Residues in leaves dropped from 4.5 to about 1.5 mg/kg eq. at day 56, mostly recovered in the ACN leaf extracts. Residues in the leaf pellets increased during the experiment to about 10% TRR. Flubendiamide was the major compound detected in both label experiments, accounting for about 70% TRR at 14 days PHI in fruit (0.014 mg/kg eq.) and 78% TRR in leaves. Flubendiamide-des-iodo was at ≤ 0.002 mg/kg in fruit in all sampling times. In leaves, the levels were below 0.5 mg/kg (< 5%TRR).

The metabolism of flubendiamide in sweet corn was investigated using [phthalic acid ring-UL-^{14}C] and [aniline ring-UL-^{14}C] flubendiamide applied four times at 0.16 kg ai/ha. Forage (includes husks) and sweet corn samples were collected one day after the fourth treatment. TRR of forage and fodder was within the range of 0.29 to 0.60 mg/kg eq., with over 85% TRR found in the acetonitrile/water extracts. TRR derived by combustion of sweet corn and corn grain samples from phthalic acid ring label experiments were 0.01 and 0.02 mg/kg eq., respectively, and < 0.005 mg/kg eq. in samples from the aniline ring label experiment. ACN/water extracts of sweet corn and corn grain of the phthalic acid label represented 37 and 15% TRR, respectively; methanol under reflux and alkaline conditions extracted an additional 20 and 13% TRR. Over 75% TRR found in forage and fodder was flubendiamide (0.21 to 0.51 mg/kg eq.). Flubendiamide-des-iodo was detected at levels from 0.03 to 0.05 mg/kg eq, representing up to 18% TRR (forage).

The metabolism of flubendiamide in rice was investigated by applying a [phthalic acid ring-UL-^{14}C]flubendiamide suspension (49.6 ± 0.5 µg eq./mL) to plants just before ear emergence. After drying of the droplets on the plant surface, the plants (four pots) were transferred to the greenhouse. Samples were taken at time zero, four and nine weeks after application. The higher radioactive residues were found in leaves and stems, decreasing from 2.1 mg/kg eq. at time zero to around one third of the initial value four weeks after application (immature plant), mainly due to plant growth, and increased to 1.4 mg/kg eq., probably due to loss of moisture. The TRRs in seed after 9 weeks was 0.023 mg/kg eq., mostly recovered from the solids. Flubendiamide was the predominant constituent of the residue in stems and leaves for all sampling times (over 90%TRR). Flubendiamide-des-iodo accounted for 4.1% of TRR and fubendiamide-3-OH was identified as a minor constituent. Flubendiamide-benzylalcohol and flubendiamide-benzoic acid were also identified. Hulls from the 9 week sampling contained 0.05 mg/kg eq., 88% as parent compound and 4% as the des-iodo metabolite.

In summary, the metabolism of flubendiamide after foliar application on plants involved mostly the des-iodination of the parent compound to yield flubendiamide-des-iodo followed by hydroxylation to flubendiamide-3-OH and the stepwise oxidation of the methyl group at the aniline ring leading to flubendiamide-benzylalcohol and flubendiamide-benzoic acid. In tomatoes, the label-specific metabolite flubendiamide-des-anilino was also observed. In apple fruits, a third route was also observed, involving the elimination of the amino-ethyl-sulfonyl substituent leading to flubendiamide-iodophthalimide and the label-specific metabolite flubendiamide-3-iodo-phthalic acid. In corn, the only metabolic reaction observed was the reductive deiodination to yield flubendiamide-des-iodo. These studies indicate little evidence of residue translocation within the plant; thus, surface residues may be expected in the crop field trial studies.

Environmental fate

The supported uses of flubendiamide concern foliar application only. Based on the 2009 FAO Manual, no studies on the fates and behaviour in soil are required for this type of use. Any metabolite from a field dissipation study that may have an impact on plant residues is covered by the rotational crop study.

Hydrolysis

Flubendiamide comprised more than 95% of the residue at 25 ± 1 °C in pH 4.0, 5.0, 7.0 and 9.0 buffer solutions over a 30 day study period; and more than 95% of the residue at 50.0 ± 0.1 °C in pH 4.0, 7.0 and 9.0 buffer solutions over a 5 day study period. Therefore, flubendiamide is considered hydrolytically stable from pH 4.0 to 9.0.

Photolysis

Flubendiamide was irradiated in distilled water, natural water, and distilled water containing 1% acetone with artificial light for up to 168 hours. An average half-life of 5.5 days was determined in distilled water and distilled water with acetone, while a half-life of 4.3 days was reported in natural waters. The results of the environmental fate studies indicate that degradation of flubendiamide is more likely to occur by photolysis than hydrolysis.

Residues on succeeding crops

The metabolism of flubendiamide after spray application onto bare soil was investigated in spring wheat, Swiss chard and turnips. [Phthalic acid ring-UL-^{14}C]flubendiamide and [aniline ring-UL-^{14}C]flubendiamide were applied by spray application (day 0) at a rate of 0.44 kg ai/ha, based on the projected annual field rate of 0.42 kg ai/ha. Crops of the first, second and third rotation were sown at day 29, day 135 and day 274, respectively. Plants of the first rotation were grown under natural temperature and light conditions and for the second and third rotation, in the greenhouse.

The maximum TRR (0.07 mg/kg) in plants treated with [phthalic acid ring-UL-^{14}C]flubendiamide was observed in wheat straw of the first rotation, which decreased to 0.05 mg/kg eq. in the third rotation. During this period, residues in forage increased from 0.013 to 0.016 mg/kg and remained practically constant in grain (0.003 mg/kg eq.). Residues in Swiss chard decreased from 0.022 to 0.015 mg/kg eq. In turnip leaves and roots, residues in the first rotation were 0.011 and 0.006 mg/kg, respectively, remaining practically constant at the second and third rotation (0.005–0.006 mg/kg eq. and 0.002 mg/kg eq., respectively). The maximum TRR (0.137 mg/kg) in plants treated with [aniline ring-UL-^{14}C]flubendiamide was observed in wheat straw of the first rotation, decreasing to 0.068 and 0.039 mg/kg in the second and third rotation. Similarly, the TRRs in wheat hay decreased from 0.045 mg/kg (first rotation) to 0.021 mg/kg (third rotation). The TRRs in forage and Swiss chard ranged from 0.009 mg/kg to 0.019 mg/kg for all rotations. The lowest residues were present in grain, turnip leaves and turnip roots amounting to ≤ 0.006 mg/kg for all rotations.

About 80–90% of the TRR was extracted from the majority of samples using acetonitrile/water in both experiments. Wheat grain of the first rotation accounted for 62 to 70% TRR, which decreased to about 14% TRR after enzymatic treatment (< 0.001 mg/kg). Unchanged parent compound was the main component of all plant samples and accounted for 22–88% of the TRR, except for grain. In grain, only 4% to 8% (< 0.001 mg/kg) of the TRR (0.003 mg/kg) was due to flubendiamide in the first rotation, decreasing to 2.2 and 0.5% TRR in the second and third rotations. The main portion of the TRR in grain was due to very polar radioactivity found in aqueous phases following conventional and enzymatic extraction. A major metabolite in confined rotational crops in the phthalic acid ring experiment was flubendiamide-des-iodo, accounting for up to 10.8% of the TRR in Swiss chard of the second rotation. The highest absolute amount of flubendiamide-des-iodo-alkylphthalimide was 0.01 mg/kg in straw of the second rotation, corresponding to 16.0% of the TRR. In the aniline ring experiment, flubendiamide-benzyl alcohol and benzoic acid were detected in some of the plant samples up to 1.4% TRR, each accounting for 0.001 mg/kg as a maximum.

The main metabolic reaction of flubendiamide in confined rotational crops was the reduction of the parent compound by elimination of the iodine-substituent. Other metabolic reaction include the elimination of the N-aryl-moiety, hydroxylation of the parent compound to form flubendiamide-benzyl alcohol which was further oxidised to the carboxylic acid, probably in soil.

In summary, total residues in the rotated crops of wheat grain, turnip leaves and roots were < 0.01 mg/kg. The rotated crop matrix with the highest level of flubendiamide was wheat straw, which contained a maximum level of 0.10 mg/kg flubendiamide in the reported studies. The highest reported level of flubendiamide in any human food item in the rotational crop studies was Swiss chard, where a maximum level of 0.015 mg/kg flubendiamide was found.

Methods of analysis

The analytical method developed for the determination of flubendiamide and flubendiamide-des-iodo residues in/on plant material (00816/M001), involves two successive microwave extractions, the first with acetonitrile/0.01% HCl and the second with acetonitrile/0.01% HCl/water. Following column clean-up the residues are eluted with cyclohexane/ethyl acetate, and dissolved in acetonitrile/water for quantification by LC-MS/MS. Oil of plant origin samples are dissolved in hexane, extracted with acetonitrile and partitioned with hexane before LC-MS/MS. Two MRM transitions for quantitation and confirmation were monitored for each analyte (flubendiamide: m/z 681→254 and m/z 681→274; flubendiamide-des-iodo: m/z 555→254 and m/z 555→274). The method was validated for a variety of crops, including tomatoes, grains, beans, cabbages and cotton and submitted also to independent laboratory validation. The limit of quantification (LOQ) for both analytes is 0.01 mg/kg for all sample materials.

The extraction efficiency of microwave and shaker procedures was evaluated using data from radiovalidation of method 00816/M002 with corn (microwave) and the metabolism study with [phthalic acid ring-UL-^{14}C]flubendiamide onto corn plants (blender). The microwave and the blender procedures extracted 100 and 86% TRR, respectively. The method that used the shaker extraction was validated for a LOQ of 0.02 mg/kg for flubendiamide and its des-iodo metabolite.

A HPLC/UV method (C18 column/ 260 nm) was developed to analyse flubendiamide and the des-iodo and 3-OH metabolites in tea samples. The samples were homogenized with ACN/0.1N HCl, extract with n-Hexane/EtOAc and cleaned up with graphite carbon, C18 and NH2 SPE. To analyse flubendiamide and the des-iodo metabolite, an addition clean-up step using silica SPE was included before HPLC/UV. The method was validated for flubendiamide at a LOQ of 0.01 mg/kg.

Method 00912 was developed for the determination of flubendiamide and the metabolite flubendiamide-iodophthalimide in animal commodities (muscle, liver, kidney, milk, fat and egg). The residues are extracted with acetonitrile/water and flubendiamide-iodophthalimide is completely converted to flubendiamide-des-alkylamino and its isomer under mild alkaline conditions. The residues are subjected to column clean-up and analysed by LC-MS/MS. The method uses matrix-matched standards for calibration or internal deuterated standards for calibration. LOQ for flubendiamide and its metabolite was 0.01 mg/kg and for flubendiamide-iodophthalimide was 0.013 mg/kg, expressed as parent equivalents compound. The transition for quantification was m/z 681→ 254 for flubendiamide and m/z 548 → 504 for the flubendiamide-des-alkylamino. Another transition for confirmation was monitored for each analyte.

Due to thermolability of flubendiamide, GC-based multiresidue methods are not recommended. HPLC-based multiresidue methods may be applicable, but no information addressing this approach were submitted.

Stability of residues in analytical samples

Flubendiamide and its des-iodo metabolite residues were shown to be stable in samples of tomatoes, oranges, beans, grapes, olive oil, must grapes and cabbages fortified at 0.10 mg/kg and stored under frozen conditions up to 18 months. Another study conducted with cotton seed and processed commodities, wheat and processed commodities, wheat forage and straw, potatoes and tomato paste fortified at 0.15 mg/kg, the compounds showed stability over one year periods.

No stability studies were conducted with flubendiamide in animal commodities, but information from the animal feeding studies showed that the samples were analysed less than a month after collection.

Definition of the residue

Metabolism studies conducted with flubendiamide in laying hens showed that the highest residues are found in fat and liver. TRR in fat (17.7 mg/kg eq) were higher than in muscle (1 mg/kg eq). The parent compound is the main residues found in edible commodities, accounting for 80 to 95% TRR. The main metabolite detected, flubendiamide-benzylalcohol, accounted for less than 10% TRR, mainly found in liver.

Goat metabolism studies also showed the highest residues in fat and liver. The ratio of flubendiamide residues in muscle vs fat was 1:12. The parent compound accounted for over 70% TRR and the main metabolite, flubendiamide-iodophthalimide, accounted for up to 24% TRR in fat and up to 17% TRR in milk, but less than 10% TRR in other tissues.

Plant metabolism studies conducted on plants have shown that flubendiamide accounted for over 90% of the residues. The main metabolite, flubendiamide-des-iodo, accounts for less than 10%

TRR. Succeeding crop studies have shown that, with the exception of flubendiamide-des-iodo-alkylphthalimide present in straw of the second rotation (0.01 mg/kg eq., 16.0% TRR), no other metabolite exceeded 11% TRR.

Proposed definition of the residue (for compliance with the MRL and for estimation of dietary intake) for plant commodities: *flubendiamide*

As the flubendiamide-iodophthalimide metabolite was found in human foods (fat and milk), the Meeting determined that it was appropriate to include this metabolite in the dietary risk assessment.

Definition of the residue (for compliance with the MRL) for animal commodities: *flubendiamide.*

Definition of the residue (for estimation of dietary intake) for animal commodities: *flubendiamide and flubendiamide-iodophthalimide.*

Results of the poultry and bovine feeding studies were consistent with the metabolism studies in showing significantly higher residue levels in fat than muscle. Flubendiamide has a Log K_{ow} of 4.2. Based on this information, the Meeting concluded that flubendiamide is fat soluble.

The residue is fat-soluble.

Residues of supervised trials on crops

With the data gathering methods that were used, residues of both flubendiamide and flubendiamide-des-iodo were analysed in all supervised trials, except tea trials. Flubendiamide des-iodo, was detected only in animal feed commodities and in some processed commodities.

Greece and the Netherlands submitted GAP for tomato and pepper uses. This was the only GAP submitted by any European countries for flubendiamide. Therefore, except for tomatoes and peppers, no European residue data were directly used for maximum residue level estimations.

In the USA, trials were conducted side-by-side applying the pesticide in concentrated and high volume spray in pome fruits and stone fruits. Generally the high volume application gave rise to higher residues. These trials were not considered independent and the higher residues were used for estimation of residue levels.

Pome fruits

Residue trials were conducted on apples and pears in Europe, Canada and the USA. Flubendiamide is registered in the USA in pome fruits with a GAP of 3 × 0.14–0.175 kg ai/ha (minimum of 93.4 L water/ha) and 14 days PHI.

In 12 trials conducted in the USA and Canada in apples at GAP rate, using diluted (1800 to 3000 L/ha) or concentrated sprays (360 to 700 L/ha), residues of flubendiamide within 14 days PHI were 0.13, 0.18 (2), 0.19, 0.21, 0.23 (2), 0.27, 0.30, 0.41, 0.47, and 0.48 mg/kg.

In six trials conducted in the USA in pears at GAP rate, also using diluted and concentrated sprays, residues of flubendiamide at 14 days PHI were 0.09, 0.23, 0.33, 0.36, 0.37, and 0.59 mg/kg.

Residues of flubendiamide in 18 trials conducted on apples and pears in the USA and Canada according to GAP for pome fruit in the USA belong to the same population and can be combined as follow: 0.09, 0.13, 0.18 (2), 0.19, 0.21, 0.23 (3), 0.27, 0.30, 0.33, 0.36, 0.37, 0.41, 0.47, 0.48, and 0.59 mg/kg.

Based on the USA and Canada trials conducted on apples and pears according to USA GAP for pome fruit, the Meeting estimated a maximum residue level of 0.8 mg/kg, a STMR of 0.25 mg/kg, and a HR of 0.59 mg/kg for flubendiamide in pome fruits.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 0.8 mg/kg.

Stone Fruits

Cherries

Flubendiamide is registered in the USA in stone fruits with a GAP of 3 × 0.14 kg ai/ha (minimum of 93.4 L water/ha) and 7 days PHI.

Eight trials were conducted in the USA and Canada in cherries according to US GAP. Residues of flubendiamide at 7 days PHI were 0.19, 0.25, 0.48, 0.57, 0.60, 0.63, 0.99 and 1.0 mg/kg.

Peaches and Nectarines

Nine trials were conducted in the USA and Canada in peaches according to US GAP. Residues of flubendiamide at 7 days PHI were 0.20 (2), 0.23, 0.24, 0.30, 0.32, 0.35, 0.39 and 0.40 mg/kg.

Plums

Six trials were conducted according to GAP in the USA in plums. Residues of flubendiamide found in plums at a 7 day PHI were: 0.02, 0.03, 0.05, 0.09, 0.14, and 0.50 mg/kg.

The Meeting recommended a group maximum residue level for stone fruit, based on the cherry data. The Meeting estimated a maximum residue level of 2 mg/kg, a STMR of 0.585 mg/kg and a HR of 1.0 mg/kg for flubendiamide in stone fruit.

Grapes

Twelve trials were conducted on grapes in the USA at GAP (3 × 0.14 kg ai/ha and 7 days PHI). Residues were 0.12 (2), 0.19 (2), 0.22, 0.40, 0.43, 0.47, 0.67, 0.68, 0.69 and 0.81 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg, a STMR of 0.415 mg/kg and a HR of 0.81 mg/kg for flubendiamide in grapes. The maximum residue level estimate derived from use of the NAFTA statistical calculator was 1.8 mg/kg.

Brassica vegetables

Broccoli

Flubendiamide is registered in Brassica vegetables in Australia at a maximum rate of 3 × 0.048 kg ai/ha (0.0048 kg ai/hL) and 3 days PHI. Nine residue trials were conducted in Australia in 2006 with broccoli; three trials conducted at GAP gave residues of flubendiamide of 0.13, 0.22 and 0.25 mg/kg.

Three trials were conducted on broccoli in the USA at 3 × 0.034 kg ai/ha, giving residues at a 1 day PHI and a 3 day retreatment interval (RTI) of 0.12, 0.16, and 0.23 mg/kg. GAP in the USA for Brassicas is 2 × 0.034 kg ai/ha. A broccoli residue decline study in California revealed negligible decline over a 7 day period, making it likely that the additional treatment would result in residues > 25% higher than would be expected from two treatments as allowed by GAP. Therefore, the broccoli trials in the USA were not considered further for MRL setting purposes.

Cauliflower

Three trials were conducted in the USA at 3 × 0.034 kg ai/ha, with residues at 1 day PHI of < 0.01, 0.02 and 0.03 mg/kg.

Cabbages

Eighteen trials were conducted on cabbages in Australia in 2006/2007. In six trials conducted according to GAP for Brassicas, residues of flubendiamide at 3 days PHI were 0.19, 0.20, 0.27, 0.43, 0.92 and 2.7 mg/kg. Twelve trials conducted at higher rates (0.072 to 0.1 kg ai/ha) gave residues within the same range.

Six trials were conducted on cabbages in the USA at 3 × 0.034 kg ai/ha, 1 day PHI, and 3 day RTI. Available residue decline data did not allow the Meeting to conclude that the initial treatment would not contribute significantly to the residue level at harvest. Therefore, the cabbage trials in the USA were not considered further for MRL setting purposes.

Brussels sprouts

Twelve trials were conducted on Brussels sprouts in Australia in 2006. In four trials conducted according to GAP residues of flubendiamide at 3 days PHI were 0.08, 0.23, 0.50 and 1.1 mg/kg. In eight trials conducted at higher rate gave residues at 3 days PHI ranging from 0.09 to 1.5 mg/kg.

The Meeting decided it was appropriate to recommend a group MRL for Brassica vegetables. Based on the cabbage data from Australia, the Meeting estimated a maximum residue level of 4 mg/kg, a STMR of 0.365 mg/kg and a HR of 2.7 mg/kg for flubendiamide in Brassica vegetables.

Fruiting vegetables, Cucurbits

Flubendiamide is registered in the USA for cucurbit vegetables at 3 × 0.05 kg ai/ha, 7 day RTI, and 1 day PHI. A total of seventeen field trials were conducted with cucumbers (six), summer squash (five) and melons (six) using five spray applications rather than three as specified by GAP. Residue levels in all cucurbit vegetables were so low that it is unlikely that residues from the first two spray treatments had any significant affect on the residue levels that would have been measured after three spray treatments. Accordingly, the Meeting decided to accept these trials for the purpose of MRL estimation.

Cucumber residues were as follows: < 0.01 (2), 0.01 (2), and 0.03 (2) mg/kg. Summer squash residues were as follows: < 0.01, 0.01 (2), 0.02, and 0.04 mg/kg. Melon residues were as follows: 0.02 (2), 0.04, 0.05, 0.07, and 0.09 mg/kg.

Noting the similarity in residue levels among cucumbers, summer squash, and melons, the Meeting recommended a group maximum residue level for cucurbit vegetables based on the melon data. The Meeting estimated a maximum residue level of 0.2 mg/kg, a STMR of 0.045 mg/kg and a HR of 0.09 mg/kg for flubendiamide in cucurbit vegetables.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 0.18 mg/kg.

Fruiting vegetables, other than Cucurbits

Peppers

Flubendiamide is registered in Australia in tomatoes and peppers at a maximum rate of 0.072 kg ai/ha (0.0072 kg ai/hL). Twenty four field trials were conducted on peppers in Australia in 2007. In seven trials conducted according to GAP, residues at a 1 day PHI were: 0.04, 0.06 (2), 0.09, 0.16, 0.21 and 0.37 mg/kg.

Flubendiamide is registered in the USA for use in fruiting vegetables (except cucurbits) at a maximum rate of 3 × 0.05 kg ai/ha. Eleven trials conducted on peppers in the USA at 5 × 0.05 kg ai/ha (1 day PHI and a 3 day retreatment interval) gave residues ranging from < 0.01 to 0.14 mg/kg. As these trials were not in accord with GAP, they were not considered further.

Flubendiamide is registered to be used in Greece and the Netherlands for use in greenhouses on peppers at 2 × 0.006 kg ai/hL (0.096 kg ai/ha) with 1 day PHI. Fourteen glasshouse trials were conducted on peppers in France, Germany, Italy and the Netherlands using two or three spray treatments. Only four of these trials were according to GAP, giving residues as follows: 0.05, 0.06, 0.07, and 0.11 mg/kg.

The trials conducted on peppers in Australia and Europe according to GAP gave different residue populations. The Australian data gave the higher residues and were used as the basis for the estimations.

The Meeting estimated a maximum residue level of 0.7 mg/kg, a STMR of 0.09 mg/kg and a HR of 0.37 mg/kg for flubendiamide in peppers.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 0.7 mg/kg.

Chili pepper, Dry

Using the default dehydration factor of 10 to extrapolate from peppers to dried chilli peppers, the Meeting estimated a maximum residue level of 7 mg/kg and a STMR of 0.9 mg/kg for flubendiamide in dry chilli peppers.

Tomatoes

Field trials were conducted in Australia on tomatoes. In five trials conducted according to Australian GAP, residues at a 1 day PHI were: 0.04, 0.07, 0.35 (2) and 0.63 mg/kg. The trials conducted at higher and lower rates gave residues within the same range.

In eight field trials conducted on tomatoes in the USA in 2004 using five spray applications instead of three as specified by USA GAP (1 day PHI and 3 day RTI), residues ranged from 0.01 to 0.16 mg/kg. These trials were not considered further for MRL estimates because they do not reflect USA GAP and show residue levels lower than those conducted in Australia.

Flubendiamide is registered to be used in Greece in greenhouses in tomatoes at 2 × 0.006 kg ai/hL (0.12 kg ai/ha) with a 3 day PHI. In the Netherlands, GAP rate is the same, but the PHI is 1 day. Trials were conducted for greenhouse tomatoes in France, Germany, Italy, the Netherlands, Portugal and Spain using the GAP application rate. However, the trials conducted with three applications are not in accord with GAP, and should not be directly used for MRL-estimating purposes.

Five trials conducted in Germany, Spain and Portugal evaluated against Netherlands GAP gave residues at 1 day PHI of 0.06 (2), 0.09, 0.10, 0.11 (2) and 0.12 mg/kg.

The trials from Australia resulted in higher residues than those conducted in Europe and are appropriate for use in MRL estimations.

The Meeting estimated a maximum residue level of 2 mg/kg, a STMR of 0.35 mg/kg and a HR of 0.63 mg/kg for flubendiamide in tomatoes.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 2.9 mg/kg.

Sweet corn

Flubendiamide is registered in the USA in sweet corn at a maximum rate of 4 × 0.10 kg ai/ha with a 1 day PHI. In 11 trials conducted according to GAP, residues in corn-on-the-cob were < 0.01 (10) and 0.01 mg/kg.

The Meeting estimated a maximum residue level of 0.02 mg/kg, a STMR and a HR of 0.01 mg/kg for flubendiamide in sweet corn (corn-on-the-cob).

Leafy vegetables

Lettuce, Head

Flubendiamide is registered in Australia for leafy vegetables, including leaf and head lettuce, at a maximum rate of 3 × 0.048 kg ai/ha and a 1 day PHI.

In six Australian trials conducted on 2006 according to GAP, residues of flubendiamide at 1 day PHI were 0.16, 0.32, 0.78, 0.97, 1.0 and 2.2 mg/kg. The Meeting estimated a maximum residue level of 5 mg/kg, a STMR of 0.875 mg/kg and a HR of 2.2 mg/kg for flubendiamide in head lettuce.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 5.8 mg/kg.

Lettuce, Leaf

In six Australian leaf lettuce trials conducted in 2006 according to GAP, residues of flubendiamide at a 1 day PHI were 0.95, 1.6 (2), 1.8, 2.7 and 4.0 mg/kg.

The Meeting estimated a maximum residue level of 7 mg/kg, a STMR of 1.7 mg/kg and a HR of 4 mg/kg for flubendiamide in leaf lettuce.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 6.0 mg/kg.

Spinach

Five trials were conducted in the USA using 5 × 0.05 kg ai/ha (GAP for leafy vegetables allow up to three applications, with a 3 day RTI). Residues at 1 day PHI ranged from 3.1 to 6.7 mg/kg.

As no residue trials were conducted according to GAP, and no residue decline data were available to indicate the rate of residue dissipation, the Meeting could not estimate a maximum residue level for flubendiamide in spinach.

Legume vegetables

Beans with pods

Flubendiamide is registered in Australia in legume vegetables at a maximum rate of 3 × 0.072 kg ai/ha and 1 day PHI. Residues at 1 day PHI in four trials conducted on 2006/2007 according to GAP were 0.11, 0.20 (2) and 0.22 mg/kg in green beans.

Trials were conducted with beans and peas in the USA according to the legume vegetable GAP (2 × 0.1 kg ai/ha). Residues at 1 day PHI in six USA trials were 0.03, 0.07, 0.09 (2), 0.14, and 0.17 mg/kg in beans with pods.

Peas with pods

Residues at 1 day PHI in five trials conducted in Australia according to GAP were 0.38, 0.39, 0.43, 0.45, and 0.90 mg/kg in peas with pods.

Residues at 1 day PHI in three trials conducted in the USA according to GAP were 0.14, 0.22, and 0.21 mg/kg in peas with pods.

Succulent shelled beans and peas

Soya bean (immature seeds)

Twenty trials were conducted on soya beans in the USA according to GAP of 2 × 0.10 kg ai/ha. Residues in green seeds at 1 day PHI were 0.02, 0.03 (2), 0.04 (4), 0.05, 0.07, 0.08 (2), 0.09, 0.10, 0.12, 0.20 (2), 0.21, 0.22, 0.29 and 0.40 mg/kg;

Beans and peas, shelled

Twelve trials were conducted in the USA according to the USA legume vegetable GAP on shelled beans (six trials) and shelled peas (six trials). Residues in shelled beans were < 0.01 (4), 0.01 and 0.03 mg/kg and in shelled peas < 0.01 (4), 0.01 and 0.03 mg/kg. Residues in shelled beans and peas can be combined as < 0.01 (8), 0.01 (2) and 0.03 (2) mg/kg.

The Meeting decided it was appropriate to make a commodity group recommendation for legume vegetables. The results from the peas with pods trials from Australia were used to make the estimations. The Meeting estimated a maximum residue level of 2 mg/kg, and a STMR of 0.43 mg/kg, and a HR of 0.90 mg/kg for flubendiamide in legume vegetables.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 1.2 mg/kg.

Pulses

Soya beans, dry

In dry soya bean seeds, residues at 14 days PHI were < 0.01, 0.01 (4), 0.02 (2), 0.03 (5), 0.04, 0.06, 0.07 (2), 0.09, 0.14, 0.25 and 0.30 mg/kg.

Dry peas and cowpeas

Fourteen trials were conducted with cowpeas and dry peas in the USA according to GAP of 2 × 0.1 kg ai/ha and 14 days PHI. Residues in cowpeas were < 0.01, 0.01 (2), 0.02, 0.04 (3), 0.06 and 0.20 mg/kg. Residues in dry peas were 0.08, 0.11, 0.18 (2) and 0.59 mg/kg.

Based on the data set for dry peas, the Meeting recommended establishing a group MRL for pulses. The Meeting estimated a maximum residue level of 1 mg/kg and a STMR of 0.18 mg/kg for flubendiamide in pulses.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 1.0 mg/kg.

Celery

Flubendiamide is registered in the USA in leafy vegetables at a maximum rate of 3 × 0.05 kg ai/ha and 1 day PHI. In six US trials conducted using five applications at the GAP rate with a 3 day RTI, residues at a 1 day PHI in celery stalks were: 0.81, 1.2, 1.3, 2.1, 2.3, and 2.6 mg/kg.

Although the number of spray applications (five) exceeded that specified by GAP (three), a residue decline study shows substantial reductions in residue levels over three days, the RTI for use in celery. The Meeting concluded that the first two sprays are unlikely to contribute more than 20% to the residue levels at harvest. Consequently, the celery results may be used to estimate maximum residue levels. The Meeting estimated a maximum residue level of 5 mg/kg a STMR of 1.7 mg/kg, and a HR of 2.6 mg/kg for flubendiamide in celery.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 4.6 mg/kg.

Corn (maize)

Flubendiamide is registered in the USA in field corn at a maximum rate of 4 × 0.10 kg ai/ha and 28 day PHI. Nineteen trials were conducted in Canada and the USA according to this GAP giving residues within 28 days PHI of < 0.01 (17) and 0.01 (2) mg/kg. The Meeting estimated a maximum residue level of 0.02 mg/kg and a STMR of 0.01 mg/kg for flubendiamide in maize grain.

Rice

Flubendiamide is registered in India in rice with a GAP of 3 × 0.024 kg ai/ha with a PHI of 40 days. Ten trials were conducted in Thailand and two in India using the GAP rate, but at a PHI of 30 days or less. Nine trials from Thailand conducted with PHIs of 27–30 days gave residues from < 0.01 to 0.11 mg/kg. Two trials conducted in India with 28 day PHIs gave residues of 0.06 and 0.20 mg/kg. One Thai trial with a 13 day PHI gave residues of 0.30 mg/kg.

As no residue trials were conducted according to GAP, the Meeting could not estimate a maximum residue level for flubendiamide in rice.

Tree nuts

Flubendiamide is registered in the USA in tree nuts at 3 × 0.14 kg ai/ha and 14 days PHI. Twenty trials were conducted in the country in almonds and pecans according to GAP. Residues in almonds were < 0.01 (4), 0.01, 0.02 (3), 0.04 and 0.05 mg/kg. Residues in pecans were < 0.01 (6), 0.01 (2), 0.02, and 0.03 mg/kg.

Based on the almond data, the Meeting estimated a maximum residue level of 0.1 mg/kg, a STMR of 0.015 mg/kg and a HR of 0.05 mg/kg for flubendiamide in tree nuts.

Cotton

Flubendiamide is registered in the USA in cotton with a GAP of 3 × 0.10 kg ai/ha with a PHI of 28 days. Residue levels found from 12 trials conducted according to GAP, were: < 0.01, 0.02, 0.03, 0.11, 0.12 (2), 0.18, 0.19, 0.25, 0.28, 0.37 and 1.0 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg and a STMR of 0.15 mg/kg for flubendiamide in cotton seed.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 0.91 mg/kg.

Tea

Flubendiamide is registered in Japan in dry tea at 1 × 0.40 kg ai/ha and 7 days PHI. Six trials were conducted in the country according to GAP, giving residues at 7 days PHI of 11, 17, 22, 24, 28 and 29 mg/kg.

The Meeting estimated a maximum residue level of 50 mg/kg, a STMR of 23 mg/kg and a HR of 29 mg/kg for flubendiamide in tea.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 49 mg/kg.

Animal feeds

The individual residue values that are reported in this section have not been adjusted for dry matter content. However, maximum residue levels were corrected for dry matter content as listed in the OECD feed tables.

Soya bean forage and hay

Twenty trials were conducted on soya bean forage and hay in the USA according to the GAP of that country (2 × 0.10 kg ai/ha). Residues in forage at a 3 day PHI were: 4.3, 6.0, 6.1, 6.7, 6.8, 7.1, 7.2, 7.6, 7.7, 7.9, 8.0, 9.1, 9.9, 10 (3), 11 (2), 13 and 15 mg/kg.

The Meeting estimated a STMR of 7.95 mg/kg and a highest residue of 15 mg/kg for flubendiamide in soya bean forage (green).

In hay, residues at 3 days PHI were 12, 13, 14, 15, 17, 22, 23, 24, 25, 26, 29 (2), 30, 32, 33, 34, 35, 39 and 41 (2) mg/kg.

The Meeting estimated an MRL of 60 mg/kg, a STMR of 27.5 mg/kg and a highest residue of 41 mg/kg for flubendiamide in soya bean fodder.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 62 mg/kg.

Cowpea and pea forage and hay

Twenty two trials were conducted on forage and hay from cowpeas and vines and hay from peas in the USA according to GAP.

Residues at 3 days PHI from six trials in cowpea forage were: 3.9, 4.2, 5.5, 6.6, 9.0 and 14 mg/kg.

The Meeting estimated a STMR of 6.05 mg/kg and a highest residue of 14 mg/kg for flubendiamide in cowpea forage.

Residues in pea vines at 3 days PHI from five trials were: 2.4 (2), 3.1, 3.6 and 5.5 mg/kg. The Meeting estimated a STMR of 3.1 mg/kg and a highest residue of 5.5 mg/kg for flubendiamide in pea vines.

Residues from six trials in cowpea hay at 3 days PHI were: 8.3, 15, 16, 25 and 26 mg/kg. Residues from five trials in pea hay at 3 days PHI were: 4.2, 9.1, 9.9, 12 and 20 mg/kg.

The Meeting decided that residues from trials conducted on cowpeas and pea hay belonged to the same population and could be combined for mutual support as: 4.2, 8.3, 9.1, 9.9, 12, 15, 16, 20, 25 and 26 mg/kg

The Meeting therefore, estimated an MRL of 40 mg/kg, a STMR of 13.5 mg/kg and a highest residue of 26 for flubendiamide in pea fodder.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 47 mg/kg.

Maize forage

In 31 trials conducted on field corn in Canada and the USA according to US GAP, residues in maize forage at 1 day PHI were 1.0, 1.7, 1.8 (2), 2.0, 2.2, 2.5, 3.4, 3.5, 3.6 (3), 3.7 (2), 3.8 (3), 3.9 (3), 4.2, 4.6 (2), 4.8, 5.0, 5.3, 5.5, 5.6 (2), 6.7 and 8.4, mg/kg. The Meeting estimated a STMR of 3.8 mg/kg and a highest residue of 8.4 mg/kg for flubendiamide in maize forage.

Almond hulls

In ten trials conducted on almonds in the USA according to GAP, residues in almond hulls at 14 days PHI were 0.98, 1.4, 1.4, 2.1, 2.4, 2.5, 2.9, 3.3, 4.7 and 5.2 mg/kg.

The Meeting estimated a maximum residue level of 10 mg/kg, and a STMR of 2.45 mg/kg for flubendiamide in almond hulls.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 8.3 mg/kg]

Cotton gin trash

In six trials conducted in the USA according to GAP, residues in cotton gin trash at a 28 day PHI were 2.3, 3.5, 6.8, 8.1 and 25 (2) mg/kg.

The Meeting estimated a STMR of 7.45 mg/kg for flubendiamide in cotton gin trash.

Processing studies

Effects on the nature of residues

[Phthalic-acid ring-UL-^{14}C]flubendiamide (0.2 mg ai/L water containing 1% acetonitrile) was incubated in buffered drinking water at three representative sets of conditions: pasteurization at 90 °C/20 min at pH 4; baking, brewing, boiling at 100 °C/60 min at pH 5; sterilization (autoclave) at 120 °C/20 min at pH 6. Radioactivity was determined by LSC and HPLC/MS for confirmation of the identity of the test compound. Radioactivity balances were in a range of 99.8 to 101.0% of applied radioactivity. In all three processing scenarios, no degradates were observed in any of the samples, and flubendiamide was the only compound in all HPLC profiles.

Fate of residues in processing

Processing studies were conducted on apples, peaches, plums, grapes, tomatoes, cucurbits (cucumbers, melons and summer squash), cabbages, broccoli, lettuce, cotton, soya bean, corn and rice. In all studies, residues of flubendiamide and its metabolite flubendiamide-des-iodo were determined by HPLC-MS/MS. Processing factors are only shown for flubendiamide.

A summary of processing factors (PF) calculated based on the data provided is shown on Table 99. Based on the estimations made on the crops, a STMR-P was estimated by multiplying the STMR of the raw commodity for the PF. As no recommendations were made for rice, no further estimations were made for processing commodities of this crop. Maximum residue levels (MRLs) were only estimated for commodities with a Codex code and of importance to international trading.

Table 99 Summary of processing factors and estimations for processing commodities

Commodity	STMR, mg/kg	HR, mg/kg	PF, mean or best estimate	STMR-P, mg/kg
Pome fruit	0.25	0.59		
Dried fruit			0.51	0.13
Apple juice			0.06	0.015
Plum	0.585	1.0		
Prunes			0.9	0.53
Grape	0.415	0.81		
Grape juice			0.13	0.054
Wine			0.19	0.079
Raisin			1.7	0.70
Grape pomace, dry			5.9	2.45
Brassica vegetables	0.365	2.7		
Tomato	0.35	0.63		
Tomato peeled			0.3	0.105
Tomato juice			0.49	0.17
Tomato preserve/canned			0.29	0.10
Tomato paste			4	1.4
Lettuce	0.875	2.2		
Soya bean	0.08	0.40		
Refined oil			< 0.04	0.001
Aspirated grain fraction			358	28.6
Soya bean meal			0.12	0.01
Soya bean hulls			2.7	0.22
Corn (maize)	0.01	0.01		
Corn flour			2.1	0.021
Corn meal			0.93	0.009
Corn oil, refined			0.45	0.0045
Corn aspirated grain fractions			318	3.18
Cotton	0.15	1.0		
Cotton oil crude			6.1	0.92
Cotton meal			0.22	0.08

Residues in the dried commodity were lower than in fresh grapes: as a consequence the meeting decided a maximum residue level need not be recommended.

The Meeting decided to estimate a maximum residue level of 0.05 mg/kg for corn flour based on a mean residue of 0.01 mg/kg for maize and a processing factor of 2.1 (0.01 × 2.1 = 0.021 mg/kg).

Residues in animal commodities

Farm animal dietary burden

The Meeting estimated the dietary burden of flubendiamide in farm animals on the basis of the diets listed in Appendix IV of the 2009 Manual on the Submission and Evaluation of Pesticide Residues Data and the STMR or highest residue levels estimated at the present Meeting. Dietary burden calculations are provided in Annex 5.

Table 100 Animal dietary burden for flubendiamide, ppm of dry matter diet

		US/CAN	EU	Australia	Japan
Beef cattle	max	4.9	32.1	47.9[a]	0.039
	mean	3.1	14.7	29.9[b]	0.039
Dairy cattle	max	25.0	32.6	47.3[c]	10.5
	mean	13.7	15.0	25.0[d]	4.78
Poultry broiler	max	0.07	0.09	0.19	0.008
	mean	0.07	0.09	0.19	0.008
Poultry layer	max	0.07	9.6[e]	0.19	0.009
	mean	0.07	5.3[f]	0.19	0.009

[a] Highest maximum beef or dairy cattle dietary burden suitable for maximum residue level estimates for mammalian meat

[b] Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian meat.

[c] Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

[d] Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

[e] Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs.

[f] Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs.

The flubendiamide burdens for animal commodity MRL estimation (residue levels in animal feeds expressed on dry weight) reached a maximum of 47.3 ppm for cattle and of 9.6 ppm for poultry. The flubendiamide dietary burdens for animal commodity STMR estimation (residue levels in animal feeds expressed on dry weight) reached a maximum of 29.9 ppm for cattle and of 5.3 ppm for poultry.

Animal feeding studies

Laying hens were fed for 28 consecutive days with feed containing flubendiamide at 0.02, 0.10, or 0.50 ppm. Eggs were collected daily. Additionally, two groups of laying hens were fed at 0.5 ppm feed for 28 consecutive days in order to investigate the depuration of flubendiamide and its metabolite in eggs and tissues (up to 14 days after the last dose). Samples were analysed by HPLC-MS/MS for flubendiamide and flubendiamide-iodophthalimide. Flubendiamide was detected in eggs at the second dosing level from day 13 (0.01 mg/kg) and it reached 0.06 mg/kg at the highest dose. No residues of flubendiamide-iodophthalimide were detected in any egg sample at any dose. The highest flubendiamide residues in tissues were observed in fat, showing evidence of a dose response (0.01, 0.07 and 0.25 mg/kg in the first, second and third dose, respectively). Flubendiamide-iodophthalimide was found only at the highest dose level in fat (0.02 mg/kg). Flubendiamide was not present in egg samples 14 days after the last dose and decreased in fat from 0.27 mg/kg at the end of the dosing period to 0.04 mg/kg 7 days after the last dose to 0.01 mg/kg after 14 days (only one fat sample).

Lactating cows were dosed orally for 29 consecutive days with flubendiamide at 2.5, 7.5, 30 or 50 mg/kg feed/day (nominal dosing rates). Milk was collected twice daily during the dosing period, and a portion of the 25 day sample from the highest dose group was separated into milk fat and skim milk whey. Additionally, two cows were fed at 50 mg/kg for 29 consecutive days in order to investigate the depuration of residues in milk (up to 21 days after the last dose) and tissues (at 7 and

21 days after the last dose). Tissue and milk samples were analysed for residues by HPLC-MS/MS for flubendiamide and flubendiamide-iodophthalimide. For the low and medium dose levels, residues of flubendiamide in milk remained very low throughout the dosing periods at the low and medium dose levels (up to 0.03 mg/kg). At higher dose levels (30 and 50 mg/kg), residues in milk reached a plateau level after 7–8 days of dosing (approximately 0.11 mg/kg). Flubendiamide residues were 0.02 mg/kg in milk whey and 1.5 mg/kg in “milk fat (cream)”, with an apparent processing factor for milk to milk fat of 13.6. However, no information on the lipid content of the milk fat (cream) sample was provided. The iodophthalimide metabolite was only detected in milk fat (0.23 mg/kg). Residues of flubendiamide were observed in tissues of all animals in all dose groups, with the lowest levels in muscle (0.01 to 0.12 mg/kg), followed by liver and kidney (0.04 to 0.46 mg/kg) and fat (from 0.06 to 0.65 mg/kg in subcutaneous fat, 0.08 to 1.0 mg/kg in omental and perirenal fat). Flubendiamide was detected in fat at a mean level up to 0.17 mg/kg. During the depuration phase, residues in milk decline from 0.16 mg/kg in the last dosing day to 0.02 mg/kg after 19 to 21 days. Residues in tissues declined to 31 to 46% of the last dosing day level in the first week of depuration and from 23 to 32% after 14 days.

Animal commodity maximum residue levels

Poultry

The dietary burdens for the estimation of maximum residue levels and STMR values for flubendiamide in poultry commodities are 9.6 and 5.3 ppm, respectively. Because the poultry dietary burden exceeds the highest dosing level in the poultry feeding study by more than 30%, no attempt was made to estimate maximum residue levels, STMR or HR values for poultry tissues and eggs.

Dosing levels in the bovine feeding study are adequate for the purposes of estimating residue levels in mammals, and the relevant data are summarised in Table 101.

Table 101 Estimations of residues in mammalian commodities

Dietary burden (mg/kg) Feeding level [ppm]		Flubendiamide and flubendiamide-iodophthalimide residues, mg/kg					
		Milk	Milk fat	Muscle	Liver	Kidney	Fat
MRL cattle beef, highest residue	(47.9) [38] [60]			(0.13) [0.09] [0.16]	(0.56) [0.53] [0.59]	(0.57) [0.55] [0.59]	(1.2) [0.93] [1.47]
MRL milk, highest residue	(47.3) [69]	(0.16) [0.17]	(4.0) [4.25][a]				
STMR cattle beef and dairy, mean residue	(29.9) [38]			(0.06) [0.07]	(0.31) [0.39]	(0.32) [0.41]	(0.62) [0.79]
STMR milk, mean residue	(25.0) [38]	(0.066) [0.10]	(1.6) [2.50][a]				

[a] Although a residue concentration factor was provided for “milk fat (cream)”, no lipid content was provided for this sample. Assuming that whole milk is 4% milk fat, and assuming all flubendiamide and flubendiamide-iodophthalimide residues partition into the fat, a milk fat residue was estimated by applying the maximum concentration factor for milk to milk fat of 25×.

The data from the cattle feeding studies were used to support the estimation of maximum residue levels for flubendiamide in mammalian meat, edible offal, and milk.

The Meeting estimated STMR values of 0.06 mg/kg for mammalian muscle and 0.62 mg/kg for mammalian fat, and a maximum residue level of 2 mg/kg for mammalian meat. The HRs were 0.13 mg/kg and 1.2 mg/kg for muscle and fat, respectively.

The Meeting estimated an STMR value of 0.32 mg/kg and a maximum residue level of 1 mg/kg for mammalian edible offal, based on liver and kidney data. The HR was 0.57 mg/kg.

The Meeting estimated an STMR value of 0.066 mg/kg and a maximum residue level of 0.2 mg/kg for flubendiamide in milks.

The Meeting estimated an STMR value of 1.6 mg/kg and a HR of 4.0 mg/kg for milk fat. The Meeting estimated a maximum residue level of 5 mg/kg for milk fat.

RECOMMENDATIONS

Definition of the residue (for compliance with the MRL and for estimation of dietary intake) for animal and plant commodities: *flubendiamide*

The residue is fat soluble.

CCN	Commodity name	Recommended maximum residue level, mg/kg	STMR (P) mg/kg	HR (P) mg/kg
AM 0660	Almond hulls	10	2.45	
VB 0040	Brassica (cole or cabbage) vegetables, Head cabbages, Flowerhead brassicas	4	0.365	2.7
VS 0624	Celery	5	1.7	2.6
SO 0691	Cotton seed	1.5	0.15	
VC 0045	Fruiting vegetables, Cucurbits	0.2	0.045	0.09
MO 0105	Edible offal (Mammalian)	1	0.32	0.57
FB 0269	Grapes	2	0.42	0.81
VP 0060	Legume vegetables	2	0.43	0.90
VL 0482	Lettuce, Head	5	0.875	2.2
VL 0483	Lettuce, leaf	7	1.7	4.0
GC 0645	Maize	0.02	0.01	
CF 1255	Maize flour	0.05	0.021	
MM 0095	Meat (from mammals other than marine mammals) (fat)	2 (fat)	0.06 muscle 0.62 fat	0.13 muscle 1.2 fat
ML 0106	Milks	0.1	0.066	
FM 0183	Milk fats	5	1.6	4.0
AL 0072	Pea hay or Pea fodder (dry)	40	13.5	26
VO 0051	Peppers	0.7	0.09	0.37
HS 0444	Peppers, Chili, dried	7	0.9	
FP 0009	Pome fruits	0.8	0.25	0.59
VD 0070	Pulses	1	0.18	
AL 0541	Soya bean fodder	60	27.5	41
FS 0012	Stone fruit	2	0.585	1.0
VO 0447	Sweet corn (corn-on-the-cob)	0.02	0.01	0.01
DT 1114	Tea, Green, Black (black, fermented and dried)	50	23	29
VO 0448	Tomato	2	0.35	0.63
TN 0085	Tree Nuts	0.1	0.015	0.05

DIETARY RISK ASSESSMENT

Long-term intake

The ADI for flubendiamide is 0–0.02 mg/kg bw. The International Estimated Daily Intakes (IEDI) for flubendiamide was estimated for the 13 GEMS/Food cluster diets using the STMR or STMR-P values estimated by the current Meeting. The results are shown in Annex 3 of the 2010 Report of the JMPR. The IEDI ranged from 3 to 20% of the maximum ADI. The Meeting concluded that the long-term intake of residues of flubendiamide from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The ARfD for flubendiamide is 0.2 mg/kg bw. The International Estimated Short Term Intake (IESTI) for flubendiamide was calculated for the plant commodities for which STMRs and HRs were estimated and for which consumption data were available. The results are shown in Annex 4 of the 2010 Report of the JMPR. The IESTI ranged from 0 to 40% of the ARfD for the general population and from 0 to 60% of the ARfD for children.

The Meeting concluded that the short-term intake of residues from the uses of flubendiamide considered by the Meeting is unlikely to present a public health concern.

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