FLONICAMID (282)

The first draft was prepared by Ms Monique Thomas, Pest Management Regulatory Agency, Canada

EXPLANATION

Flonicamid is a new insecticide for control of aphids and other sucking insects. It belongs to a new class of chemistry known as pyridinecarboxamide. Flonicamid has been registered in Canada since 2009. At the 46th Session of the CCPR (2014), flonicamid was scheduled for evaluation as a new compound by 2015 JMPR.

The Meeting received information on the metabolism of flonicamid in peaches, Bell peppers, potatoes, wheat, lactating goats, laying hens and rotational crops, environmental fate, methods of residue analysis, freezer storage stability, GAP, supervised residue trials on various fruits, vegetables, tree nuts, oil seeds, dried hops, mint and tea, processing studies, as well as livestock feeding studies.

IDENTITY

ISO common name: Flonicamid

Chemical name:

IUPAC: *N*-cyanomethyl-4-(trifluoromethyl)nicotinamide

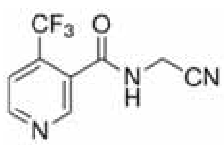
CAS: *N*-(cyanomethyl)-4-(trifluoromethyl)-3-pyridinecarboxamide

CAS Registry. No.: 158062-67-0

CIPAC No.: 763

Trade Name: IKI-220

Structural formula:



Molecular formula: $C_9H_6F_3N_3O$

Molecular weight: 229.16 g/mol

Physical and chemical properties

Property	Findings		Report, Reference
Pure Active Ingredient			
Melting Point	157.5 ºC		010153-1
Mean Relative Density (20 ºC)	1.54 g/mL		
Physical State, colour	solid powder, off white		
Odour	odourless		
Vapour Pressure	Temperature, ºC	Pa	010341-1
	20	9.43×10^{-7} (extrapolated)	

Property	Findings		Report, Reference
	25	2.55×10^{-6} (extrapolated)	
	30	6.48×10^{-6} (experimental)	
	40.1	4.40×10^{-5} (experimental)	
	50.1	2.31×10^{-4} (experimental)	
Solubility in water	5.2 g/L at 20 ºC		010251-1
Solubility in organic solvents (20 ºC)	Solvent	g/L	0.10250-1
	Acetone	163.5	
	Ethyl Acetate	34.2	
	Methanol	104.3	
	Dichloromethane	4.5	
	Toluene	0.55	
	Hexane	0.0002	
	n-Octanol	3	
	Acetonitrile	132.8	
	Isopropyl Alcohol	18.7	
Partition coefficient	1.9 (Log P_{ow} = 0.3) at 29.8 ºC		010252-1
Hydrolysis rate at 25 ºC		DT_{50} (days)	008076-2
	pH 5	no hydrolysis	
	pH 7	no hydrolysis	
	pH 9	204 (max. 31% TFNG-AM)	
at 50 ºC	pH 4	no hydrolysis	
	pH 5	no hydrolysis	
	pH 7	578 (no major degradation products)	
	pH 9	9 (max. 65% TFNG-AM, max. 86% TFNG)	
at 40 ºC	pH 9	17 (max. 63% TFNG-AM, max. 26% TFNG)	
Flonicamid is stable at pH 4 and pH 5. The amide TFNG-AM is formed from this reaction (under alkaline conditions) and can then be hydrolyzed to TFNG.			
Quantum yield	DT_{50} at pH 7 and 23 ºC was 267 days. flonicamid did not degrade in dark controls. The major degradate TFNA-AM, only degraded slightly (from 2.4% at time 0 and increased slightly to 2.9% by Day 15)		011050-1
Dissociation constant	11.60 ± 0.03 in 5% ethanol/water at 20 ± 1 ºC		010141-1
Flammability	Not flammable		20334
Auto-flammability	No relative self-ignition temperature		
Explosive properties	Flonicamid is not a potential explosive and does not have a potential for rapid energy release (decomposition energy = 374 J/g)		
Technical material			
Physical State, colour	Solid powder, light beige		012575-1
pH	4.5 at 25 ºC		
Mean Relative Density (20 ºC)	1.531 g/mL		
Odour	Odourless		
Solubility in organic solvents (20 ºC)	Solvent	g/L	011201-1
	Acetone	157.1	
	Ethyl Acetate	34.9	
	Methanol	89	
	Dichloromethane	4	
	Toluene	0.3	
	Hexane	0.0003	
	n-Octanol	2.6	
	Acetonitrile	111.4	
	Isopropyl Alcohol	14.7	

Formulation

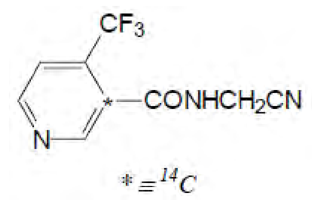
Flonicamid is commercially marketed as a soluble or wettable granule containing 50% flonicamid.

Specification

Flonicamid has not been evaluated by the FAO/WHO Joint Meeting of Pesticide Specifications (JMPS).

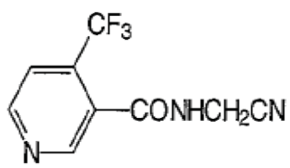
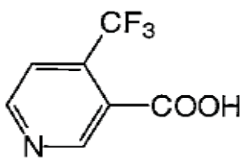
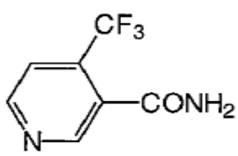
METABOLISM AND ENVIRONMENTAL FATE

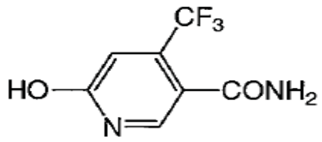
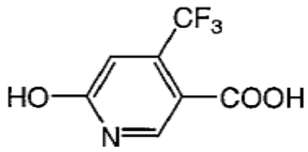
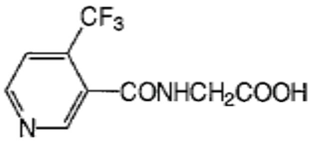
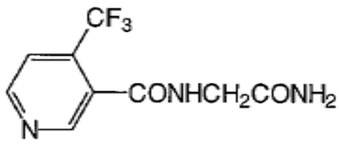
The metabolism and distribution of flonicamid in plants and animals was investigated using ^{14}C-labelled test material as shown below:



Chemical names, structures and code names of metabolites and degradation products of flonicamid are summarized in the following table. Compounds are referred to primarily by the code name.

Code names, chemical names and structures of flonicamid related substances

Code Name	Structure	Chemical Name	Occurrence
Flonicamid IKI-220		*N*-cyanomethyl-4-(trifluoromethyl)nicotinamide	Goat Hen Peach Peppers Potato Wheat Rotational crop (wheat) Soil
TFNA		4-trifluoromethylnicotinic acid	Goat Hen Peach Peppers Potato Wheat Rotational crop (wheat) Soil
TFNA-AM		4-trifluoromethylnicotinamide	Goat Hen Peach Peppers Potato Wheat Rotational crop (wheat)

Code Name	Structure	Chemical Name	Occurrence
OH-TFNA-AM		6-hydroxy-4-trifluoromethylnicotinamide	Goat Hen
TFNA-OH		6-hydroxy-4-trifluoromethylnicotinic acid	Rotational crop (wheat)
TFNG		*N*-(4-trifluoromethylnicotinoyl)glycine	Peach Peppers Potato Wheat Rotational crop (wheat) Soil
TFNG-AM		*N*-(4-trifluoromethylnicotinoyl)glycinamide	Hen Peach Peppers Wheat Rotational crop (wheat)

Plant metabolism

The Meeting received information on the fate of flonicamid radio-labelled at the 3 position of the pyridine ring following foliar application to peaches, Bell peppers, potatoes and wheat (immature and mature).

Peach

Flonicamid, radio-labelled at the 3 position of the pyridine ring (specific activity: 1.67 MBq/mg), formulated as a wettable granule formulation, was applied to single peach trees (variety: Elberta), grown outdoors in individual 1.4 m^2 plots of clay loam. Each tree received two foliar applications, at a 14-day re-treatment interval, at rates of 100 g ai/ha (low rate) or 500 g ai/ha (high rate) per application, resulting in total seasonal rates of 200 g ai/ha or 1000 g ai/ha. Mature fruits and leaves were harvested 21 days following the second application.

To remove surface residues the fruit was washed with deionised water. Following the removal of pits, the surface-washed fruits were then cut into small pieces and homogenised. The homogenates were then centrifuged to give an aqueous fraction (juice) and a solid fraction (pomace). The juice was decanted, total volume measured and the radioactivity measured. The remaining pomace was weighed and then ground with dry ice in a blender.

Radioactivity was measured by liquid scintillation counting (LSC). Dry-ice-ground peach leaves, pomace and PES were combusted in an oxidizer (Table 1).

Overall total radioactive residues (TRRs) in fruits at the low rate and the high rate were 0.10 mg eq/kg and 0.32 mg eq/kg, respectively, while in the leaves, TRRs were higher than those of fruits, 6.24 mg eq/kg at the low rate and 24.21 mg eq/kg at the high rate.

Table 1 TRRs in peach fruits and leaves

Crop part	TRRs (mg eq/kg)	
	Low rate (200 g ai/ha)	High rate (1000 g ai/ha)
Fruits	0.10	0.32
Leaves	6.24	24.21

Subsamples of both pomace and leaves were extracted twice with acetonitrile:water:phosphoric acid (40:60:0.1, v/v/v). The extracts of each subsample were pooled and the PES were air dried. Quantification and characterization/identification of residues were done by HPLC. To determine the ^{14}C residue profiles, fractions were collected from the HPLC effluent and analysed by LSC. Flonicamid and metabolites were isolated and purified from peach juice and leaf extracts of both treatment groups. The isolated radioactive components were purified by reverse phase HPLC. The identification was supported by other methods such as TLC and LC-MS.

Table 2 Distribution of TRRs in mature fruit harvested 21 days following application at low rate and high rate

Fraction	Low Rate (200 g ai/ha)		High Rate (1000 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Surface wash	0.006	5.6	0.05	15.3
Flonicamid	0.006	2.7	0.03	8.6
TFNG	0.000	0.2	0.001	0.3
TFNA	0.000	0.3	0.001	0.3
TFNG-AM	0.000	0.2	0.003	0.9
TFNA-AM	0.000	0.2	0.003	0.8
Unknowns	0.000	0.4	0.006	1.9
Polar	0.000	0.2	0.001	0.3
Nonpolar	0.001	1.0	0.007	2.0
Diffuse Radioactivity	0.001	0.6	0.001	0.2
Juice (aqueous fraction)	0.073	73.2	0.205	63.7
Extracted	0.073	73.2	0.205	63.7
Flonicamid	0.020	20.3	0.13	40.2
TFNG	0.005	5.0	0.01	3.0
TFNA	0.040	39.9	0.04	12.9
TFNG-AM	0.001	1.2	0.006	1.8
TFNA-AM	0.001	1.1	0.005	1.6
Unknowns	0.003	3.2	0.007	2.1
Polar	0.002	2.0	0.005	1.5
Nonpolar	–	–	0.001	0.3
Diffuse Radioactivity	0.001	0.6	0.000	0.1
Pomace (solid fraction)	0.021	21.1	0.067	21.0
Extracted	0.019	19.5	0.06	19.1
Flonicamid	0.007	7.1	0.04	11.8
TFNG	0.001	0.8	0.003	1.0
TFNA	0.009	9.0	0.14	4.2
TFNG-AM	0.000	0.3	0.001	0.4
TFNA-AM	0.000	0.3	0.001	0.4
Unknowns	0.001	1.4	0.003	0.9
Polar	0.000	0.4	0.000	0.1
Nonpolar	0.000	0.1	0.000	0.1
Diffuse Radioactivity	0.000	0.1	0.000	0.0
Nonextracted	0.002	1.6	0.006	1.9
Total	0.10	100.0	0.322	100.0

Table 3 Identification/Characterization of TRRs in whole fruit harvested 21 days following application at low rate and high rate

Analyte	Low Rate (200 g ai/ha)		High Rate (1000 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Flonicamid	0.033	30.1	0.20	60.6
TFNG	0.006	6.0	0.14	4.3
TFNA	0.049	49.2	0.55	17.4
TFNG-AM	0.001	1.7	0.01	3.1
TFNA-AM	0.001	1.6	0.009	2.8

Analyte	Low Rate (200 g ai/ha)		High Rate (1000 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Total Identified	0.09	88.6	0.91	88.2
Total Unidentfied [a]	0.006	6.3	0.017	5.2
Total Characterized [b]	0.003	3.7	0.014	4.3
Total Extracted [c]	0.098	98.3	0.315	98.1
Total Nonextracted	0.002	1.6	0.006	1.9
Total	0.10	100.0	0.322	100.0

[a] Total unidentified = Unknowns + Diffuse Radioactivity

[b] Total Characterized = Polar + Nonpolar

[c] Total extracted = Surface wash + Juice extracted + Pomace extracted

Table 4 Distribution of TRRs in mature leaves harvested 21 days following application at low rate and high rate

	Low Rate (200 g ai/ha)		High Rate (1000 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	5.58	89.2	22.27	92.0
Flonicamid	2.05	32.9	15.72	64.9
TFNG	1.21	19.3	2.06	8.5
TFNA	0.99	15.8	1.28	5.3
TFNG-AM	0.21	3.4	0.40	1.6
TFNA-AM	0.25	4.1	0.48	2.0
Unknowns	0.18	2.8	0.18	0.7
Polar	0.39	6.3	0.92	3.8
Nonpolar	0.29	4.6	1.24	5.1
Diffuse Radioactivity	0.001	0.0	0.002	0.0
Unextracted	0.67	10.8	1.94	8.0
Total	6.25	100.0	24.21	100.0

Representative samples of peach fruit fractions (surface wash, pomace and juice) were extracted and analysed immediately after collection and re-analysed after 5 months of storage. Considering the metabolite profiles from the initial and final analyses were very similar, flonicamid and the metabolites were stable during this storage interval.

According to Table 2, the surface wash removed very little radioactivity, 6–15%, demonstrating limited penetration. The majority of the radioactivity in peach fruits was partitioned in the juice fraction, accounting for 64–73% of the TRR while the radioactivity in pomace represented 21% of the TRR.

While juice was not further extracted with organic solvents, extraction of the pulp with acetonitrile:water:phosphoric acid recovered 92% TRR. When treated at the low rate, flonicamid (30.1% of the TRR). and TFNA (49.2% of TRR) were the predominant residues. In peaches treated at the high rate, flonicamid accounted for 60.6% of the TRR while TFNA accounted for 17.4% of the TRR. All other metabolites, TFNG, TFNG-AM and TFNA- AM, were ⩽ 6% of the TRR.

In leaves, (Table 4), flonicamid accounted for 33–65% of the TRR followed by the major metabolites TFNG (8–19% of the TRR) and TFNA (5–16% of the TRR). All other metabolites, TFNG-AM and TFNA-AM, were ⩽ 6% of the TRR.

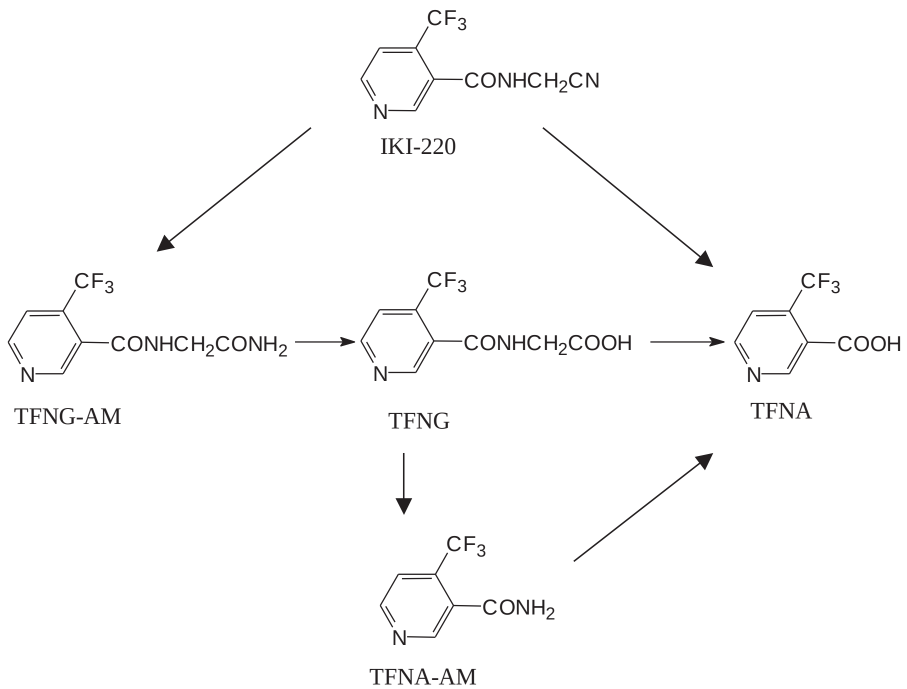


Figure 1 Proposed metabolic pathway in peaches

Bell pepper

Bell pepper plants (variety Wanderbell), grown in individual pots maintained in greenhouses, received a single application of flonicamid, radio-labelled at the 3 position of the pyridine ring (specific activity: 0.182 MBq/mg) and formulated as a 50% wettable granule formulation, at 100 g ai/ha. Bell pepper plants (fruits and leaves) were harvested 7 days and 14 days after application.

Bell pepper fruits and leaves were surface washed with methanol:water (10:90, v/v) prior to being homogenised in a food processor with dry ice. TRRs were determined by combusting triplicate aliquots of the homogenates.

TRRs in leaves decreased from 2.23 mg eq/kg, when harvested 7 days after treatment (DAT) to 1.35 mg eq/kg at 14 DAT. Similarly in fruits, the TRRs decreased insignificantly from 0.17 mg eq/kg (7 DAT) to 0.11 mg eq/kg (14 DAT).

TRRs in each tissue was determined by combusting the samples using an oxidizer. Unextracted residues in post-extraction solids were also determined by combustion.

Table 5 TRRs in bell pepper leaves and fruits

Crop part	TRRs (mg eq/kg)	
	7-DAT	14-DAT
Leaves	2.22	1.35
Fruits	0.17	0.11

Aliquots of fruit and leaf homogenates were extracted twice with methanol:water (50:50) followed by partitioning with hexane and ethyl acetate. The remaining aqueous phase was further separated by open column chromatography. All extracts were analysed by HPLC and TLC.

Table 6 Distribution of radioactivity in bell pepper leaves and fruits

Fraction	7-DAT		14-DAT	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Leaves				
Surface wash	0.81	36.1	0.23	17.3
Extracted	1.35	60.5	1.05	78.2

Fraction	7-DAT		14-DAT	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Hexane	0.004	0.2	0.00	0.0
Ethyl acetate	0.88	39.7	0.46	34.1
Aqueous soluble	0.46	20.6	0.59	44.1
PES	0.08	3.4	0.06	4.3
Total	2.23	100.0	1.35	100.0
Fruits				
Surface wash	0.06	33.6	0.02	18.2
Extracted	0.11	65.6	0.09	80.5
Hexane	0.00	0.0	0.00	0.0
Ethyl acetate	0.10	56.9	0.06	60.8
Aqueous soluble	0.02	8.6	0.02	19.6
PES	0.001	0.8	0.001	1.3
Total	0.17	100.0	0.11	100.0

Table 7 Identification/Characterization of TRRs in mature bell pepper leaves harvested 7 DAT and 14 DAT

	Surface wash		Extracted		Total	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
7 DAT						
Flonicamid	0.81	36.1	0.85	38.2	1.66	74.3
TFNA	n.d.	n.d.	0.04	2.0	0.04	2.0
TFNG	n.d.	n.d.	0.27	12.2	0.27	12.2
TFNA-AM	n.d.	n.d.	0.02	0.7	0.02	0.7
TFNG-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Others	n.d.	n.d.	0.10	4.6	0.10	4.6
Total	0.81	36.1	1.28	57.7	2.09	93.8
14 DAT						
Flonicamid	0.22	16.1	0.42	31.3	0.64	47.4
TFNA	n.d.	n.d.	0.03	2.4	0.03	2.4
TFNG	n.d.	n.d.	0.38	28.2	0.38	28.2
TFNA-AM	n.d.	n.d.	0.02	1.1	0.02	1.1
TFNG-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Others	n.d.	n.d.	0.15	10.8	0.16	12.0
Total	0.24	17.3	0.99	73.7	1.22	91.0

n.d. = Not detected

Others: consists of multiple peaks, each of which accounted for ≤2.1% of the TRR

Table 8 Identification/Characterization of TRRs in mature bell pepper fruits harvested 7 DAT and 14 DAT

	Surface wash		Extracted		Total	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
7 DAT						
Flonicamid	0.06	33.6	0.10	57.8	0.16	91.4
TFNA	n.d.	n.d.	0.001	0.9	0.001	0.9
TFNG	n.d.	n.d.	0.005	2.8	0.005	2.8
TFNA-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
TFNG-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Others	n.d.	n.d.	0.005	2.9	0.005	2.9
Total	0.06	33.6	0.11	64.3	0.17	97.9
14 DAT						
Flonicamid	0.02	17.8	0.06	58.8	0.08	76.6
TFNA	n.d.	n.d.	0.004	3.7	0.004	3.7
TFNG	n.d.	n.d.	0.008	7.8	0.008	7.8
TFNA-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
TFNG-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Others	n.d.	n.d.	0.008	7.1	0.008	7.5

	Surface wash		Extracted		Total	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Total	0.02	18.2	0.08	77.4	0.10	95.6

n.d. = Not detected

Others: consists of multiple peaks, each of which accounted for ≤2.0% of the TRR

No information was provided on the duration of storage of the fruit and leaf samples.

While the %TRR in the surface wash decreased with increasing DAT in the leaves and fruit (36% to 17% of the TRR in leaves and 34% to 18% of the TRR in fruits). The extracted TRRs and those in the PES increased with increasing DAT: 61–78% of the TRR and 3–4% of the TRR in leaves, respectively, and 66–81% of the TRR and 0.8–1% of the TRR in fruits, respectively. This trend demonstrates the translocation of the radioactivity from the surface into the leaves and fruits (Table 6).

Analysis of each of the fractions indicated that flonicamid and TFNG were the predominant residues in leaves and fruits at both harvest intervals. In leaves, the parent accounted for 47–74% of the TRR (0.6–1.7 mg/kg) while TFNG accounted for 12–28% of the TRR (0.3–0.4 mg/kg). Similarly in fruits, flonicamid accounted 77–91% of the TRR (0.08–0.16 mg/kg) while TFNG accounted for 3–8% of the TRR (0.005–0.008 mg/kg). All identified metabolites (TFNA, TFNA-AM and TFNG-AM) were either not detected or were ≤ 12% of the TRR.

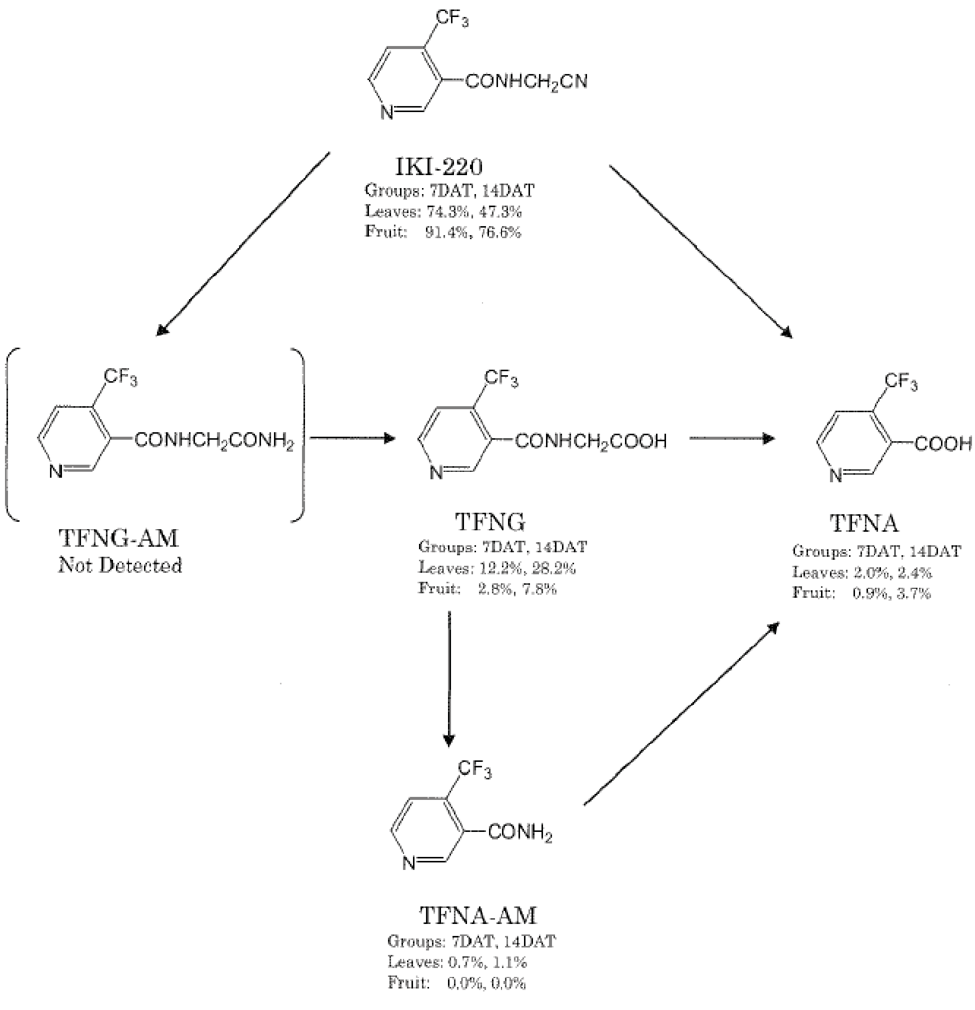


Figure 2 Proposed metabolic pathway in peppers

Potato

Flonicamid, radio-labelled at the 3 position of the pyridine ring (specific activity: 9.08 MBq/mg) and formulated as a 50% wettable powder formulation was applied to potted potato plants (variety Kennebec) maintained outdoors. The plants were treated at either the lower rate of 100 g ai/ha or the higher rate of 500 g ai/ha. Both treatments were repeated after a two-week interval and potato tubers and foliage were harvested 14 days following the second application.

One subsample of potato tubers from each group was washed with ACN:water (80:20, v/v) prior to homogenisation to determine the radioactivity in the surface wash whilst another subsample of potato tubers from each group was homogenised without rinsing the tubers. Potato foliage was processed to isolate the metabolites and to further elucidate the metabolic pathway of flonicamid. Total radioactive residue in each tissue was determined by combusting the samples using an oxidizer. Unextracted residues in PES were also determined by combustion. The radioactivity in the samples was measured by Liquid Scintillation Counting (LSC).

Overall total radioactive residues (TRRs) in unwashed tubers at the low rate and the high rate were 0.11 mg eq/kg and 0.20 mg eq/kg, respectively, whilst those in washed tubers were slightly higher; 0.14 mg eq/kg and 0.53 mg eq/kg. TRRs in mature foliage were higher than those in tubers; 1.53 mg eq/kg at the low rate and 7.67 mg eq/kg at the high rate (Table 9).

Table 9 TRRs in potato tubers and foliage

Crop part	TRRs (mg eq/kg)	
	Low rate (200 g ai/ha)	High rate (1000 g ai/ha)
Unwashed potato tubers	0.11	0.20
Washed potato tubers	0.14	0.53
Foliage	1.53	7.67

Tuber samples were homogenised and consecutively extracted with ACN, ACN:water (80:20, v/v) and twice with ACN:water (50:50, v/v), vortexed, sonicated and centrifuged. The extracts were combined. Foliage samples were homogenised with dry ice and extracted three times with ACN:water:acetic acid (60:40:0.1, v/v/v) and then filtered. The extracts were combined and concentrated.

Metabolites were first identified with HPLC by comparison with reference compounds isolated from repeated HPLC separations of foliage extract of the high treatment group. Most of the isolated metabolites were further purified by normal-phase chromatography. Their identification was supported by other methods such as LC-MS, HPLC on a C8 column and acid hydrolysis (with 3 N HCl).

Table 10 Identification/Characterization of TRRs in unwashed and washed potato tubers following treatment at the low rate and high rate

Fraction	Unwashed Potato Tubers				Washed Potato Tubers			
	Low rate (200 g ai/ha)		High rate (1000 g ai/ha)		Low rate (200 g ai/ha)		High rate (1000 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Surface wash	–	–	–	–	0.0004	0.3	0.002	0.4
Extracted	0.098	92.6	0.18	90.9	0.14	94.6	0.49	92.6
Flonicamid	0.006	5.6	0.04	19.3	0.02	11.5	0.04	7.5
TFNG	0.042	39.3	0.05	25.1	0.05	35.9	0.18	33.6
TFNA	0.036	34.4	0.07	33.7	0.05	31.8	0.21	40.0
TFNA conjugate	0.006	6.0	0.01	4.8	0.01	5.2	0.02	4.8
TFNG-AM	0.001	1.0	0.002	1.2	0.001	1.0	0.01	1.1
TFNA-AM	0.001	1.0	0.003	1.4	0.002	1.2	0.01	1.1
PM-3a	0.0	0.0	0.004	1.8	0.006	3.9	n.d.	n.d.
Others	0.006	5.3	0.007	3.7	0.006	4.2	0.02	4.5
Unextracted	0.008	7.4	0.018	9.1	0.007	5.1	0.04	7.0

Fraction	Unwashed Potato Tubers				Washed Potato Tubers			
	Low rate (200 g ai/ha)		High rate (1000 g ai/ha)		Low rate (200 g ai/ha)		High rate (1000 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Total	0.11	100	0.20	100	0.14	100	0.53	100.0

PM-3a: Conjugate of TFNA-AM

Table 11 Identification/Characterization of TRRs in foliage following treatment at the low rate and high rate

Fraction	Potato Foliage			
	Low rate (200 g ai/ha)		High rate (1000 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	1.36	88.7	6.91	90.1
Flonicamid	0.15	9.8	1.87	24.5
TFNG	0.56	36.4	2.13	27.8
TFNA	0.26	17.3	0.91	11.9
TFNA conjugate	0.08	5.2	0.30	3.9
TFNG-AM	0.06	4.0	0.22	2.8
TFNA-AM	0.07	4.8	0.60	7.9
PM-1a	0.05	3.2	0.19	2.4
PM-1b	0.06	3.6	0.21	2.7
Others	0.07	4.4	0.47	6.2
Unextracted	0.17	11.3	0.76	9.9
Total	1.53	100	7.67	100

PM-1a/1b: Acid hydrolyzable conjugates of TFNA

Tuber samples (from the low treatment rate) were analysed 10 days and 397 days after being placed in frozen storage. Extracted and bound residues at the 397-day interval were found to be comparable to those at the 10-day interval. The profiles were also similar between the initial and final analysis.

Considering the applications were made to the foliage of the potato plants, the presence of measurable TRRs in the tubers is evidence of translocation of the radioactivity from the foliage to the tubers. Furthermore, while the TRRs in tubers and foliage increased with increased application rate, the distribution of TRRs was relatively the same irrespective of the treatment rate.

The identity of the radioactive residues in the surface wash of tubers was not further investigated as the TRRs were too low. Analysis of each of the extracted fractions of unwashed and washed potato tubers and foliage from the low and high rate demonstrated that the predominant metabolites, TFNA and TFNG, accounted for a significant portion of the TRRs. Moreover, TFNA accounted for 32–40% of the TRR (0.04–0.21 mg eq/kg) in the unwashed and washed tubers and 12–17% TRR (0.26–0.91 mg eq/kg) in the foliage while TFNG accounted for 25–39% of the TRR in tubers (0.04–0.18 mg eq/kg) and 28–36% of the TRR in foliage (0.6–2.1 mg eq/kg). The parent, flonicamid, was also a major residue in tubers (6–12% of the TRR; 0.01–0.04 mg eq/kg) and foliage (10–25% of the TRR; 0.2–1.9 mg eq/kg), but accounted for less than the major metabolites. All other identified metabolites (TFNA conjugate, TFNG-AM< TFNA-AM, PM-1a, PM-1b and PM-3a) accounted for ≤ 6% of the TRR (0.02 mg eq/kg) in tubers and ≤ 8% of the TRR (0.6 mg eq/kg) in foliage. Overall, the general metabolic profile in foliage was similar to that in tubers.

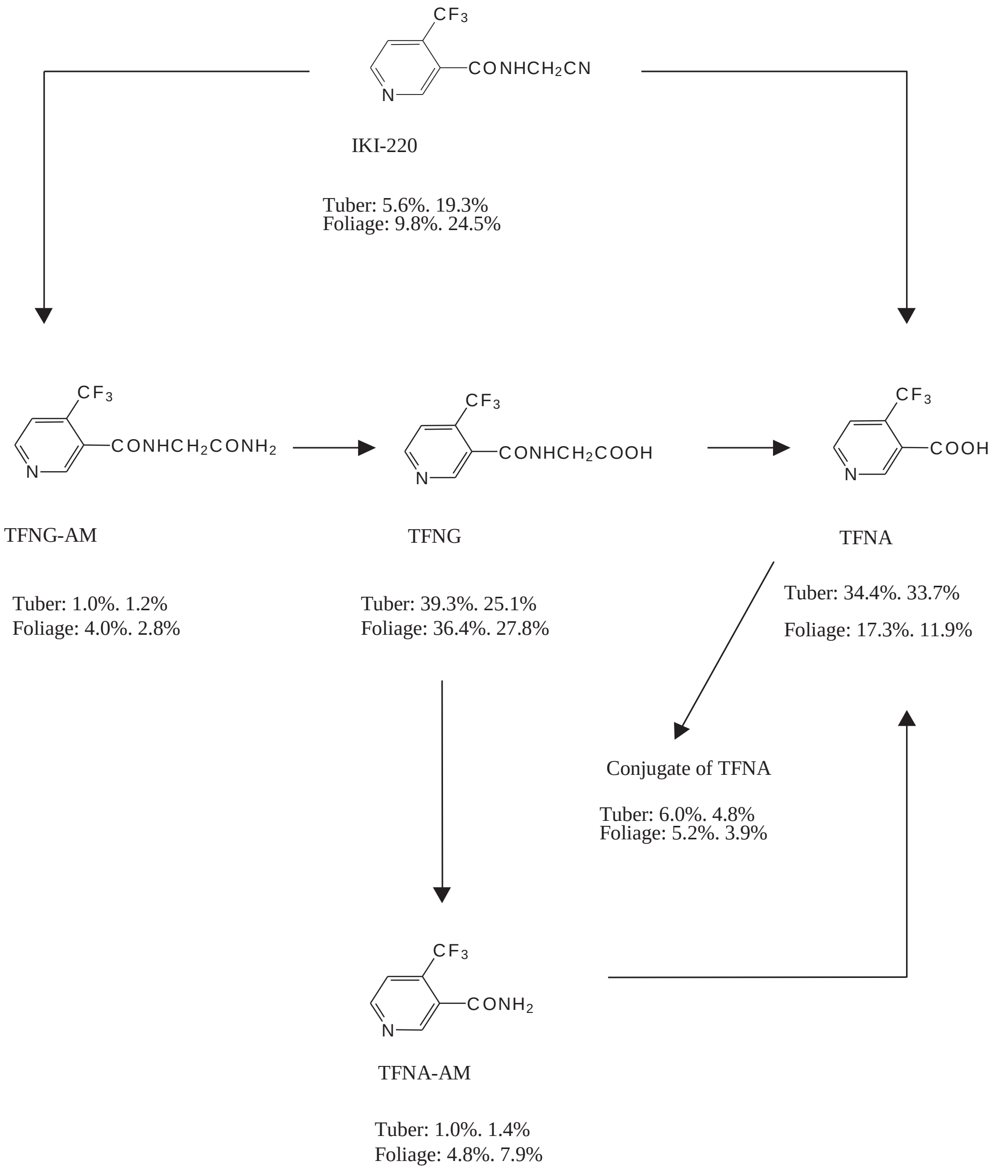


Figure 3 Proposed metabolic pathway in potato

Wheat

Study 1

Spring wheat plants (variety: Kulm) were grown in four separate plots maintained outdoor. Wheat plants grown in Plot I were designated as the control plants. Plants from Plots II and III were treated with flonicamid, radio-labelled at the 3 position of the pyridine ring (specific activity: 9.08 MBq/mg), formulated as a wettable powder, at a single application rate of 100 g ai/ha. Plants in Plot IV were treated twice at 100 g ai/ha/application with a re-treatment interval of 7 days. Forage was harvested 14 days after treatment, from Plot II. Hay was harvested from Plot III, 42 days after treatment. At final harvest, approximately 95 DAT, mature plants from Plot IV were separated into straw, chaff and grain.

The forage, hay, grain, straw and chaff were analysed to determine the total radioactive residue (TRR) levels. The radioactivity was measured with by LSC. Homogenised samples of forage, hay, straw, chaff and grain as well as the PES were combusted in an oxidizer.

Overall residues were lower in the wheat grain sample than the straw or chaff. The TRR levels in the chaff were higher compared to straw potentially because of tissue size differences (higher surface area to weight ratio) assuming a uniform application.

Table 12 Distribution of TRRs in wheat forage, hay, straw, chaff and grain

Plant part	Application rate (g ai/ha)	DAT (days)	TRRs (mg eq/kg)
Forage (Plot II)	100	14	0.648
Hay (Plot III)	100	42	0.951
Straw (Plot IV)	200	95	5.571
Chaff (Plot IV)			6.553
Grain (Plot IV)			2.559

Only samples of forage and hay were analysed to elucidate the nature of the flonicamid residues. These were homogenised in a blender with dry ice, extracted with ACN: water: acetic acid (60:40:0.1, v/v/v), blended with a tissue homogeniser, and then vacuum filtered. The process was repeated twice. The extracts were combined and concentrated by rotary evaporation under vacuum to a small volume. The concentrate was transferred to a vial with appropriate solvent and analysed by HPLC. The radioactivity in the eluate was detected using a radioactivity flow detector.

Table 13 Identification/Characterization of TRRs in wheat forage and hay

Fraction	Forage		Hay	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	0.58	96.0	0.89	96.0
Flonicamid	0.26	42.8	0.20	21.7
TFNA	0.04	6.5	0.04	3.8
TFNG	0.20	32.7	0.49	52.6
TFNA-AM	0.002	0.3	0.01	1.1
TFNG-AM	0.07	11.0	0.12	13.1
TFNA conjugate	0.004	0.7	n.d.	n.d.
Unknowns	< 0.012	<1.9	0.02	1.6
Polar	n.d.	n.d.	0.02	2.0
Unextracted	0.02	4.0	0.04	4.0
Total	0.60	100.0	0.93	100.0

The extracts were re-analysed by HPLC after storage in the freezer for approximately 2–3 months. Re-analysis confirmed the stability of metabolites in the matrices during storage.

The analysis of forage and hay samples showed that the metabolite profiles were qualitatively similar. Identified residues included flonicamid, TFNA, TFNG, TFNA-AM and TFNG-AM. Differences were observed in the distribution of minor metabolites only. Minor unknown components were observed at less than 0.02 mg eq/kg, ⩽ 2% of the TRR. A trace amount (0.1% of the TRR) of the N-oxide of flonicamid was tentatively identified in forage.

The nature and distribution of metabolites were similar in both wheat forage and hay. The parent compound, flonicamid, accounted for 42.8 and 21.7% of the TRR in forage and hay, respectively. TFNG was the predominant metabolite, accounting for 32.7 and 52.6% of the TRR in forage and hay, respectively. TFNG-AM was present at 11.0–13.1% of the TRR. Metabolites TFNA and TFNA-AM were present at < 7% of the TRR. The unextracted ^{14}C residues in forage and hay represented 0.02–0.04 mg eq/kg (4% of the TRR). Since the samples contained less than 10% of the TRR, the PES were not characterised.

Study 2

Spring wheat plants (variety: Kulm), grown outdoors in metal containers were treated with flonicamid radio-labelled at the 3 position of the pyridine ring (specific activity: 9.08 MBq/mg), formulated as a wettable granule (WG) and applied on wheat plants as an over-the-top foliar spray at Zadok's stage 86 (soft dough stage), 76 days after sowing. A single application was made at a rate equivalent to 100 g ai/ha. An additional set of wheat plants was treated at a higher rate of 500 g ai/ha. Plants were protected from rain for one week after the spray application. The wheat plants were harvested at maturity, i.e. 21 days after application and separated to straw (leaves and stem), chaff and grain (with hulls attached).

The radioactivity was measured by LSC. Homogenised samples of straw, chaff and grain as well as the PES were combusted in an oxidizer.

The TRRs in wheat straw, chaff and grain samples were 2.03 mg eq/kg, 3.60 mg eq/kg and 0.28 mg eq/kg in the 100 g ai/ha treatment plot (low rate), and 9.28 mg eq/kg, 18.88 mg eq/kg and 1.47 mg eq/kg in the 500 g ai/ha treatment plot (high rate), respectively (Table 14).

Table 14 Distribution of TRRs in wheat straw, chaff and grain

Crop part	TRRs (mg eq/kg)	
	Low rate (100 g ai/ha)	High rate (500 g ai/ha)
Straw	2.03	9.28
Chaff	3.60	18.88
Grain	0.28	1.47

Samples of homogenised straw, grain and chaff were mixed with ACN: water: acetic acid (60:40:0.1, v/v/v), blended with a tissue homogeniser, and then vacuum filtered. This process was repeated twice, following which, the extracts were concentrated and analysed using various HPLC and TLC techniques. The identification of metabolites was supported by other methods such as LC-MS, HPLC on different columns and TLC. The PES were allowed to dry, then combusted to quantitate the unextracted residues. The unextracted residues in the PES obtained by extraction of a second set of subsamples of the normal treatment rate Plot were characterised by acid (1 N HCl) and base hydrolysis (1 N NaOH). For straw and chaff the HCl digestion was followed by treatment with 72% H_2SO_4 to digest the carbohydrate (cellulose) fraction from the matrix leaving behind lignin fraction.

Table 15 Identification/Characterization of TRRs in wheat straw, chaff and grain

Fraction	Straw				Chaff				Grain			
	Low rate (100 g ai/ha)		High rate (500 g ai/ha)		Low rate (100 g ai/ha)		High rate (500 g ai/ha)		Low rate (100 g ai/ha)		High rate (500 g ai/ha)	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	1.63	80.1	7.42	79.9	2.73	75.7	15.40	81.6	0.25	89.4	1.38	94.3
Flonicamid	1.02	50.2	4.10	44.2	1.47	40.7	8.85	46.9	0.08	29.9	0.35	23.9
TFNG	0.40	19.6	1.98	21.3	0.60	16.6	3.57	18.9	0.11	39.4	0.65	44.1
TFNA	0.04	2.0	0.36	3.8	0.20	5.7	0.57	3.0	0.02	8.1	0.05	3.8
TFNG-AM	0.09	4.5	0.52	5.6	0.19	5.4	0.77	4.1	0.01	3.1	0.08	5.7
TFNA-AM	0.04	1.8	0.22	2.4	0.09	2.5	0.71	3.8	0.02	6.2	0.14	9.5
N-oxide of TFNA-AM	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.008	2.7	0.09	6.1
Unknown (M10)	0.04	2.0	0.09	1.0	0.18	4.9	0.31	1.6	0.0	0.0	0.0	0.0
Others			0.14	1.5			0.61	3.2			0.02	1.5
Unextracted	0.40	19.9	1.86	20.1	0.88	24.3	3.48	18.4	0.03	10.6	0.08	5.7
Total	2.03	100.0	9.28	100.0	3.60	100.0	18.88	100.0	0.28	100.0	1.47	100.0

Homogenized straw, chaff and grain, from the low treatment rate experiment, were extracted and analysed immediately after collection and subsequently stored for 480–505 days in a freezer prior to re-analysis. For all three commodities, extracted and bound residues were found to be comparable to those of the initial extraction. The results indicate a similar metabolite profile for straw and chaff between the first and final analysis; however, for grain a decrease in the concentration of parent and TFNA with a simultaneous increase in TFNG is observed. This does not have any significant impact on the metabolite profile.

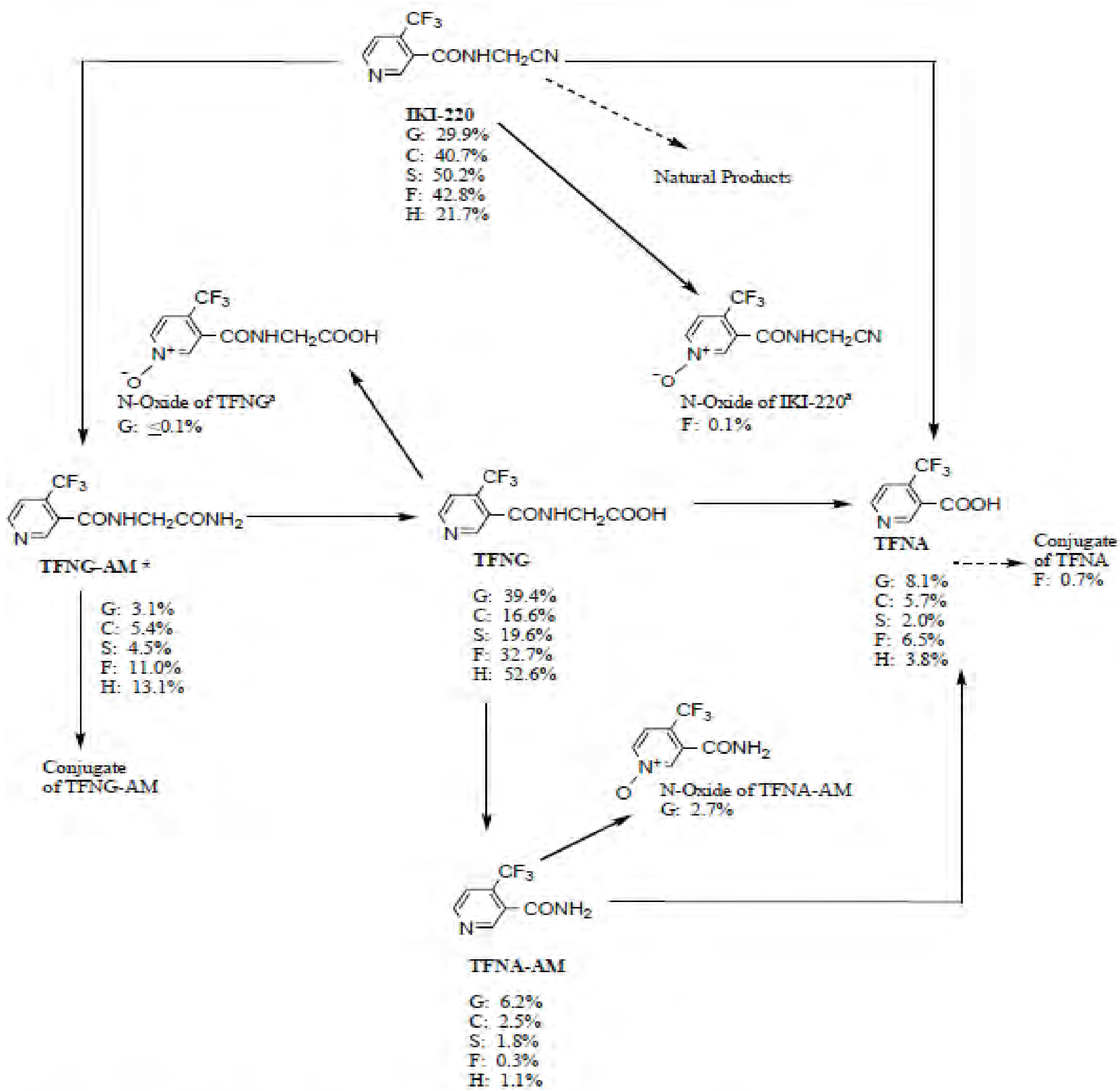
Although the TRRs in wheat straw, chaff and grain increased with increased application rates, the distribution of TRRs was relatively the same irrespective of the treatment rate. Considering the timing of application of the test material and the measurable TRRs in grain, chaff and straw at maturity, there appears to have been translocation of the radioactivity from the site of application to the mature plant parts.

The majority of the radioactivity (76–94% of the TRR) in straw, chaff and grain was extracted with organic solvents. Analysis of the organic fractions indicated that flonicamid and TFNG were the predominant residues at both treatment rates. In straw, chaff and grain, the parent accounted for 44–50% of the TRR (1.0–4.1 mg/kg), 41–47% of the TRR (1.5–8.8 mg/kg) and 24–30% of the TRR (0.08–0.4 mg/kg), respectively. The major metabolite TFNG accounted for approximately 20% of the TRR (0.4–2.0 mg eq/kg), 17–19% of the TRR (0.6–3.6 mg eq/kg) and 39–44% of the TRR (0.11–0.65 mg eq/kg) in straw, chaff and grain, respectively. All identified metabolites (TFNA, TFNG-AM, TFNA-AM and N-oxide of TFNA AM) were either not detected or were $\leqslant$ 8% of the TRR.

The unextracted residues amounted to approximately 20, 24 and 11% of the TRR in straw, chaff and grain, respectively, at the low rate and to about 20, 18 and 6% TRR at the high rate. Samples from the low rate treatment were further characterized by:

- Hydrolysis with 1 N HCl at 40 ºC to release covalently bound residues
- Hydrolysis with 1 N HCl followed by digestion with 72% H2SO4 to determine the reincorporated activity in the carbohydrate (cellulose) and lignin fractions of straw and chaff
- Hydrolysis with 1 N HCl followed by 1 N NaOH.

The radioactivity released following hydrolysis with 1 N HCl accounted for 7% of the TRR (straw), 6% of the TRR (chaff) and 3% TRR (grain) and was identified as either parent and metabolites (straw and chaff) or as metabolites of flonicamid only (grain; TFNG, TFNG-AM and TFNA-AM). Each of the identified metabolites was present at $\leqslant$ 2% of the TRR. As a result of sequential digestion with HCl and H_2SO4, ^{14}C incorporation into carbohydrates amounted to 3% of the TRR in straw and 5% of the TRR in chaff. Base digestion with 1 N NaOH released 56, 61 and 59% of the bound radioactivity in straw, chaff and grain, respectively, corresponding to 12, 14 and 5% of the TRR, respectively. Part of this radioactivity may have been due to polar sugars as released by sulphuric acid digestion, with the remainder of ^{14}C attributed to lignin (straw: 10.9% of the TRR, chaff: 13.6% of the TRR).



G = Grain, C - Chaff, S = Straw, F = Forage, H = Hay

[a] Trace quantities of N-Oxide of TFNG and N-Oxide of IKI-220 were tentatively identified in grain and in forage, respectively.

* Conjugate of TFNG-AM was detected which was converted to TFNG-AM during isolation.

Figure 4 Proposed metabolic pathway in mature wheat

Animal metabolism

The Meeting received information on the fate of 3-pyridine -^{14}C- labelled flonicamid in lactating goats and laying hens. Metabolism in laboratory animals (rat) was summarized and evaluated by the WHO panel of the 2015 JMPR.

Lactating goat

The metabolism of [^{14}C]flonicamid was investigated in two lactating goats (*Capra hircus*), weighing 45–47 kg, dosed orally once daily, using a balling gun, immediately after the morning milking, for 5 consecutive days. The animals were dosed with 3-pyridine-^{14}C-labelled flonicamid (specific activity: 245 µC/mg) at a dose level of 15 mg/day equivalent to 10 ppm in the diet. Milk production ranged from 1.5–1.9 L/day. During the treatment period, milk was collected twice daily, after the morning and evening milking, while urine and faeces were collected once daily. At sacrifice (within 5–8 hours after the last dose) samples of liver, kidney, muscle (loin and hind leg), fat (omental and peri-renal), heart and GI tract were collected.

Radioactivity in liquid samples (milk, urine, stanchion wash and extracts) was measured LSC. Samples were combusted to verify the total radioactive residues (TRR) prior to extraction. Liver, kidney, muscle, fat, faeces, heart samples and post-extraction solids (PES) were combusted in an oxidizer. The extracted samples were dried before combustion.

The major route of elimination of the radioactivity was via the urine which accounted for 49% of the total administered radioactivity (AD), while faeces accounted for 17–21% of the AD and milk accounted for 1% of the AD. Overall, the tissue burden was low, accounting for < 10% of the AD. The overall recovery of administered radioactivity averaged 95%.

The total radioactive residues (TRRs) were highest in liver (1.2 mg eq/kg), followed by kidney (0.70 mg eq/kg), muscle (0.34–0.39 mg eq/kg) and fat (0.05–0.14 mg eq/kg).

Table 16 Balance of radioactivity in goats following oral administration of [^{14}C]flonicamid for 5 days

Sample	Goat 1		Goat 2	
	%AD	mg eq/kg	%AD	mg eq/kg
Milk	1.18	0.078–0.204	0.97	0.081–0.216
Liver	1.67	1.21	1.71	1.22
Kidney	0.17	0.67	0.15	0.66
Loin muscle	3.80	0.38	3.97	0.39
Hind muscle	3.35	0.34	3.48	0.34
Perirenal fat	0.09	0.14	0.05	0.07
Omental fat	0.07	0.11	0.03	0.05
Heart	0.08	0.22	0.08	0.22
Blood	0.77	0.18	0.88	0.21
Feces	17.06	–	20.59	–
Urine	48.79	–	48.65	–
Cage wash	1.23	–	0.80	–
Subtotal	78.26	–	81.36	–
GI tract	16.62	–	14.09	–
Total Recovery	94.88	–	95.45	–

For collection Days 1–4, evening and morning milk were combined while Day 5 samples consisted of evening milk only. As TRRs were consistently higher in evening milk compared with the morning milk, in the absence of the morning milk on Day 5, ^{14}C-residues were higher than on other collection days.

Table 17 TRRs in goat milk following oral administration of [^{14}C]flonicamid for 5 days

Collection Day	Goat 1		Goat 2	
	mg eq/kg	% AD	mg eq/kg	% AD
Day 1	0.086	0.26	0.081	0.17
Day 2	0.078	0.26	0.090	0.21
Day 3	0.087	0.25	0.090	0.21
Day 4	0.095	0.27	0.096	0.22
Day 5	0.204	0.19	0.216	0.16

Extraction of milk samples with ethanol and ethanol:water (80:20, v/v) and partitioning with hexane released 97–98% of the TRRs. The PES was combusted. The procedure used for the extraction of organs and tissues was relatively similar to that of milk. However, different solvents were used. Kidney and liver samples were extracted with acetonitrile and acetonitrile:water (50:50, v/v) containing 1% acetic acid while muscle samples (loin and rear leg) were extracted with acetonitrile only. The fat samples (omental and peri-renal) were first extracted with hexane and then with acetonitrile. Use of these solvents resulted in extraction efficiencies ranging from 42–57% of the TRRs for organs, 43–52% of the TRRs for muscle and 81–86% of the TRRs for fat.

The unextracted ^{14}C residues in liver, kidney, fat and muscle tissues were sequentially hydrolysed with 1 N HCl and 6 N HCl. The PES from these organs also underwent protease digestion.

Quantification and identification of parent and metabolites were carried out by HPLC (using different solid phases). Purified metabolite isolates were analysed by mass spectrometry.

Table 18 Characterization and identification of radioactivity in goat milk, kidney and liver

Fraction	Milk				Liver				Kidney			
	Goat 1		Goat 2		Goat 1		Goat 2		Goat 1		Goat 2	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	0.084	97.4	0.09	97.7	0.57	47.3	0.513	42.1	0.362	53.9	0.377	57.3
Flonicamid	n.d.	n.d.	0.001	1.2	0.008	0.6	0.006	0.5	0.009	1.3	0.010	1.6
TFNA	n.d.	n.d.	n.d.	n.d.	0.009	0.4	0.008	0.7	0.009	1.4	0.037	5.6
TFNA unstable conjugate	n.d.	n.d.	0.004	4.6	0.082	6.8	0.051	4.2	0.080	11.9	0.010	1.6
TFNA-AM	0.084	97.4	0.082	91.9	0.355	29.4	0.352	28.9	0.207	30.8	0.270	41.1
6-OH TFNA-AM	n.d.	n.d.	n.d.	n.d.	0.069	5.7	0.078	6.4	0.041	3.2	0.041	6.3
Others	n.d.	n.d.	n.d.	n.d.	0.049	4.1	0.018	1.5	0.016	2.3	0.007	1.1
Unextracted residues	0.002	2.6	0.002	2.3	0.635	52.7	0.705	57.9	0.309	46.1	0.281	42.7
Total	0.087	100.0	0.090	100.0	1.206	100.0	1.218	100.0	0.671	100.0	0.658	100.0

n.d.= Not detected

Table 19 Characterization and identification of radioactivity in goat fat

Fraction	Omental fat				Perirenal fat			
	Goat 1		Goat 2		Goat 1		Goat 2	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	0.094	85.2	0.038	80.7	0.123	85.9	0.058	81.1
Flonicamid	0.006	5.5	0.001	2.1	0.004	3.2	0.002	2.6
TFNA	0.005	4.6	0.001	2.6	0.009	6.5	0.002	2.2
TFNA unstable conjugate	n.d.	n.d.	0.000	0.9	0.002	1.5	0.000	1.0
TFNA-AM	0.080	72.9	0.035	73.7	0.105	73.5	0.0532	74.1
6-OH TFNA-AM	0.002	1.4	n.d.	n.d.	n.d.	n.d.	0.000	0.4
Others	0.001	0.9	0.001	1.4	0.002	1.2	0.001	0.8
Unextracted residues	0.016	14.8	0.009	19.3	0.020	14.1	0.014	18.9
Total	0.110	100.0	0.047	100.0	0.143	100.0	0.072	100.0

n.d.= Not detected

Table 20 Characterization and identification of radioactivity in goat muscle

Fraction	Loin muscle				Hind leg muscle			
	Goat 1		Goat 2		Goat 1		Goat 2	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	0.170	44.3	0.167	43.1	0.177	52.1	0.172	50.8
Flonicamid	0.006	1.5	0.004	1.0	0.007	2.0	0.005	1.4
TFNA	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
TFNA unstable conjugate	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
TFNA-AM	0.165	42.8	0.163	42.1	0.170	50.2	0.166	48.8
6-OH TFNA-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.002	n.d.
Others	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Unextracted residues	0.214	55.7	0.220	56.9	0.162	47.9	0.167	49.2
Total	0.385	100.0	0.387	100.0	0.340	100.0	0.339	100.0

n.d.= Not detected

Table 21 Distribution of flonicamid and metabolites released from unextracted residues of selected goat samples

	1 N HCl digestion			6 N HCl digestion			Protease digestion		
	mg/kg	% NER	% TRR	mg/kg	% NER	% TRR	mg/kg	% NER	% TRR
Liver									
Flonicamid	–	–	–	0.006	1.0	0.5	–	–	–
TFNA	–	–	–	0.033	5.2	2.8	–	–	–
TFNA-AM	0.025	4.0	2.1	0.250	39.5	20.8	0.340	53.5	28.2
6-OH TFNA-AM	0.082	13.0	6.5	0.051	8.1	4.2	–	–	–
Kidney									
Flonicamid	n.a.	n.a.	n.a.	–	–	–	n.a.	n.a.	n.a.
TFNA	n.a.	n.a.	n.a.	0.024	7.6	3.5	n.a.	n.a.	n.a.
TFNA-AM	n.a.	n.a.	n.a.	0.169	54.6	25.2	n.a.	n.a.	n.a.
6-OH TFNA-AM	n.a.	n.a.	n.a.	0.13	4.2	1.9	n.a.	n.a.	n.a.
Loin muscle									
Flonicamid	n.a.	n.a.	n.a.	–	–	–	n.a.	n.a.	n.a.
TFNA	n.a.	n.a.	n.a.	–	–	–	n.a.	n.a.	n.a.
TFNA-AM	n.a.	n.a.	n.a.	0.120	56.1	31.2	n.a.	n.a.	n.a.
6-OH TFNA-AM	n.a.	n.a.	n.a.	–	–	–	n.a.	n.a.	n.a.

NER = Unextracted residues

n.a.= Not analysed

All samples of liver, kidney, muscle, fat and Day 3 milk were extracted and analysed within one month of collection, and re-extracted and analysed after 9 months of storage. A comparison of distribution of the TRRs in the initial and final profiles demonstrated minimal changes, indicating stability of the radioactive components under the storage conditions.

Flonicamid was rapidly metabolised in lactating goats, accounting for 0.5–5.5% of the TRRs in tissues and organs. TFNA-AM was the major metabolite in organs (29% of the TRRs in liver, 31–41% of the TRRs in kidney), tissues (74% of the TRRs in fat, 42–50% of the TRRs in muscle) and milk (97% of the TRRs). The metabolite 6-hydroxy TFNA-AM accounted for approximately 3–6% of the TRRs in liver and kidney and less than 1.4% of TRRs in tissue samples and milk.

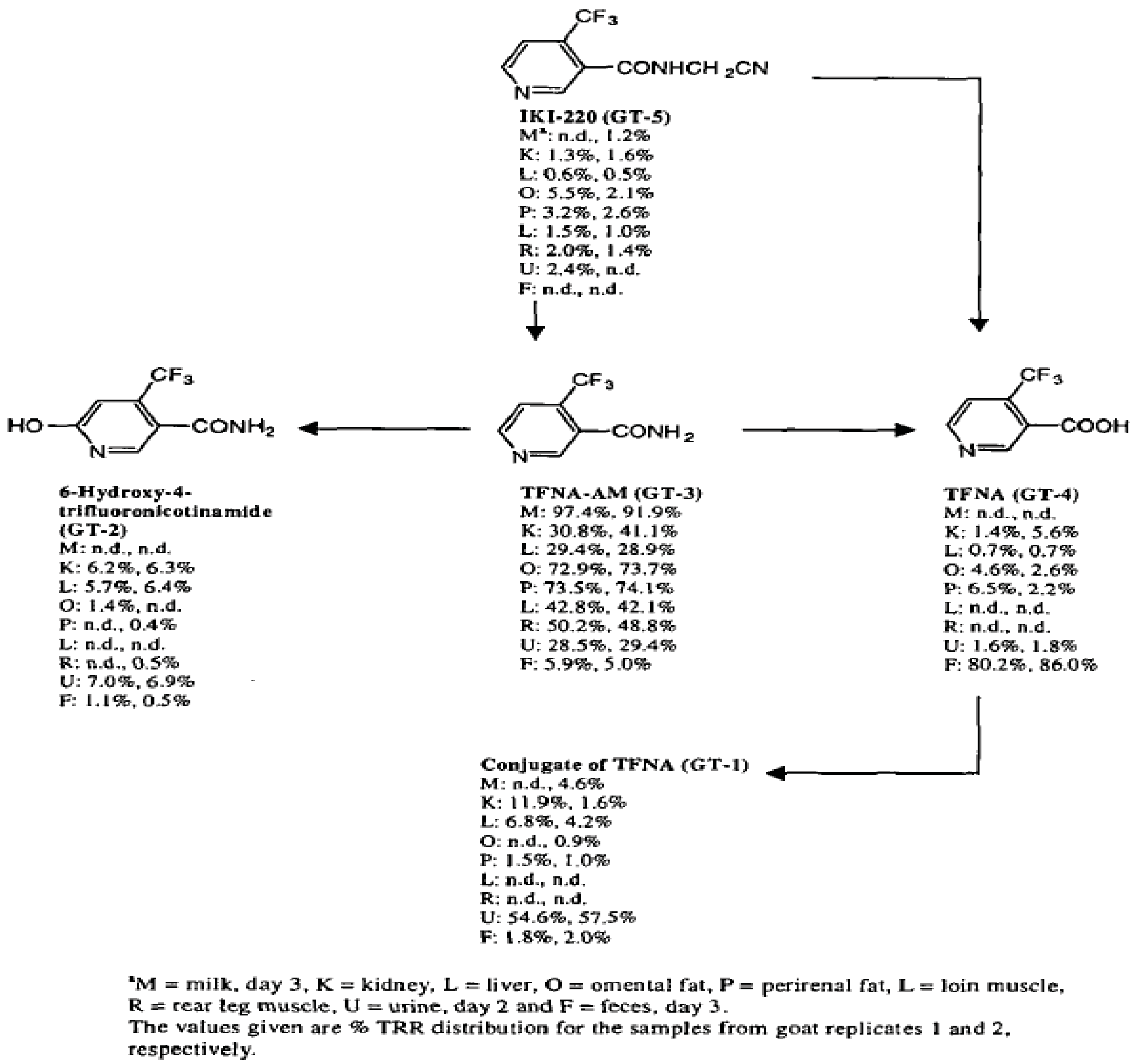


Figure 5 Proposed metabolic pathway in lactating goats

Laying Hen

Leghorn laying hens (*Gallus domesticus*), weighing 1.37–1.87 kg, were dosed orally once daily for 5 consecutive days with 3-pyridine-^{14}C-labelled flonicamid (specific activity: 1.67 MBq/mg), at 1.3 mg/day, equivalent to 10 mg/kg feed. Eggs were collected twice daily, in the morning before and in the afternoon after administration, while excreta were collected once daily. The average egg production was 95%. The animals were sacrificed approximately 6 h after the last dose and the liver, kidney, thigh muscle, breast muscle, skin and fat were collected and pooled per dose group. Radioactivity in sample solutions was determined by LSC. Solid samples of liver, kidney, muscle, fat, skin, egg and excreta were first combusted in an oxidizer to verify the total radioactive residues (TRR) prior to extraction. Both the dried extracts of tissues and the PES were combusted.

Approximately 91.1% of the administered dose (AD) including 5.7% from the gastrointestinal tract and its contents was recovered. Most of the AD (72.3%) was excreta-related. Total radioactive residues (TRR) in egg white and egg yolk accounted for about 2.4% of AD (1.8% AD in egg white plus 0.6% AD in yolk). The TRR levels in both egg white and egg yolk reached a plateau by Day 3 of dosing. The tissue burden was very low (< 6% of the AD) with highest concentrations found in skin (2.3% of the AD), followed by muscle (evenly distributed between breast and thigh muscle; each approximately 1.1% of the AD), liver (0.8% of the AD), fat (0.3% of the AD) and kidney (0.2% of the AD). Blood contained 4.7% of the AD.

Table 22 Balance of radioactivity in hens following oral administration of [^{14}C]flonicamid for 5 days

Sample	%AD	mg eq/kg
Egg white	1.84	0.04–0.89
Egg yolk	0.60	0.01–0.68
Liver	0.79	1.18
Kidney	0.22	1.42
Breast muscle	1.13	0.99
Thigh muscle	1.08	0.95
Skin	2.31	0.70
Fat	0.33	0.15
Blood	4.72	1.26
Excreta	67.18	5.20–9.51 [a]
Cage wash	5.16	1.55
Gastrointestinal tract	5.72	–
Total Recovery	91.08	

[a] Excreta collected just before sacrifice

Table 23 TRRs in eggs following oral administration of [^{14}C]flonicamid for 5 days

Day	Egg White		Egg Yolk	
	%TAR	mg/kg eq	%TAR	mg/kg eq
1	0.02	0.04	0.00	0.01
2	0.34	0.56	0.09	0.31
3	0.46	0.74	0.15	0.50
4	0.53	0.87	0.18	0.63
Sacrifice	0.49	0.89	0.18	0.68
Total	1.84		0.60	

Extraction of egg yolk and white (Day 3), liver, kidney, breast and thigh muscle with acetonitrile, and acetonitrile:water (80:20, v/v) containing 1% acetic acid released 81–99% of the TRRs. The radioactivity in each extract and in the PES, after combustion, was quantitated. Skin and fat were extracted in the same manner except that an extraction with hexane was done initially, which resulted in an extraction efficiency of 99% of the TRRs.

Each of the PES of liver and kidney was sequentially hydrolysed with 1 N HCl and 6 N HCl. Aliquots of the PES from liver and kidney were additionally hydrolysed using protease. In a separate experiment, digestion of liver PES was carried out with enzyme.

Quantification and identification of parent and metabolites were carried out by HPLC using different columns. For samples containing low levels of radioactivity, fractions of the effluent were collected and analysed by LSC. Analytical methods (HPLC) were validated with authentic standards and shown to achieve the necessary resolution and sensitivity. HPLC column performance and chromatographic resolution were validated with authentic labelled and non-labelled standards.

Table 24 Characterization and identification of radioactivity in eggs, liver and kidney

Fraction	Day 3 Egg yolk		Day 3 Egg white		Liver		Kidney	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	0.493	99.3	0.739	99.9	1.117	94.6	1.149	81.2
Flonicamid	0.019	3.8	0.018	2.5	0.004	0.3	0.005	0.4
TFNA	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.020	1.4
TFNA-AM	0.047	94.7	0.710	96.0	1.100	92.9	1.081	76.4
OH TFNA-AM	n.d.	n.d.	n.d.	n.d.	0.001	0.1	0.034	2.4
TFNG-AM	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.002	0.1

Fraction	Day 3 Egg yolk		Day 3 Egg white		Liver		Kidney	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Unknown	n.d.	n.d.	0.002	0.3	0.005	0.4	0.001	0.1
Others	0.004	0.8	0.009	1.2	0.011	0.9	0.006	0.4
Unextracted residues	0.004	0.7	0.0005	0.1	0.063	5.4	0.266	18.8
Total	0.497	100.0	0.740	100.0	1.182	100.0	1.42	100.0

Table 25 Characterization and identification of radioactivity in muscle, skin and fat

Fraction	Breast muscle		Thigh muscle		Skin		Fat	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Extracted	0.988	99.4	0.939	99.1	0.694	98.9	0.147	98.9
Flonicamid	0.006	0.6	0.004	0.4	0.003	0.4	0.001	0.7
TFNA	n.d.	n.d.	0.001	0.1	n.d.	n.d.	n.d.	n.d.
TFNA-AM	0.961	96.8	0.918	96.8	0.677	96.4	0.141	94.7
OH TFNA-AM	0.003	0.3	0.012	1.3	0.002	0.3	0.0007	0.5
TFNG-AM	n.d.	n.d.	0.001	0.1	0.0004	0.1	0.0001	0.1
Unknown	0.011	1.1	0.0003	0.0	0.002	0.3	n.d.	n.d.
Others	0.006	0.6	0.003	0.3	0.009	1.3	0.004	3.0
Unextracted residues	0.006	0.6	0.009	0.9	0.008	1.1	0.002	1.1
Total	0.99	100.0	0.95	100.0	0.70	100.0	0.15	100.0

Table 26 Distribution of metabolites released from unextracted residues of liver and kidney

	1 N HCl digestion			6 N HCl digestion			Protease digestion		
	mg/kg	% NER	% TRR	mg/kg	% NER	% TRR	mg/kg	% NER	% TRR
Liver (NER = 0.063 mg/kg)									
Flonicamid	0.036	56.5	3.0	0.028	43.5	2.3	0.063	100	5.4
TFNA	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	–	–	
TFNA-AM	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.020	–	1.7
OH TFNA-AM	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	–	–	–
Unknown	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.043	–	3.6
Kidney (NER = 0.267 mg/kg)									
Flonicamid	0.090	33.9	6.4	0.176	66.1	12.4	0.257	96.6	–
TFNA	–	–	–	0.021	7.9	1.5	–	–	–
TFNA-AM	0.022	8.3	1.6	0.143	53.6	10.1	0.216	–	15.3
OH TFNA-AM	0.068	25.6	4.8	0.012	4.6	0.9	–	–	–
Unknown	–	–	–	–	–	–	0.041	–	2.9

NER = Unextracted residues

n.a.= Not analysed

All samples of liver, kidney, muscle, fat and Day 3 egg yolks and egg whites were extracted and analysed approximately 9 months after collection. A comparison of distribution of the TRRs in the initial and final profiles demonstrated minimal changes, indicating stability of the radioactive components under the storage conditions.

Flonicamid was rapidly metabolised and excreted with only a very small percentage of the administered dose found in eggs, tissues and organs. TFNA-AM was the predominant metabolite in egg whites and egg yolks (⩽ 96.0% of the TRR), liver (92.9% of the TRR), kidney (76.4% of the TRR) and tissues (96.8% of the TRR in both breast muscle and thigh muscle, 96.4% of the TRR in skin and 94.7% of the TRR in fat).

Other metabolites identified in organs and tissues were OH-TFNA-AM and TFNG-AM; however, neither of these accounted for greater than 4.8% of TRR. One metabolite found in breast muscle and accounting for 1.1% of the TRR remained unidentified (named HN-1).

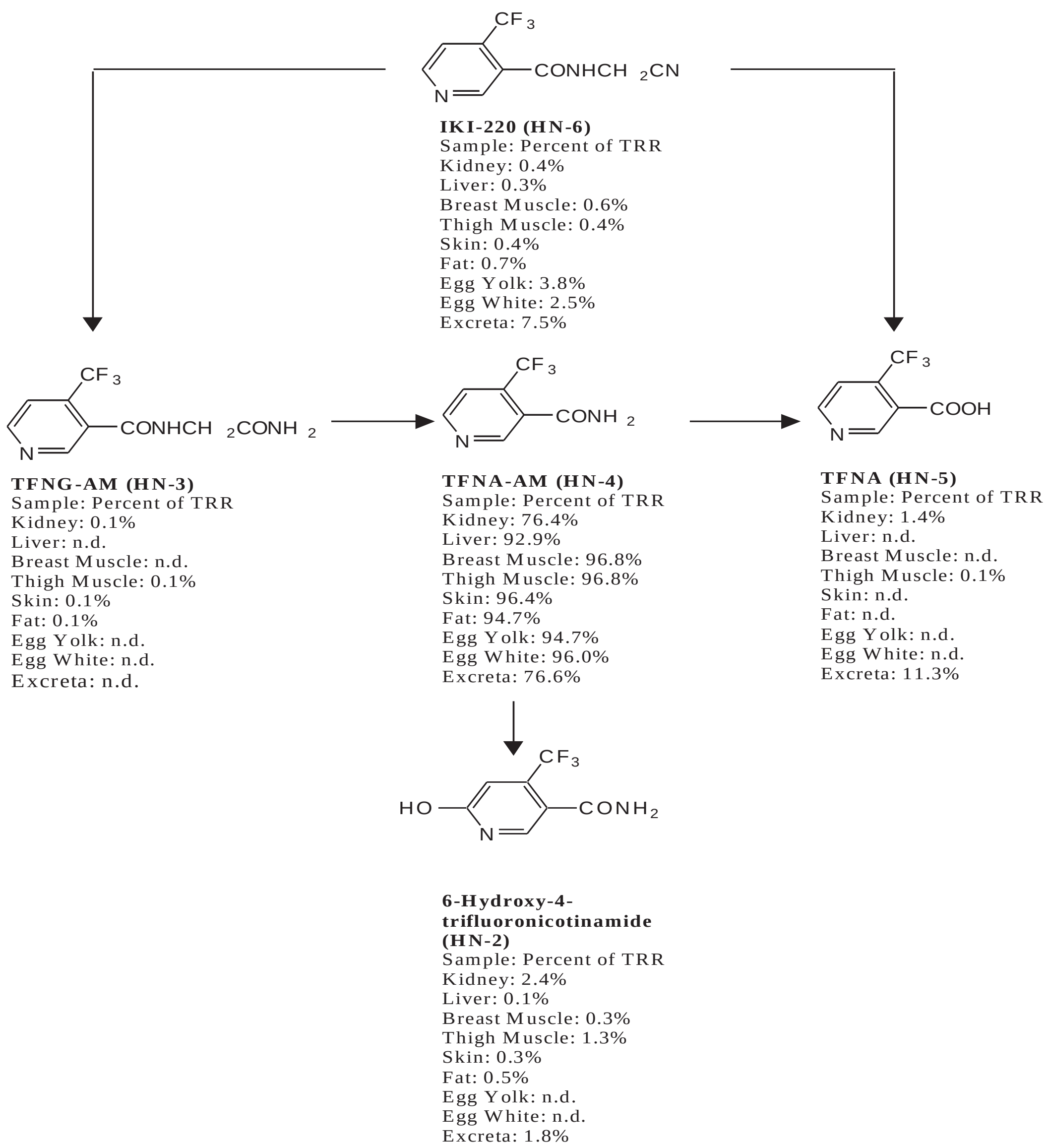


Figure 6 Proposed metabolic pathway in laying hens

Environmental fate in soil

The FAO Manual (FAO, 2009) explained the data requirements for studies of environmental fate. The focus should be on those aspects that are most relevant to MRL setting. For flonicamid, supervised residue trials data were received for foliar spray on permanent crops and on annual crops. Therefore, according to the FAO manual, only studies on aerobic degradation, photolysis and rotational crops (confined, field) were evaluated. For information on hydrolysis and photolysis see also Physical and Chemical Properties.

Aerobic degradation

The route of degradation of [^{14}C]flonicamid (specific activity 8.91 MBq/mg) in soil under aerobic conditions was investigated in a biologically active loamy sand soil collected from Madison, Ohio, USA. A subsample was transferred to a growing pot and stored in a greenhouse. Subsamples of the sieved moist soil were weighed into separate plastic bottles and treated after 43 days of equilibration.

The dosing solution was prepared in water and each subsample was treated to produce a soil concentration of 0.1 µg/g (0.1 ppm). The soil sample was connected to a series of traps to retain any volatiles. Duplicate samples of the treated soil were extracted after treatment on Day 0 and after 0.5, 1, 2, 3, 7, 14 and 30 days of incubation.

The average recovery of applied radioactivity (AR) over the 30-day course of the study was 86.3%. The recovery of radiocarbon was low in the definitive experiment for sampling times days 3, 7, 14 and 30 days due to very rapid extensive metabolism and mineralization which formed $^{14}CO_2$.

A second set of soil samples (mass balance experiment) was dosed and sampled at Days 3, 7, 14 and 30 to correct for mass balance and volatiles. The average recovery of applied radioactivity for the mass balance experiment was 93.3%. Extracted residues decreased from 101.4% AR on Day 0 to 13.7% AR after 30 days incubation. Unextracted soil residues increased steadily from 0.7% on Day 0 to 35.2% on Day 30. Evolution of $^{14}CO_2$ increased throughout the study, reaching a maximum of 47.0% AR after thirty days in this experiment.

Table 27 Distribution of radioactivity in loamy sand soil treated with [^{14}C]flonicamid and incubated at 20 °C and 45% WHC_{max} (values are the average of duplicate analyses)

Sampling	Extracted	Unextracted	CO2	Total recovery
	[% AR]			
Day 0	101.4	0.7	NA	102.1
Day 0.5	94.8	1.5	NA	96.3
Day 1	99.7	4.0	0.2	103.9
Day 2	82.7	8.0	0.3	91.0
Day 3	75.8	12.0	8.1 [a]	95.9
Day 7	51.9	30.8	26.1 [a]	108.8
Day 14	20.0	34.9	40.0 [a]	94.9
Day 30	13.7	35.2	47.0 [a]	95.9

NA = Not analysed

[a] Values corrected with the data of the mass balance experiment. In the definitive study, the $^{14}CO_2$ was not trapped efficiently.

Flonicamid rapidly declined from 99.3% AR at Day 0 to 2.3% by Day 30. Five metabolites were identified; TFNA, TFNA-OH, TFNG, TFNG-AM and TFNA-AM. TFNA and TFNA-OH were the major metabolites exceeding 10% AR. TFNA peaked at 36.4% AR on Day 3, before declining to 0% by the end of the 30-day interval. Levels of the metabolite TFNA-OH increased steadily, to 20.2% AR through 7 days, then declined to 0.5% AR at Day 30. The metabolite TFNG-AM reached a maximum of 9.6% AR by Day 0.5, but decreased to 0% on Day 7 and 1.8% AR on Day 30. TFNA-AM remained below 7% AR and TFNG below 3% AR over the course of the experiment. TFNA-AM was present in the dose solution at a level of 2.3% AR. Other more polar, minor components were detected on several days, for a combined total of less than 7% AR. The distribution of metabolites in soil treated with [^{14}C]flonicamid is shown in Table 28.

Table 28 Distribution of extracted components from soil treated with [^{14}C]flonicamid and incubated at 20 °C and 45% WHC_{max} (values are the average of duplicate analyses)

Sampling	Flonicamid	TFNA	TFNA-OH	TFNG	TFNG-AM	TFNA-AM	Others [a]	Total
	[% AR]							
Day 0	99.3	0.0	0.0	0.0	0.0	2.1	0.0	101.4
Day 0.5	66.8	14.9	0.0	0.0	9.6	3.5	0.0	94.8
Day 1	52.1	28.0	5.6	0.8	8.2	5.1	0.0	99.7
Day 2	25.2	33.5	9.7	1.7	5.0	6.2	1.4	82.7
Day 3	13.8	36.4	14.0	0.7	2.3	6.9	1.7	75.8
Day 7	4.6	20.4	20.2	0.0	0.0	5.4	1.3	51.9
Day 14	4.3	1.2	1.9	2.5	2.4	2.1	5.6	20.0
Day 30	2.3	0.0	0.5	1.6	1.8	0.9	6.6	13.7

[a] Region of diffuse radioactivity containing multiple minor components.

Flonicamid degraded rapidly at 20 °C and 45% WHC_{max} in the loamy sand soil with a DT_{50} of 1 day and a DT_{90} of 3.4 days (r^2 = 0.9960), exhibiting first-order decay kinetics.

Table 29 Degradation of [^{14}C]Flonicamid in soil under aerobic conditions

Soil	DT_{50} [days]	DT_{90} [days]	r^2
loamy sand (Madison, Ohio, USA)	1.0	3.4	0.9960

An effort was made to extract larger quantities of bound residues from the PES and to determine if flonicamid or metabolites were less extracted with time. After acid hydrolysis with 6 N HCl (at ca. 40 °C overnight), the additional radioactivity extracted from the PES represented 37% of the bound residues. Approximately 46% of the released radioactivity was organosoluble. HPLC analysis of the organic phase showed negligible amounts of parent material. The majority of residues released from soil PES were polar in nature. Based on HPLC/LSC of the acid extraction, it was concluded that the unextracted residues remaining after the initial extractions did not contain significant amounts of flonicamid (or its known metabolites).

The major degradates observed exceeding 10% of AR were TFNA and TFNA-OH. TFNG-AM, TFNG and TFNA-AM were detected as minor degradates and were formed as intermediate products. Rapid hydrolysis of the cyano group and the resulting amide group led to the formation of TFNG. Further cleavage of the glycine moiety led to the formation of TFNA. TFNA was apparently rapidly hydroxylated to TFNA-OH by micro-organisms. Mineralisation of the radioactive residues to CO_2 and binding to the soil matrix were the terminal steps in soil metabolism of flonicamid. At the end of the 30-day period, approximately half of the applied dose was mineralized to $^{14}CO_2$ and incorporated into the soil organic matter, primarily into the fulvic acid fraction.

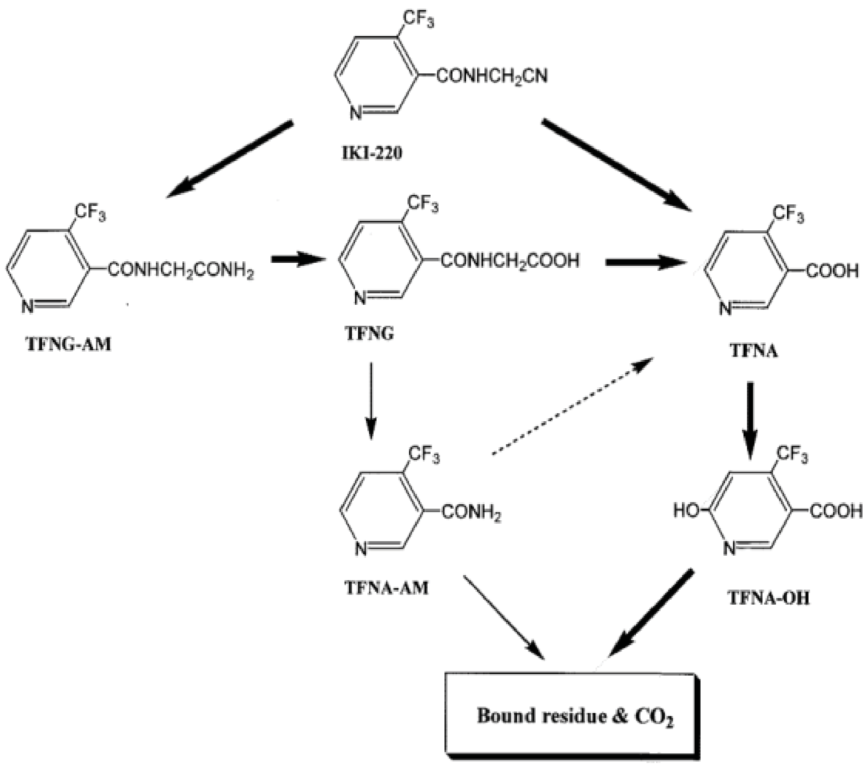


Figure 7 Aerobic degradation pathway in soil

Rate of aerobic degradation in soil

The rate of degradation of [^{14}C]flonicamid, radio-labelled at the 3 position of the pyridine ring (specific activity 9.08 MBq/mg), was investigated in three biologically active soils from the U.K. (Bedfordshire; loamy sand and Birmingham; sandy loam) and one from Germany (LUFA Speyer 2.1; sand) under aerobic conditions at 20 ± 2 °C. In addition, the degradation under aerobic conditions at 10 ± 2 °C was studied in the Bedfordshire soil.

The soil concentration of [^{14}C]flonicamid was 0.1 mg/kg (2 µg/20 g dry soil weight). The moisture content of each subsample was adjusted to approximately 50% of its maximum water holding capacity (MWHC). All three soils were connected to a series of traps to retain any volatiles and maintained in dark environmental chambers at 10 ± 2 °C or 20 ± 2 °C. Soil samples were taken at 0.33, 0.67, 1, 2, 3, 7, 14 and 30 days after treatment.

In UK soils incubated at 20 °C, the total ^{14}C recovery (based on the Day 0 dose) averaged 96% (Table 30). Extracted radioactivity decreased from 98% on Day 0 to 3% after 30 days. For the Bedfordshire soil, unextracted radioactivity reached a maximum of 37.7% by Day 30 and evolution of $^{14}CO_2$ reached 56.6% by Day 30. In Birmingham soil, the PES reached 46.2% by Day 3 and decreased slightly to 43.3% at Day 30 and evolution of $^{14}CO_2$ reached 49.3% by Day 30.

In the German soil, the total ^{14}C recovery (based on the Day 0 dose) averaged 97.2%. Extracted radioactivity decreased from 100.0% on Day 0 to 8.5% at 30 days. The PES reached a maximum of 34.6% by Day 14 and decreased slightly to 29.6% AR by Day 30. Evolution of $^{14}CO_2$ reached 56.2% by Day 30.

In all soils negligible quantities of ^{14}C (< 1%) were detected in the volatile organic compound traps.

Table 30 Mass balance of radioactivity of [^{14}C]flonicamid incubated at 20 °C in three soils (average of duplicates)

Sampling	Bedfordshire					Birmingham					Speyer 2.1				
	Extrac-ted	Unex-tracted	CO_2	VOC	Total	Extrac-ted	Unex-tracted	CO_2	VOC	Total	Extrac-ted	Unex-tracted	CO_2	VOC	Total
	[% of applied radioactivity]														
Day 0	98.1	1.7	NA	NA	99.8	98.4	2.2	NA	NA	100.6	100.0	0.7	NA	NA	100.7
Day 0.33	96.8	3.4	NA	NA	100.2	88.6	8.4	NA	NA	97.0	96.9	2.5	NA	NA	99.4
Day 0.67	90.8	6.3	NA	NA	97.1	81.0	15.8	NA	NA	96.8	96.1	3.9	NA	NA	99.9
Day 1	84.8	12.3	4.1	n.d.	101.2	69.3	23.3	5.3	0.01	97.8	94.5	3.1	1.7	0.01	99.2
Day 2	61.8	19.8	12.4	0.04	94.0	42.6	37.8	14.8	0.1	95.3	84.9	7.7	4.7	0.03	97.3
Day 3	44.1	29.2	21.7	0.08	95.1	21.7	46.2	26.1	0.2	94.1	76.8	9.4	8.5	0.06	94.8
Day 7	8.2	34.1	46.9	0.20	89.4	7.0	46.0	40.8	0.4	94.2	50.2	16.5	25.6	0.24	92.5
Day 14	4.9	36.4	52.3	0.27	93.9	4.6	42.3	45.4	0.5	92.8	13.4	34.6	47.8	0.43	96.2
Day 30	3.2	37.7	56.6	0.31	97.8	2.8	43.3	49.3	0.5	95.9	8.5	29.6	56.2	0.5	94.8

VOC = Volatile organic compounds trapped in ethylene glycol

NA = not analysed

n.d. = Not detected

In Bedfordshire soil incubated at 10 °C, the total ^{14}C recovery (based on the Day 0 dose) from this soil averaged 97.9%. Extracted radio-label decreased from 99.2% on Day 0 to 6.4% after 30 days. The PES reached a maximum of 39.6% and evolution of $^{14}CO_2$ reached 52.4% by Day 30. Negligible quantities of ^{14}C (< 1%) were detected in the volatile organic compounds traps.

Table 31 Mass Balance of radioactivity of [^{14}C]flonicamid incubated at 10 °C in Bedfordshire soil (average of duplicates)

Sampling	Bedfordshire				
	Extracted	Unextracted	CO_2	VOC	Total
	[% of applied dose]				
Day 0	99.2	1.4	NA	NA	100.5
Day 0.33	99.9	2.9	NA	NA	102.8
Day 0.67	95.1	3.9	NA	NA	99.0
Day 1	92.3	4.0	0.7	nd	97.0
Day 2	93.3	5.5	1.9	0.03	100.6
Day 3	82.7	9.4	3.7	0.13	95.8
Day 7	61.9	19.5	12.4	0.15	93.9
Day 14	25.5	32.0	34.8	0.18	92.5
Day 30	6.4	39.6	52.4	0.4	98.8

VOC = Volatile organic compounds trapped in ethylene glycol

NA = Not analysed

Nd = Not detected

[^{14}C]Flonicamid incubated in UK soils at 20 °C declined rapidly from 95% on Day 0 to 0.5% of the applied radioactivity (AR) by Day 14 (Bedfordshire soil) or to 1.5% by Day 7 and non-detectable at Day 14 (Birmingham soil). TFNA rose to a maximum of 19.2–30.6% by Day 1, and then dropped to non-detectable levels by Day 7. TFNA-OH rose to a maximum of 12.1–21.3% by Day 2/3 and declined to non-detectable levels by Day 7. TFNG-AM rose to a maximum of 7.8–9.7% by Day 0.33 and declined to less than 1.0% by Day 3 (Bedfordshire soil) or non-detectable levels by Day 7 (Birmingham soil). Minor metabolites TFNA-AM and TFNG were detected at levels ⩽ 3.7% between Day 0.33 and Day 14. Minor polar components were observed in the HPLC chromatograms at Days 1–14. All polar components were ⩽ 2.2% AR.

In Speyer 2.1 soil, a similar trend was observed where flonicamid decreased from 96.8% of the AD at Day 0 to less than 1% by Day 14. TFNA rose to a maximum of 12.2% by Day 3 then dropped to less than 0.5% of the AD at Day 14. TFNA-OH rose to a maximum of 17.6% by Day 7 and declined to 1.0% of the AD at Day 14. TFNG-AM rose to a maximum of 10.2% by Day 2 and dropped to less than 1% of the applied dose at Day 14. TFNA-AM rose to a maximum of 7.6% at Day 7 and then declined to less than 0.5% by Day 14. Minor metabolite TFNG was detected at levels below 4% between Day 0.33 and Day 14. Several minor polar components were observed in the HPLC chromatograms at Days 2–14. All polar components were less than 7.1% of the applied dose.

Table 32 Distribution of radioactivity in three soils treated with [^{14}C]flonicamid and incubated at 20 °C (average of duplicates)

Sampling [day]	Loamy sand (Bedfordshire)						Sandy loam (Birmingham)						Sand (Speyer 2.1)					
	TFNG-AM	TFNA-AM	TFNG	TFNA-OH	TFNA	Flonicamid	TFNG-AM	TFNA-AM	TFNG	TFNA-OH	TFNA	Flonicamid	TFNG-AM	TFNA-AM	TFNG	TFNA-OH	TFNA	Flonicamid
	[% of applied radioactivity]																	
0	n.d.	2.5 [a]	n.d.	n.d.	n.d.	95.1	n.d.	2.7 [a]	n.d.	n.d.	ND	95.3	n.d.	2.5 [a]	n.d.	n.d.	n.d.	96.8
0.33	9.7	2.9	2.8	2.5	12.1	66.6	7.8	2.5	2.0	3.0	11.6	61.6	4.8	2.4	1.7	n.d.	1.3	86.2
0.67	8.4	3.6	2.6	6.5	22.8	46.6	6.6	2.4	2.0	6.4	17.9	44.9	8.0	3.3	2.3	1.0	4.4	76.7
1	5.6	3.5	2.2	12.4	30.6	29.5	4.9	1.8	1.8	9.7	19.2	29.9	9.8	4.1	3.0	2.0	6.6	68.6
2	1.9	1.6	1.8	20.6	21.8	12.4	2.0	0.3	1.5	12.1	12.7	12.1	10.2	5.5	3.5	5.8	11.3	47.2
3	0.8	0.6	1.8	21.3	13.0	4.9	0.9	0.3	1.6	6.2	4.4	5.1	8.1	7.5	3.4	10.6	12.2	32.9
7	n.d.	0.6	2.9	n.d.	n.d.	1.5	n.d.	n.d.	2.1	n.d.	n.d.	1.5	2.2	7.6	3.9	17.6	6.4	6.6
14	n.d.	n.d.	2.9	n.d.	n.d.	0.5	n.d.	n.d.	2.7	n.d.	n.d.	n.d.	0.7	0.3	3.3	1.0	0.2	0.7
30	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA

[a] TFNA-AM existed as an impurity in the dose test solution at approximately 2.5%

n.d.= Not detected

NA = Not analysed

In Bedfordshire soil incubated at 10 °C parent flonicamid decreased from 96.3% AR at Day 0 to 1.7% by Day 14 (Table 33). TFNA rose to a maximum of 24.3% by Day 3, and then dropped to less than 2% by Day 14. TFNA-OH rose to a maximum of 32.7% by Day 7 and declined to 16.1% at Day 14. TFNG-AM rose to a maximum of 8.1% by Day 0.67 and dropped to less than 1% by Day 14. Minor metabolites TFNA-AM and TFNG were detected at levels below 6% between Day 0.33 and Day 14. Minor polar components were observed in the HPLC chromatograms at Days 2–14. All polar components were less than 2.3% of the administered dose.

Table 33 Distribution of radioactivity in Bedfordshire soil treated with [^{14}C]IKI-220 and incubated at 10 °C (average of duplicates)

Sampling	Loamy sand (Bedfordshire)					
	TFNG-AM	TFNA-AM	TFNG	TFNA-OH	TFNA	Flonicamid
	[% of applied radioactivity]					
Day 0	n.d.	2.6 [a]	n.d.	n.d.	n.d.	96.3
Day 0.33	7.4	3.0	2.4	0.8	5.1	80.6
Day 0.67	8.1	2.9	2.8	2.2	9.3	69.0
Day 1	8.0	3.9	3.9	4.7	15.6	55.6
Day 2	6.6	5.3	3.0	12.3	24.0	41.4
Day 3	4.1	5.4	2.7	20.4	24.3	25.0
Day 7	1.0	4.1	2.5	32.7	11.8	6.7
Day 14	0.5	0.4	2.5	16.1	0.5	1.7
Day 30	NA	NA	NA	NA	NA	NA

[a] TFNA-AM existed as an impurity in the dose test solution at approximately 2.5%

n.d. = Not detected

NA = not analysed

The DT_{50} and DT_{90} values were calculated for each soil set. First-order kinetics were observed for all soils. For the soils incubated at 20 °C the DT_{50} and DT_{90} values ranged from 0.7 to 1.8 days and 2.3 to 6.0 days, respectively. The DT_{50} value was 2.4 days and the DT_{90} value was 7.9 days for the soil incubated at 10 °C. The most rapid degradation kinetics were observed for the Bedfordshire soil and the Birmingham soil at 20 °C. The DT_{50} and DT_{90} values for each soil set are shown in Table 34.

Table 34 Aerobic degradation of flonicamid in soil incubated at 20 °C and 10 °C

Soil		Incubation	DT_{50}	DT_{90}	
Type	Origin	temperature	[days]	[days]	r^2
Loamy sand	Bedfordshire	20 °C	0.7	2.3	0.9898
Sandy loam	Birmingham		0.7	2.4	0.9890
Sand	Speyer 2.1		1.8	6.0	0.9989
Loamy sand	Bedfordshire	10 °C	2.4	7.9	0.9721

TFNA, TFNA-OH and TFNG-AM were the major degradates in all soils over the course of the study which peaked at levels of 12.2 to 30.6%, 12.1 to 32.7% and 7.8 to 10.2% respectively, of the applied radioactivity. Minor degradates TFNG and TFNA-AM were detected at less than 7.7% AR at all sampling points over the course of the study. All of the degradates were metabolised and mineralised to carbon dioxide and immobilised as soil-bound residue.

Soil photolysis

The photochemical degradation of [pyridyl-^{14}C]flonicamid (specific activity 9.08 MBq/mg) was investigated in a loamy sand soil (pH 7.2, 0.98% organic matter, origin Madison, Ohio, USA) under laboratory conditions.

Ten grams (10 g) of dried soil was weighed into each photolysis vessel, made of clear glass, to a depth of approximately 3 mm. The fortification solution of [^{14}C]flonicamid (ca. 50 µL) was added onto the soil surface of each sample jar at a rate of approximately 0.1 mg eq/kg (0.1 µg/g) by means of a syringe. A total of 16 dark control and 16 light exposed samples were prepared. Two additional sample vessels were prepared for each treatment condition at an exaggerated rate (4×) for use in metabolite isolation as required. The temperature of the light-exposed and dark control samples was maintained at 20 ± 1 °C throughout the study. The light exposed and dark control samples were analysed at 0, 1, 3, 7, 9, 11 and 15 days after fortification.

Whilst volatile radioactivity was not trapped, based on the overall recoveries, good material balance was achieved, precluding the requirement for volatile traps.

[^{14}C]Flonicamid decreased from 99.0% of the applied radioactivity (AR) on Day 0 to 59.5% AR after 15 days continuous illumination. Concurrently, the metabolite TFNG-AM was detected in Day 1 sample extracts at 2.9% AR and increased to 29.5% AR by Day 15. TFNA-AM and TFNG were also detected as minor metabolites in the illuminated soils, reaching maximum concentrations of 5.0% (Day 11 and Day 15) and 2.0% AR (Day 15), respectively.

In the dark controls flonicamid decreased from 99.0% of the AR on Day 0 to 80.4% AR after 15 days dark storage. The metabolite TFNG-AM was detected in Day 1 sample extracts at 1.2% AR and increased to 13.8% AR by Day 15 samples. TFNA-AM and TFNG were detected as minor metabolites reaching maximum concentrations of 2.8% (Day 9 and Day 15) and 2.0% AR (Day 15), respectively. The mass balance and ^{14}C distribution of radioactivity from the photochemical degradation of [^{14}C]flonicamid on soil is shown in Table 35.

Table 35 ^{14}C distribution of radioactivity from the photochemical degradation of [^{14}C]flonicamid on soil—light-exposed and dark-control samples

Sampling	Extracted	Flonicamid [a] [% AR]	TFNG-AM [a] [% AR]	TFNA-AM [a] [% AR]	TFNG [a] [% AR]	Bound	Recovery
Exposed							
Day 0	101.3	99.0	–	2.3	–	0.3	101.6
Day 1	97.8	92.0	2.9	2.9	–	0.8	98.5
Day 3	96.5	88.0	5.9	2.6	–	1.2	97.7
Day 7	99.8	79.8	15.0	4.6	0.4	2.0	101.8
Day 9	99.1	77.2	17.0	3.9	1.0	1.7	100.7
Day 11	98.5	69.9	22.0	5.0	1.6	1.7	100.2
Day 15	96.0	59.5	29.5	5.0	2.0	1.7	97.7
Dark							
Day 0	101.3	99.0	–	2.3	–	0.3	101.6
Day 1	99.6	95.8	1.2	2.5	–	0.7	100.3
Day 3	99.4	93.2	3.8	2.3	–	1.1	100.4
Day 7	99.5	88.3	7.8	2.5	1.0	1.2	100.7
Day 9	100.3	86.1	10.1	2.8	1.4	1.2	101.6
Day 11	99.4	85.7	11.1	2.6	–	0.9	100.3
Day 15	98.9	80.4	13.8	2.8	2.0	1.8	100.7

[a] (% ^{14}C in designated fractions) × (% extracted)

Recovery = Extracted plus bound residues

Values are average of duplicate samples

The degradation of flonicamid appears to have followed first order kinetics. A linear regression analysis was performed on the data generated by the HPLC analyses of the samples. The resultant DT_{50} values were 22.4 days for the exposed samples (correlation coefficient (R^2) = 0.9729) and 53.3 days (R^2 = 0.9589) for the dark control samples (Table 36).

Table 36 Calculated values of the DT_{50} from the soil photolysis of flonicamid

	DT_{50} [days]	R^2
Loamy sand (Madison, Ohio, USA)		
exposed	22.4	0.9729
dark controls	53.3	0.9589

Residues in succeeding crops

Confined rotational crop

Flonicamid, radio-labelled at the 3 position of the pyridine ring (specific activity: 9.08 MBq/mg), formulated as a wettable granule (WG) was applied twice to loamy sand soil at a rate equivalent to 100 g ai/ha at an interval of two weeks. Soils were allowed to age under greenhouse conditions after treatment and prior to planting. After the appropriate plant-back intervals (PBIs) of 30, 120 or 360 days, the rotational crops, representative of the root vegetable (carrot), small grain (wheat), and leafy vegetable (lettuce) crop groups, were planted.

The crop samples of lettuce, carrot and wheat (forage, straw, chaff and grain) were homogenised with dry ice and analysed for total ^{14}C residues by combustion analysis. The PES were also analysed by combustion analysis. The TRR combustion data for crop samples are summarized in Table 37.

Table 37 Distribution of TRR levels of harvested crops

Plant-back Interval (days)	Plant part	TRR (mg/kg)	%TRR			
			Extracted	Unextracted	Total Identified	Total Characterized
30	Immature lettuce	0.006	76.4	23.6	45.4	16.5
	Mature lettuce	0.004	59.9	40.1	36.1	20.1
	Immature carrot	0.011	78.4	21.7	Not reported	
	Mature carrot root	0.004	71.6	28.4	48.7	19.3
	Mature carrot foliage	0.019	73.5	26.5	50.8	18.9
	Wheat forage	0.077	92.6	7.4	85.6	3.1
	Wheat straw	0.140	77.7	22.3	62.1	9.8
	Wheat chaff	0.078	82.2	17.8	63.3	12.8
	Wheat grain	0.029	81.5	18.6	73.3	3.9
120	Immature lettuce	0.004	Not extracted			
	Mature lettuce	0.004	Not extracted			
	Immature carrot	0.006	65.9	34.1	48.2	11.0
	Mature carrot root	0.003	Not extracted			
	Mature carrot foliage	0.005	55.0	45.0	39.1	12.1
	Wheat forage	0.009	80.8	19.2	70.1	7.8
	Wheat straw	0.031	73.0	27.0	54.7	9.5
	Wheat chaff	0.023	67.2	32.8	55.9	6.7
	Wheat grain	0.010	67.2	32.9	59.8	2.5
360	Immature lettuce	0.002	n.e.	n.e.	Not analysed	
	Mature lettuce	0.001	n.e.	n.e.		
	Immature carrot	0.003	n.e.	n.e.		
	Mature carrot root	< 0.001	n.e.	n.e.		
	Mature carrot foliage	0.002	n.e.	n.e.		
	Wheat forage	0.007	40.8	59.1		
	Wheat straw	0.017	43.6	56.4		
	Wheat chaff	0.013	47.5	52.5		
	Wheat grain	0.005	27.7	72.3		

Table 38 Identification/Characterization of TRRs

Plant-back Interval (days)	Plant part	%TRR (mg/kg in parentheses)						
		Flonicamid	TFNA	TFNA-OH	TFNG	TFNA-AM	TFNG-AM	Unknowns
30	Immature lettuce	8.9 (0.0)	8.2 (0.0)	4.9 (0.0)	10.0 (0.0)	4.4 (0.0)	9.0 (0.0)	16.5 (0.001)
	Mature lettuce	5.7 (0.0)	1.8 (0.0)	2.0 (0.0)	2.7 (0.0)	3.7 (0.0)	20.2 (0.001)	20.1 (0.001)
	Immature carrot	Not reported						
	Mature carrot root	2.3 (0.0)	1.0 (0.0)	1.9 (0.0)	4.9 (0.0)	15.4 (0.001)	23.2 (0.001)	19.3 (0.001)
	Mature carrot foliage	5.2 (0.001)	2.0 (0.0)	2.2 (0.0)	7.7 (0.001)	7.9 (0.002)	25.8 (0.005)	18.9 (0.004)
	Wheat forage	3.3 (0.003)	11.4 (0.010)	37.8 (0.033)	15.0 (0.013)	6.9 (0.006)	11.2 (0.010)	3.1 (0.003)
	Wheat straw	4.5 (0.007)	2.5 (0.004)	9.6 (0.023)	15.4 (0.023)	8.6 (0.013)	21.5 (0.032)	9.8 (0.014)
	Wheat chaff	4.1 (0.003)	9.4 (0.008)	9.1 (0.008)	18.3 (0.015)	7.5 (0.006)	14.9 (0.013)	12.8 (0.011)
	Wheat grain	5.1 (0.001)	19.6 (0.005)	4.7 (0.001)	36.3 (0.010)	2.2 (0.001)	5.4 (0.002)	3.9 (0.001)
120	Immature	Not further analysed						

Plant-back Interval (days)	Plant part	%TRR (mg/kg in parentheses)						
		Flonicamid	TFNA	TFNA-OH	TFNG	TFNA-AM	TFNG-AM	Unknowns
	lettuce							
	Mature lettuce	Not further analysed						
	Immature carrot	4.7 (0.0)	4.6 (0.0)	4.0 (0.0)	11.0 (0.001)	7.1 (0.0)	16.8 (0.001)	11.0 (0.001)
	Mature carrot root	Not further analysed						
	Mature carrot foliage	1.6 (0.0)	4.0 (0.0)	2.1 (0.0)	7.9 (0.0)	10.8 (0.001)	12.7 (0.001)	12.1 (0.001)
	Wheat forage	10.5 (0.001)	4.5 (0.0)	6.2 (0.001	20.9 (0.002)	8.0 (0.001)	19.9 (0.002)	7.8 (0.001)
	Wheat straw	1.4 (0.0)	2.3 (0.001)	4.4 (0.001)	10.9 (0.004)	10.7 (0.003)	25.0 (0.006)	9.5 (0.003)
	Wheat chaff	5.9 (0.001)	1.8 (0.0)	6.4 (0.002)	15.9 (0.004)	8.2 (0.002)	17.7 (0.004)	6.7 (0.002)
	Wheat grain	12.9 (0.001)	8.6 (0.001)	1.0 (0.0)	32.1 (0.003)	3.0 (0.0)	3.2 (0.0)	2.5 (0.0)

The homogenized tissue samples were extracted three times with acetonitrile/water 40:60 v/v (0.1% phosphoric acid). Each extract was centrifuged or vacuum filtered. The PES was allowed to air dry and then was subjected to combustion analysis. The solvent extracts were pooled then reduced to a small volume by rotary evaporation under reduced pressure. If sufficient residue was detected in the extract or the PES, additional analysis was conducted by HPLC-LSC to characterize the nature of the ^{14}C residue present.

TRRs in all raw agricultural commodities (RACs) declined with prolonged PBIs such that, at the 120-day PBI, no further characterization/identification of the TRRs was performed for immature and mature lettuce and mature carrot roots due to the low total radioactivity. Further to this, at the 360-day PBI, none of the TRRs from any of the crop parts were further subjected to characterization/identification as these were too low.

Overall, extraction of the TRRs with organic solvents released greater than 55% of the TRRs. In most commodities, only small amounts of flonicamid and TFNA-OH were detected with TFNG and TFNG-AM identified as major metabolites. In wheat grain, TFNA was also observed as a major metabolite while in wheat forage, TFNA and TFNA-OH accounted for greater than 10% of the TRRs. TFNA-AM was the only predominant metabolite in mature carrot root.

Field rotational crop

At each of the six field trials conducted in the US, three applications of flonicamid 50WG were made to the primary crop (cotton) at the maximum rate of 0.1 kg ai/ha at 7 ± 1 day intervals, resulting in a total seasonal application rate of approximately 0.31 kg ai/ha. Following harvest of the treated cotton, the rotational crops, wheat (four sites) and turnips (two sites), were planted at 30 and 60 days following the last application. The wheat and turnip samples were taken for analysis at normal maturity for each crop matrix.

Aliquots of homogenised samples were extracted twice with acetonitrile:water (50/50, v/v). Concentrated HCl was added to the combined extracts prior to being filtered and partitioned with ethyl acetate (twice). The combined ethyl acetate extract was evaporated just to dryness and residues taken up in acetonitrile:water (30/70, v/v) and analysed by LC-MS/MS. For wheat straw, an additional SPE clean-up step using a C_{18} cartridge before the partitioning with ethyl acetate was inserted.

Table 39 Maximum residues in rotated crop samples 30 and 60 days following the last application of Flonicamid 50WG to cotton

	Plant back interval [days]	Flonicamid [mg/kg]	TFNG [mg/kg]	TFNA [mg/kg]	TFNA-AM [mg/kg]
Wheat forage	30–32	n.d.	n.d.	< LOQ (< 0.01)	n.d.
Wheat straw	30–32	n.d.	< LOQ (< 0.02)	n.d.	n.d.
Wheat grain	30–32	n.d.	n.d.	< LOQ (< 0.01)	n.d.
Turnip tops	30	n.d.	n.d.	n.d.	n.d.
Turnip roots	30	n.d.	n.d.	n.d.	n.d.
Wheat forage	58–63	n.d.	n.d.	n.d.	n.d.
Wheat straw	58–63	n.d.	n.d.	n.d.	n.d.
Wheat grain	58–63	n.d.	n.d.	n.d.	n.d.
Turnip tops	59–60	n.d.	n.d.	n.d.	n.d.
Turnip roots	59–60	n.d.	n.d.	n.d.	n.d.

n.d. = Not detected

LOD = 0.005 mg eq/kg

No quantifiable residues of flonicamid or its metabolites TFNG, TFNA, and TFNA-AM were detected in any crop matrix in any rotational crop planted at either 30 or 60 days after the last application of flonicamid to the primary crop of cotton.

RESIDUE ANALYSIS

Analytical Methods

The Meeting received descriptions and validation data for analytical methods for residues of flonicamid and its metabolites TFNA-AM, TFNA and TFNG in plant commodities and flonicamid, TFNA-AM, TFNA, TFNG and OH-TFNA-AM in animal commodities. All residue analytical methods rely on LC-MS/ MS. Typical LOQs achieved for plant and animal commodities fall in the range of 0.01–0.02 mg/kg. The LOQs for milk and animal products (liver, kidney, muscle, eggs) were 0.01 mg/kg for each analyte. Methods have been subjected to independent laboratory validation. The methods described briefly below have been used for the analysis of the samples generated during the supervised field trials, processing studies and storage stability investigations.

Table 40 Characterization of Enforcement Analytical Methods for Plant and Animal Commodities

Method ID	Method Type	Detector	Analytes	LOQ/analyte	Matrices	Report
Plant Commodities						
P-3561M	Enforcement	HPLC-MS/MS	Flonicamid TFNA-AM TFNA TFNG	0.01 mg/kg for peach and potato tuber 0.02 mg/kg for wheat straw	Peach Potato tuber Wheat straw	IB-2002-JLW-011-00
	ILV	HPLC-MS/MS	Flonicamid TFNA-AM TFNA TFNG	0.01 mg/kg	Cottonseed	02-0031
P-3822	Enforcement	HPLC-MS/MS	Flonicamid TFNA-AM TFNA TFNG	0.01 mg/kg all matrices 0.02 mg/kg wheat straw and cotton matrices	Various RACs and processed commodities	178MVL05 R1
	ILV	HPLC-MS/MS	Flonicamid TFNA-AM TFNA TFNG	0.01 mg/kg	Various RACs and processed commodities	
AGR/MOA/IKI220-1 v.1	Enforcement	HPLC-MS/MS	Flonicamid TFNA-AM	0.01 mg/kg	Lemon Potato	ISK/IKI/06001

Method ID	Method Type	Detector	Analytes	LOQ/analyte	Matrices	Report
			TFNA TFNG		Oilseed rape Wheat grain Plum Prune	
	ILV	HPLC-MS/MS	Flonicamid TFNA-AM TFNA TFNG	0.01 mg/kg	Lemon Potato Oilseed rape Wheat grain Plum Prune	S09-01231
Animal Commodities						
842993	Enforcement	HPLC-MS/MS	Flonicamid TFNA TFNA-AM OH-TFNA-AM TFNG	0.01 mg/kg	Milk	
844743	Enforcement	HPLC-MS/MS	Flonicamid TFNA TFNA-AM OH-TFNA-AM TFNG	0.01 mg/kg	Bovine muscle, liver, kidney, fat Poultry muscle, liver, fat and eggs	
P-3581	ILV	HPLC-MS/MS	Flonicamid TFNA TFNA-AM OH-TFNA-AM TFNG	0.01 mg/kg	Eggs	178ILV02R1
ADPEN-2K2-1126	ILV	HPLC-MS/MS	Flonicamid TFNA TFNA-AM OH-TFNA-AM TFNG	0.01 mg/kg	Beef muscle	
AGR/MOA/IKI-5	Enforcement	HPLC-MS/MS	Flonicamid TFNA-AM	0.01 mg/kg	Bovine muscle, fat and liver, milk and eggs	S12-04426
P-2960	ILV	HPLC-MS/MS	Flonicamid TFNA-AM	0.01 mg/kg	Bovine muscle, fat and liver, milk and eggs	

Plant Commodities

Method P-3561M

Residues of flonicamid and its metabolites TFNG, TFNA and TFNA-AM were extracted twice with acetonitrile/deionised water (1/1, v/v). After centrifugation, the extracts were combined and evaporated until dryness. The sample extract was then acidified and filtered. In the case of wheat straw, the sample extract underwent clean-up using a C_{18} SPE column eluted with acetonitrile/deionised water (1/4, v/v). The eluate (in the case of wheat straw sample) or the filtrate (in the case of potato tuber or peach sample) was liquid-liquid partitioned twice in ethyl acetate. The ethyl acetate layer was evaporated to near dryness and diluted in acetonitrile/deionised water (3/7, v/v) before quantification by HPLC-MS/MS.

The method underwent successful inter-laboratory validation by EN-CAS laboratories using cottonseed. Average recoveries of flonicamid, TFNG, TFNA and TFNA-AM ranged from 70–110% with RSD of ⩽ 16%, demonstrating good reproducibility.

Method P-3822

The HPLC-MS/MS method P3822 quantifies residues of flonicamid and its metabolites TFNG, TFNA and TFNA-AM in raw agricultural commodities and processed commodities. The extraction and clean-up steps of method P-3822 are very similar to those of P-3561M, however; for oily crop samples (e.g. cotton matrices and potato chips), an additional hexane partition step and acidification is included before filtration.

The method underwent successful inter-laboratory validation by EN-CAS laboratories using the same commodities. Average recoveries of flonicamid, TFNG, TFNA and TFNA-AM ranged from 70–110% with RSD of ≤ 16% (with the exception of pepper/TFNG at 0.1 mg/kg where the RSD was 33%), demonstrating overall good reproducibility.

AGR/MOA/IKI220-1 v.1

Flonicamid and its major metabolites were extracted with a mixture of acetonitrile/water/acetic acid (60/40/0.1, v/v/v), followed by a washing with hexane (except for potato) and a clean-up using a C_{18} phase SPE cartridge (except for potato and lemon), and followed by a liquid/liquid partition with ethyl acetate. After evaporation to dryness, the residues of flonicamid and its major metabolites were dissolved in a mixture of acetonitrile/water (30/70, v/v) prior to analysis by HPLC-MS/MS using two mass transitions. For oil-seed rape, the TFNA results were confirmed by the use of another liquid chromatographic column.

The method underwent successful inter-laboratory validation by Eurofins laboratories using the same commodities. Average recoveries of flonicamid, TFNG, TFNA and TFNA-AM ranged from 70–110% with RSD of ≤ 20%, demonstrating good reproducibility.

AATM-R-165

Residues of flonicamid and its metabolites were extracted by shaking with acetonitrile:water (1:1). The extract was then decanted and the extraction was repeated with acetonitrile. An aliquot of the combined acetonitrile extracts was diluted with water, filtered and analysed by ultra performance liquid chromatography (UPLC) with positive-ion electrospray ionization (ESI) tandem mass spectrometry (MS/MS). Quantitation of the analytes was achieved by comparison with mixed external standards of flonicamid and its metabolites.

Due to the nature of the cottonseed oil samples, the method was modified such that cottonseed oil was dissolved in hexane and then partitioned with acetonitrile. An aliquot of the acetonitrile layer was taken and diluted with water and analysed.

Determination of Residues of IKI-220 and its Metabolites TFNG, TFNA and TFNA-AM in Various Crops—Validation of the Method"

This method was used for the freezer storage stability study A-22-00-03 whereby residues of flonicamid and its metabolites TFNG, TFNA and TFNA-AM were extracted with methanol. After filtration, the sample solution was washed with n-hexane, concentrated and then cleaned-up on a C_{18} SPE cartridge. The eluate was evaporated to dryness and methylated with diazomethane/diethylether. After concentration, the residues were liquid-liquid partitioned twice in ethyl acetate. The ethyl acetate layer was filtered through anhydrous sodium sulphate and evaporated to dryness. The residues were purified on a Florisil SPE cartridge and reconstituted in acetone prior to analysis by GC/MSD.

Animal Commodities

Method 842993

Residues of flonicamid and its metabolites TFNA, TFNA-AM, OH-TFNA-AM and TFNG in milk samples were extracted twice with ethanol/water (4/4, v/v). The sample extracts were combined and evaporated to dryness prior to liquid-liquid partitioning twice with hexane. The aqueous phase was evaporated to dryness and the residue was dissolved in water/acetonitrile/trifluoroacetic acid (90/10/0.1, v/v/v) prior to HPLC-MS/MS analysis.

Method 844743

Bovine and Poultry Tissues

Residues of flonicamid and its metabolites TFNA, TFNA-AM, OH-TFNA-AM and TFNG were extracted twice with acetonitrile/water (8/2, v/v). After addition of silicon anti foaming agent to the combined extracts of each tissue/egg sample, the solution was evaporated to dryness. The residue was then dissolved in methanol/water/acetic acid (2000/500/15, v/v/v) prior to liquid-liquid partitioning twice with hexane. The aqueous phase was evaporated to dryness and the residue was dissolved in water/acetonitrile/trifluoroacetic acid (90/10/0.1, v/v/v) prior to clean-up using gel permeation chromatography (GPC). The eluate was evaporated to dryness and redissolved in methanol/water (1/9, v/v) and subject to HPLC-MS/MS analysis.

Eggs

Residues of flonicamid and its metabolites TFNA, TFNA-AM, OH-TFNA-AM and TFNG in egg samples were extracted with acetonitrile/water (8:2; v/v) and the suspension was treated at 60 °C for 1 h. After denaturisation, the process was repeated. The extracts were subsequently combined and partitioned by a liquid-liquid extraction with cyclohexane. The acetonitrile/water phase was evaporated to dryness and the residue was dissolved in methanol/water/acetic acid (2000:500:15; v/v/v) followed by clean-up using GPC. The eluate was evaporated to dryness and the residue was re-dissolved with 30% acetonitrile in water prior to analysis using HPLC-MS/MS.

The method underwent successful inter-laboratory validation by FMC Princeton Environmental Sciences Laboratory using poultry eggs. Average recoveries of flonicamid, TFNG, TFNA and TFNA-AM ranged from 77–108% with RSD of ⩽ 21%, demonstrating good reproducibility.

AGR/MOA/IKI-5

Residues of flonicamid and its metabolite TFNA-AM in samples of bovine, muscle, fat, liver, milk and eggs were extracted with acidified acetonitrile/water (80/20, v/v). All the contents of the dispersive SPE citrate extraction tube were added to the extracts and shaken vigorously by hand, vortexed and centrifuged. The supernatant was evaporated to dryness and residues were dissolved in acetonitrile/ water (10/90, v/v) and filtered (liver only) prior to analysis by HPLC-MS/MS.

The method underwent successful inter-laboratory validation by PTRL Europe using the same commodities. Average recoveries of flonicamid and TFNA-AM were within the range of 70–110% with RSD of ⩽ 20%, demonstrating good reproducibility.

P-3580

While the Meeting did not receive a description of the method P-3580 titled `Radio-validation of Goat Muscle Treated with $^{14}$C-Radio-labelled IKI-220 (F1785) Insecticide and method Validation of Residue Methodology for IKI-220 (F1785) and its Major Metabolites in/on Cow Muscle, Kidney and Liver`, the results of the ILV performed by ADPEN Laboratories was provided, and the method was subsequently renumbered to P-3581. Average recoveries of flonicamid and its metabolites TFNA, TFNA-AM, OH-TFNA-AM and TFNG in bovine muscle were within the range of 82–110% with RSD of ≤ 17%, demonstrating good reproducibility.

Validation data for the methods described above are available from specific method validation studies or from residue studies where specific method validation recovery experiments were performed separately from routine sample analysis. These method recovery data, for plant and animal commodities are summarized in Table 41.

Table 41 Method recovery data of flonicamid and metabolites in plants and animal products

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
Plant Commodities								
Wheat Straw	Flonicamid	0.02	3	83–127	109.7	21.4	P-3561M	IB-2002-JLW-011-00
		0.04	3	107–128	114.7	10.1		
		0.1	3	105–122	114	7.5		
	TFNG	0.02	3	66–96	78.3	20		
		0.04	3	72–88	81.3	10.2		
		0.1	3	80–97	86.7	10.5		
	TFNA	0.02	3	84–109	92.3	15.6		
		0.04	3	63–95	83.7	21.4		
		0.1	3	86–110	97.3	12.4		
	TFNA-AM	0.02	3	61–80	70.3	13.5		
		0.04	3	63–71	66	6.6		
		0.1	3	77–86	80.3	6.1		
Peach	Flonicamid	0.01	3	95–113	105.3	8.8		
		0.02	3	105–108	106.3	1.4		
		0.05	3	102–106	104	1.9		
	TFNG	0.01	3	74–95	84.7	12.4		
		0.02	3	96–99	97	1.8		
		0.05	3	102–110	106	3.8		
	TFNA	0.01	3	92–97	94.7	2.7		
		0.02	3	94–108	102.3	7.2		
		0.05	3	105–107	106	0.9		
	TFNA-AM	0.01	3	88–96	93.3	4.9		
		0.02	3	97–103	100	3.0		
		0.05	3	97–98	97.7	0.6		
Potato Tuber	Flonicamid	0.01	3	92–108	98	8.9		
		0.02	3	98–109	103.7	5.3		
		0.05	3	82–106	95.7	12.9		
	TFNG	0.01	3	82–107	91.7	14.6		
		0.02	3	80–95	86.7	8.8		
		0.05	3	74–87	78.7	9.2		
	TFNA	0.01	3	102–115	108	6.1		
		0.02	3	107–122	114.7	6.5		
		0.05	3	93–106	98.3	6.9		
	TFNA-AM	0.01	3	80–98	92	11.3		
		0.02	3	83–89	86.3	3.5		
		0.05	3	73–85	79.3	7.6		
Apple	Flonicamid	0.05	1	84	NA	NA	P-3822	IB-2001-MDG-003
		0.1	4	90–100	94	4.5		
		0.2	1	102	NA	NA		
	TFNA-AM	0.05	1	76	NA	NA		
		0.1	4	73–80	75.7	3		
		0.2	1	90	NA	NA		
	TFNA	0.05	1	79	NA	NA		
		0.1	4	79–86	82.5	3.1		
		0.2	1	96	NA	NA		
	TFNG	0.05	1	69	NA	NA		
		0.1	4	67–77	72.5	5.3		
		0.2	1	90	NA	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%] Range	Mean	RSD	Method	Reference
Apple Juice	Flonicamid	0.25	5	90–113	96.2	9.6		01LJL045C
		0.5	1	125	NA	NA		
	TFNA-AM	0.25	5	72–86	78.6	5.4		
		0.5	1	97	NA	NA		
	TFNA	0.25	5	89–105	96.4	6.9		
		0.5	1	136	NA	NA		
	TFNG	0.25	5	65–115	86.4	18.1		
		0.5	1	133	NA	NA		
Apple Pomace	Flonicamid	0.01	2	91	NA	NA		IB-2001-MDG-003
		0.2	2	79–99	89	NA		
	TFNA-AM	0.01	2	75–84	79.5	NA		
		0.2	2	76–89	82.5	NA		
	TFNA	0.01	2	81–83	82	NA		
		0.2	2	100–112	106	NA		
	TFNG	0.01	2	73–107	90	NA		
		0.2	2	104–115	109.5	NA		
Pear	Flonicamid	0.01	1	92	NA	NA		IB-2001-MDG-003
		0.2	2	94–95	94.5	NA		
	TFNA-AM	0.01	1	75	NA	NA		
		0.2	2	74–86	80	NA		
	TFNA	0.01	1	97	NA	NA		
		0.2	2	93–94	93.5	NA		
	TFNG	0.01	1	65	NA	NA		
		0.2	2	73–88	80.5	NA		
Peach	Flonicamid	0.01	3	95–113	105.3	9.3		01LJL071C
		0.02	3	105–108	106.3	1.5		
		0.05	3	102–106	104	2		
	TFNA-AM	0.01	3	88–96	93.3	4.6		
		0.02	3	97–103	100	3		
		0.05	3	97–98	97.7	0.6		
	TFNA	0.01	3	92–97	94.7	2.5		
		0.02	3	97–103	102.3	7.4		
		0.05	3	105–107	106	1		
	TFNG	0.01	3	74–95	84.7	10.5		
		0.02	3	96–99	97	1.7		
		0.05	3	102–110	106	4		
Peach	Flonicamid	0.01	1	97	NA	NA		IB-2001-MDG-005
		0.05	2	107–111	109	NA		
		0.2	1	89	NA	NA		
		0.4	1	108	NA	NA		
	TFNA-AM	0.01	1	100	NA	NA		
		0.05	2	106–112	109	NA		
		0.2	1	82	NA	NA		
		0.4	1	102	NA	NA		
	TFNA	0.01	1	99	NA	NA		
		0.05	2	93–97	95	NA		
		0.2	1	97	NA	NA		
		0.4	1	93	NA	NA		
	TFNG	0.01	1	92	NA	NA		
		0.05	2	98–127	112.5	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.2	1	85	NA	NA		
		0.4	1	120	NA	NA		
Cherry	Flonicamid	0.01	2	95–115	105	NA	P-3822	IB-2001-MDG-005
		0.5	2	85–92	88.5	NA		
	TFNA-AM	0.01	2	92–114	103	NA		
		0.5	2	76–77	76.5	NA		
	TFNA	0.01	2	87–109	98	NA		
		0.5	2	93–106	99.5	NA		
	TFNG	0.01	2	99–119	109	NA		
		0.5	2	77–89	83	NA		
Plum	Flonicamid	0.05	1	85	NA	NA		IB-2001-MDG-005
		0.1	1	83	NA	NA		
		0.2	1	86	NA	NA		
		0.01	2	103–109	106	NA		
		1	1	102	NA	NA		
	TFNA-AM	0.05	1	85	NA	NA		
		0.1	1	74	NA	NA		
		0.2	1	82	NA	NA		
		0.01	2	103–107	105	NA		
		1	1	92	NA	NA		
	TFNA	0.05	1	78	NA	NA		
		0.1	1	84	NA	NA		
		0.2	1	88	NA	NA		
		0.01	2	110	110	NA		
		1	1	106	NA	NA		
	TFNG	0.05	1	77	NA	NA		
		0.1	1	74	NA	NA		
		0.2	1	74	NA	NA		
		0.01	2	96–111	103.5	NA		
		1	1	101	NA	NA		
Prune	Flonicamid	0.01	2	100–109	104.5	NA		IB-2001-MDG-005
		0.5	2	80–90	85	NA		
	TFNA-AM	0.01	2	92–98	95	NA		
		0.5	2	79–82	80.5	NA		
	TFNA	0.01	2	94–105	99.5	NA		
		0.5	2	73–82	77.5	NA		
	TFNG	0.01	2	98–100	99	NA		
		0.5	2	76–79	77.5	NA		
Pepper	Flonicamid	0.01	3	78–99	90	11		IB-2001-MDG-006
		0.05	1	65	NA	NA		
		0.1	1	77	NA	NA		
		0.2	1	87	NA	NA		
		0.5	1	81	NA	NA		
		1	1	104	NA	NA		
	TFNA-AM	0.01	3	68–79	74	6		
		0.05	1	79	NA	NA		
		0.1	1	82	NA	NA		
		0.2	1	64	NA	NA		
		0.5	1	67	NA	NA		
		1	1	67	NA	NA		
	TFNA	0.01	3	81–101	90	8		
		0.05	1	83	NA	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.1	1	94	NA	NA		
		0.2	1	89	NA	NA		
		1	1	96	NA	NA		
	TFNG	0.01	3	63–124	101	33		
		0.05	1	87	NA	NA		
		0.1	1	84	NA	NA		
		0.2	1	66	NA	NA		
		0.5	1	66	NA	NA		
		1	1	77	NA	NA		
Tomato	Flonicamid	0.01	1	131	NA	NA	P-3822	IB-2001-MDG-006/
		0.1	4	88–93	90	2.2		
		1.5	1	96	NA	NA		
	TFNA-AM	0.01	1	80	NA	NA		
		0.1	4	61–76	69.3	6.2		
		1.5	1	88	NA	NA		
	TFNA	0.01	1	136	NA	NA		
		0.1	4	80–98	89.3	7.4		
		1.5	1	90	NA	NA		
	TFNG	0.01	1	74	NA	NA		
		0.1	4	67–80	72.5	5.4		
		1.5	1	89	NA	NA		
Tomato	Flonicamid	0.05	1	87	NA	NA		CA147-A
		0.25	1	102	NA	NA		
		0.5	5	82–111	93.2	11.3		
	TFNA-AM	0.05	1	74	NA	NA		
		0.25	1	79	NA	NA		
		0.5	5	73–89	80.8	7.4		
	TFNA	0.05	1	84	NA	NA		
		0.25	1	102	NA	NA		
		0.5	5	89–122	101.8	13.8		
	TFNG	0.05	1	74	NA	NA		
		0.25	1	112	NA	NA		
		0.5	5	82–120	91.8	16		
Tomato Paste	Flonicamid	0.01	2	75–85	80	NA		CA137-S
		1	2	92–93	92.5	NA		
	TFNA-AM	0.01	2	77–84	80.5	NA		
		1	2	84–85	84.5	NA		
	TFNA	0.01	2	103–111	107	NA		
		1	2	100–101	100.5	NA		
	TFNG	0.01	2	90–92	91	NA		
		1	2	113–115	114	NA		
Tomato Puree	Flonicamid	0.01	2	81–85	83	NA		CA137-V
		0.5	2	97–100	98.5	NA		
	TFNA-AM	0.01	2	70–76	73	NA		
		0.5	2	82–85	83.5	NA		
	TFNA	0.01	2	66–71	68.5	NA		
		0.5	2	93–94	93.5	NA		
	TFNG	0.01	2	66–68	67	NA		
		0.5	2	101–109	105	NA		
Potato Tuber	Flonicamid	0.01	4	92–108	98.8	7.3	P-3822	01JRA/IB-2001-MDG-002
		0.02	3	98–109	103.7	5.5		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.05	5	77–106	89.8	12.2		
		0.1	5	78–102	89	10		
		0.4	1	100	NA	NA		
	TFNA-AM	0.01	4	80–98	89.3	10.1		
		0.02	3	80–89	85.3	4.7		
		0.05	5	63–85	75.2	8.3		
		0.1	5	64–97	79.2	13.8		
		0.4	1	86	NA	NA		
	TFNA	0.01	4	99–115	105.8	7		
		0.02	3	107–122	114.7	7.5		
		0.05	5	73–106	88.8	14		
		0.1	5	82–96	88.6	5.5		
		0.4	1	86	NA	NA		
	TFNG	0.01	4	68–107	85.8	16.1		
		0.02	3	80–95	86.7	7.6		
		0.05	5	69–87	77	6.8		
		0.1	5	64–80	72.2	6.1		
		0.4	1	91	NA	NA		
Potato Tuber	Flonicamid	0.25	5	92–112	103.8	8.4		01JFC667C
		0.5	5	92–124	106.6	15.6		
	TFNA-AM	0.25	5	75–105	86	11.9		
		0.5	5	71–102	88	14.4		
	TFNA	0.25	5	85–119	103.4	12		
		0.5	5	86–104	95	7.7		
	TFNG	0.25	5	78–102	91	9.2		
		0.5	5	86–104	95	7.7		
Potato Flakes	Flonicamid	0.01	2	78–86	82	NA		IB-2001-MDG-002
		0.2	1	96	NA	NA		
		0.5	2	63–89	76	NA		
	TFNA-AM	0.01	2	83–89	86	NA		
		0.2	1	87	NA	NA		
		0.5	2	76–80	78	NA		
	TFNA	0.01	2	91–102	96.5	NA		
		0.2	1	92	NA	NA		
		0.5	2	76–91	83.5	NA		
	TNFG	0.01	2	100–109	104.5	NA		
		0.2	1	87	NA	NA		
		0.5	2	90–95	92.5	NA		
Potato Wet Peel	Flonicamid	0.01	2	101–117	109	NA		IB-2001-MDG-002
		0.1	2	103–117	110	NA		
	TFNA-AM	0.01	2	86–98	92	NA		
		0.1	2	84–98	91	NA		
	TFNA	0.01	2	69–77	73	NA		
		0.1	2	97–113	105	NA		
	TFNG	0.01	2	64–85	74.5	NA		
		0.1	2	90–102	96	NA		
Potato Chips	Flonicamid	0.01	2	71–100	85.5	NA		IB-2001-MDG-002
		0.2	2	100–101	100.5	NA		
	TFNA-AM	0.01	2	83–86	84.5	NA		
		0.2	2	90–92	91	NA		
	TFNA	0.01	2	87–90	88.5	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.2	2	100–103	101.5	NA		
	TFNG	0.01	2	72–73	72.5	NA		
		0.2	2	110–117	103.5	NA		
Cottonseed	Flonicamid	0.02	2	113–118	115.5	NA		IB-2001-MDG-004/99AWC
		0.05	1	76	NA	NA		
		0.1	3	79–105	88.7	14.2		
		0.2	4	78–112	90.5	15		
		0.25	5	78–97	85	7.4		
		0.5	8	84–128	98.5	14.3		
		1	1	93	NA	NA		
	TFNA-AM	0.02	2	78–81	79.5	NA		
		0.05	1	69	NA	NA		
		0.1	3	72–102	84	15.9		
		0.2	4	74–93	80.5	9		
		0.25	5	65–86	74.6	8.7		
		0.5	8	66–104	83.5	14.6		
		1	1	82	NA	NA		
	TFNA	0.02	2	115–128	121.5	11.6		
		0.1	3	81–103	90.7	11.2		
		0.05	1	68	NA	NA		
		0.2	4	77–101	89.3	11.6		
		0.25	5	81–95	89.2	5.6		
		0.5	8	66–120	101	17.6		
		1	1	86	NA	NA		
	TFNG	0.02	2	97–100	98.5	NA		
		0.1	3	69–100	85	15.5		
		0.05	1	71	NA	NA		
		0.2	4	69–97	81.5	12.6		
		0.25	5	71–91	82	7.2		
		0.5	8	77–111	92.9	12.6		
		1	1	83	NA	NA		
Cotton Meal	Flonicamid	0.02	2	81–93	87	NA		IB-2001-MDG-004/99AWC
		0.25	5	73–122	97.6	18		
		0.5	1	99	NA	NA		
		2	2	81–88	84.5	NA		
	TFNA-AM	0.02	2	94–117	105.5	NA		
		0.25	5	67–78	73.2	5.4		
		0.5	1	72	NA	NA		
		2	2	78–79	78.5	NA		
	TFNA	0.02	2	79–119	99	NA		
		0.25	5	76–99	83.2	9.4		
		0.5	1	77	NA	NA		
		2	2	118–123	120.5	NA		
	TFNG	0.02	2	73–108	90.5	NA		
		0.25	5	69–85	79	6.8		
		0.5	1	81	NA	NA		
		2	2	117	117	NA		
Cotton Hulls	Flonicamid	0.02	2	70–90	80	NA		IB-2001-MDG-004/99AWC
		0.5	2	84–125	104.5	NA		
		1	2	100–101	100.5	NA		
		0.25	5	72–100	88	11		
	TFNA-AM	0.02	2	75–83	79	NA		
		0.5	2	80–100	90	NA		
		1	2	76–91	83.5	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.25	5	65–89	77	9.2		
	TFNA	0.02	2	71–92	81.5	NA		
		0.5	2	91–133	112	NA		
		1	2	119–126	122.5	NA		
		0.25	5	86–121	96.6	14.2		
	TFNG	0.02	2	87–95	91	NA		
		0.5	2	80–106	93	NA		
		1	2	99–118	108.5	NA		
		0.25	5	73–92	82.2	7		
Refined Oil	Flonicamid	0.02	2	81–101	91	NA		P06-2/IB-2001-MDG-004/99AWC
		0.25	5	86–125	101.6	15.2		
		0.5	5	82–110	93	14		
		1	1	92	NA	NA		
	TFNA-AM	0.02	2	82–87	84.5	NA		
		0.25	5	65–102	78.8	14.2		
		0.5	5	68–100	84.6	12.3		
		1	1	74	NA	NA		
	TFNA	0.02	2	73–84	78.5	NA		
		0.25	5	69–126	91.2	21.3		
		0.5	5	68–100	84.6	12.3		
		1	1	84	NA	NA		
	TFNG	0.02	2	90–94	92	NA		
		0.25	5	64–114	84.2	18.3		
		0.5	5	63–100	78.4	13.6		
		1	1	80	NA	NA		
Gin Trash	Flonicamid	0.02	2	69	69	NA		IB-2001-MDG-004
		0.5	1	80	NA	NA		
		5	1	91	NA	NA		
	TFNA-AM	0.02	2	114–119	116.5	NA		
		0.5	1	84	NA	NA		
		5	1	81	NA	NA		
	TFNA	0.02	2	85–95	90	NA		
		0.5	1	82	NA	NA		
		5	1	83	NA	NA		
	TNFG	0.02	2	110–111	110.5	NA		
		0.5	1	72	NA	NA		
		5	1	81	NA	NA		
Cucumber	Flonicamid	0.01	1	117	NA	NA		IB-2001-MDG-007
		0.1	2	84–94	89	NA		
	TFNA-AM	0.01	1	85	NA	NA		
		0.1	2	66–79	72.5	NA		
	TFNA	0.01	1	95	NA	NA		
		0.1	2	77–97	87	NA		
	TFNG	0.01	1	75	NA	NA		
		0.1	2	77–80	78.5	NA		
Summer Squash	Flonicamid	0.01	2	70–84	77	NA		IB-2001-MDG-007
		0.05	1	84	NA	NA		
		0.1	2	86–90	88	NA		
	TFNA-AM	0.01	2	74–87	80.5	NA		
		0.05	1	74	NA	NA		
		0.1	2	72–75	73.5	NA		
	TFNA	0.01	2	73–82	77.5	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.05	1	85	NA	NA		
		0.1	2	88–98	93	NA		
	TNFG	0.01	2	72–79	75.5	NA		
		0.05	1	77	NA	NA		
		0.1	2	69–81	75	NA		
Muskmelon	Flonicamid	0.01	2	87–92	89.5	NA		IB-2001-MDG-007
		0.1	1	93	NA	NA		
		0.2	2	95–107	101	NA		
		0.5	1	94	NA	NA		
	TFNA-AM	0.01	2	74–87	80.5	NA		
		0.1	1	71	NA	NA		
		0.2	2	83–84	83.5	NA		
		0.5	1	80	NA	NA		
	TFNA	0.01	2	90–95	92.5	NA		
		0.1	1	89	NA	NA		
		0.2	2	97–102	99.5	NA		
		0.5	1	99	NA	NA		
	TFNG	0.01	2	76–89	82.5	NA		
		0.1	1	70	NA	NA		
		0.2	2	87–88	87.5	NA		
		0.5	1	83	NA	NA		
Wheat Forage	Flonicamid	0.01	1	115	NA	NA		IB-2001-JLW-001/99WDN
		0.1	2	108–122	110	NA		
		0.2	1	93	NA	NA		
		0.25	1	79	NA	NA		
		0.5	5	72–100	92.2	11.5		
	TFNA-AM	0.01	1	85	NA	NA		
		0.1	2	76–92	84	NA		
		0.2	1	78	NA	NA		
		0.25	1	73	NA	NA		
		0.5	5	74–91	82	7.6		
	TFNA	0.01	1	94	NA	NA		
		0.1	2	85–102	93.5	NA		
		0.2	1	83	NA	NA		
		0.25	1	109	NA	NA		
		0.5	5	81–121	97.8	14.8		
	TFNG	0.01	1	75	NA	NA		
		0.1	2	73–102	87.5	NA		
		0.2	1	83	NA	NA		
		0.25	1	80	NA	NA		
		0.5	5	84–94	89.6	3.8		
Wheat Straw	Flonicamid	0.02	4	65–127	98.5	29.4		WCS/IB-2001-JLW-001/99WDN
		0.04	3	107–128	114.7	11.6		
		0.05	4	61–85	74.8	10		
		0.1	5	69–122	97.8	23.2		
		0.25	1	93	NA	NA		
		0.5	5	71–76	78.4	10		
	TFNA-AM	0.02	4	61–80	71.8	8.3		
		0.04	3	63–71	66	4.4		
		0.05	4	72–92	80.3	8.4		
		0.1	5	73–86	79	4.8		
		0.25	1	69	NA	NA		
		0.5	5	62–77	69	6.4		
	TFNA	0.02	4	77–109	88.5	14		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.04	3	63–95	83.7	18		
		0.05	4	73–99	86.8	11		
		0.1	5	81–110	92.8	11.1		
		0.25	1	83	NA	NA		
		0.5	5	71–87	82	6.3		
	TFNG	0.02	4	66–96	82.5	7.6		
		0.04	3	72–88	81.3	8.3		
		0.05	4	85–101	93.8	8		
		0.1	5	80–97	88.4	8		
		0.25	1	76	NA	NA		
		0.5	5	75–89	80	5.6		
Wheat Grain	Flonicamid	0.01	1	121	NA	NA		IB-2001-JLW-001/99WDN
		0.05	1	91	NA	NA		
		0.1	2	79–121	100	NA		
		0.25	5	76–103	85.2	10.6		
		0.5	1	89	NA	NA		
	TFNA-AM	0.01	1	67	NA	NA		
		0.05	1	83	NA	NA		
		0.1	2	79–80	79.5	NA		
		0.25	5	60–80	72	9.7		
		0.5	1	71	NA	NA		
	TFNA	0.01	1	75	NA	NA		
		0.05	1	115	NA	NA		
		0.1	2	96–97	96.5	NA		
		0.25	5	73–105	84.4	12		
		0.5	1	93	NA	NA		
	TFNG	0.01	1	74	NA	NA		
		0.05	1	89	NA	NA		
		0.1	2	83–89	86	NA		
		0.25	5	64–93	78.2	10.5		
		0.5	1	78	NA	NA		
Wheat Bran	Flonicamid	0.25	1	76	NA	NA		99WDN
		0.5	5	83–100	92.2	6.9		
	TFNA-AM	0.25	1	67	NA	NA		
		0.5	5	71–89	78	8.8		
	TFNA	0.25	1	63	NA	NA		
		0.5	5	76–99	88	9.9		
	TFNG	0.25	1	71	NA	NA		
		0.5	5	74–96	83.6	8		
Wheat Germ	Flonicamid	0.25	1	99	NA	NA		02JRA
		0.5	5	80–110	94	11.2		
	TFNA-AM	0.25	1	68	NA	NA		
		0.5	5	67–80	72.4	5.2		
	TFNA	0.25	1	66	NA	NA		
		0.5	5	70–92	78.8	9.7		
	TFNG	0.25	1	80	NA	NA		
		0.5	5	70–92	79.6	9.7		
Wheat Middlings	Flonicamid	0.25	5	76–114	85.6	16		99WDN
		0.5	5	77–89	82.6	6		
	TFNA-AM	0.25	5	80–94	87.8	6.8		
		0.5	5	68–97	84.6	10.7		
	TFNA	0.25	5	84–94	89.6	4.4		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.5	5	75–104	87.6	10.7		
	TFNG	0.25	5	91–105	98.8	5		
		0.5	5	87–118	97	12		
Turnip Tops	Flonicamid	0.01	1	83	NA	NA		IB-2001-JLW-001
		0.05	1	89	NA	NA		
	TFNA-AM	0.01	1	82	NA	NA		
		0.05	1	75	NA	NA		
	TFNA	0.01	1	77	NA	NA		
		0.05	1	73	NA	NA		
	TFNG	0.01	1	76	NA	NA		
		0.05	1	70	NA	NA		
Turnip Roots	Flonicamid	0.01	1	96	NA	NA		IB-2001-JLW-001
		0.1	1	95	NA	NA		
	TFNA-AM	0.01	1	88	NA	NA		
		0.1	1	74	NA	NA		
	TFNA	0.01	1	91	NA	NA		
		0.1	1	70	NA	NA		
	TFNG	0.01	1	94	NA	NA		
		0.1	1	72	NA	NA		
Leaf Lettuce	Flonicamid	0.01	1	79	NA	NA		01JWB
		0.1	1	73	NA	NA		
		0.5	1	84	NA	NA		
		10	1	81	NA	NA		
	TFNA-AM	0.01	1	68	NA	NA		
		0.1	1	74	NA	NA		
		0.5	1	70	NA	NA		
		10	1	73	NA	NA		
	TFNA	0.01	1	72	NA	NA		
		0.1	1	80	NA	NA		
		0.5	1	73	NA	NA		
		10	1	80	NA	NA		
	TFNG	0.01	1	72	NA	NA		
		0.1	1	76	NA	NA		
		0.5	1	75	NA	NA		
		10	1	79	NA	NA		
Head Lettuce	Flonicamid	0.01	2	88–115	101.5	NA		01JWB
		0.1	2	72–85	78.5	NA		
		0.5	2	88–91	89.5	NA		
		1	2	93–115	104	NA		
	TFNA-AM	0.01	2	90–101	95.5	NA		
		0.1	2	72–74	73	NA		
		0.5	2	79	79	NA		
		1	2	80–88	84	NA		
	TFNA	0.01	2	77–113	95	NA		
		0.1	2	73–83	78	NA		
		0.5	2	89–97	93	NA		
		1	2	94–110	102	NA		
	TFNG	0.01	2	122–125	123.5	NA		
		0.1	2	77–78	77.5	NA		
		0.5	2	82–88	85	NA		
		1	2	86–107	96.5	NA		
Celery	Flonicami	0.01	2	66–73	69.5	NA		01JWB

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
	d							
		0.05	1	72	NA	NA		
		0.1	2	75–108	91.5	NA		
		0.25	1	78	NA	NA		
		5	1	83	NA	NA		
	TFNA-AM	0.01	2	76–85	80.5	NA		
		0.05	1	71	NA	NA		
		0.1	2	66–88	77	NA		
		0.25	1	67	NA	NA		
		5	1	72	NA	NA		
	TFNA	0.01	2	76–78	77	NA		
		0.05	1	73	NA	NA		
		0.1	2	91–97	94	NA		
		0.25	1	0.25	NA	NA		
		5	1	77	NA	NA		
	TFNG	0.01	2	112	112	NA		
		0.05	1	81	NA	NA		
		0.1	2	75–98	86.5	NA		
		0.25	1	66	NA	NA		
		5	1	81	NA	NA		
Spinach	Flonicamid	0.01	2	97–100	98.5	NA		01JWB
		0.1	1	88	NA	NA		
		0.2	1	104	NA	NA		
		0.25	5	88–107	96.4	8		
		0.5	1	117	NA	NA		
		2	1	116	NA	NA		
	TFNA-AM	0.01	2	90–94	92	NA		
		0.1	1	66	NA	NA		
		0.2	1	99	NA	NA		
		0.25	5	73–93	83.2	8.2		
		0.5	1	98	NA	NA		
		2	1	88	NA	NA		
	TFNA	0.01	2	83–105	94	NA		
		0.1	1	83	NA	NA		
		0.2	1	108	NA	NA		
		0.25	5	77–117	90.6	16.1		
		0.5	1	118	NA	NA		
		2	1	99	NA	NA		
	TFNG	0.01	2	82–84	83	NA		
		0.1	1	73	NA	NA		
		0.2	1	111	NA	NA		
		0.25	5	68–99	88	13.2		
		0.5	1	101	NA	NA		
		2	1	92	NA	NA		
Broccoli	Flonicamid	0.01	2	74–81	77.5	NA		03WDN
		0.025	1	113	NA	NA		
		1	4	88–95	92	3.2		
	TFNA-AM	0.01	2	82–87	84.5	NA		
		0.025	1	112	NA	NA		
		1	4	77–92	85	6.3		
	TFNA	0.01	2	71–85	78	NA		
		0.025	1	119	NA	NA		
		1	4	78–106	96.3	13.2		
	TFNG	0.01	2	69–72	70.5	NA		
		0.025	1	97	NA	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%] Range	Mean	RSD	Method	Reference
		1	4	79–98	89.3	8		
Cabbage	Flonicamid	0.01	2	94	94	NA		03WDN
		0.025	1	123	NA	NA		
		0.1	1	123	NA	NA		
		1	2	109–128	118.5	NA		
		2	1	118	NA	NA		
	TFNA-AM	0.01	2	93–97	95	NA		
		0.025	1	108	NA	NA		
		0.1	1	95	NA	NA		
		1	2	86–105	95.5	NA		
		2	1	83	NA	NA		
	TFNA	0.01	2	88	88	NA		
		0.025	1	119	NA	NA		
		0.1	1	78	NA	NA		
		1	2	87–111	99	NA		
		2	1	97	NA	NA		
	TFNG	0.01	2	97–100	NA	NA		
		0.025	1	128	NA	NA		
		0.1	1	87	NA	NA		
		1	2	94–105	99.5	NA		
		2	1	90	NA	NA		
Mustard Greens	Flonicamid	0.5	1	117	NA	NA		03WDN
		2	1	100	NA	NA		
		16	1	99	NA	NA		
	TFNA-AM	0.5	1	90	NA	NA		
		2	1	89	NA	NA		
		16	1	92	NA	NA		
	TFNA	0.5	1	105	NA	NA		
		2	1	107	NA	NA		
		16	1	99	NA	NA		
	TFNG	0.5	1	102	NA	NA		
		2	1	99	NA	NA		
		16	1	106	NA	NA		
Lemon	Flonicamid	0.01	5	86–100	93	6	AGR/MOA/IKI220-1 v.1	ISK/IKI/06001
		0.1	5	76–87	84	5		
	TFNG	0.01	10	68–84	76	7		
		0.1	10	63–85	70	10		
	TFNA	0.01	5	78–89	85	5		
		0.1	5	70–80	77	6		
	TFNA-AM	0.01	10	68–86	78	7		
		0.1	10	56–94	72	15		
Oilseed rape	Flonicamid	0.01	5	77–106	90	13		
		0.1	5	75–96	83	5		
	TFNG	0.01	5	58–84	75	13		
		0.1	5	74–100	86	12		
	TFNA	0.01	5	74–90	81	8		
		0.1	5	67–91	77	13		
	TFNA-AM	0.01	5	67–98	88	14		
		0.1	5	74–101	87	13		
Wheat grain	Flonicamid	0.01	5	102–117	109	5		
		0.1	5	85–110	93	11		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.5	5	84–101	91	8		
	TFNG	0.01	5	85–113	101	12		
		0.1	5	81–113	94	13		
		1.0	5	74–89	83	7		
	TFNA	0.01	5	88–95	92	3		
		0.1	5	87–92	89	3		
	TFNA-AM	0.01	5	94–114	108	8		
		0.1	5	83–116	96	13		
Plum	Flonicamid	0.01	5	89–121	105	12		
		0.1	5	80–102	94	9		
		0.5	5	82–93	89	5		
	TFNG	0.01	5	83–119	103	16		
		0.1	5	77–98	85	10		
	TFNA	0.01	5	95–113	102	7		
		0.1	5	76–105	88	13		
	TFNA-AM	0.01	5	101–113	107	5		
		0.1	5	77–91	82	7		
Prune	Flonicamid	0.01	5	87–103	92	7		
		0.1	5	81–93	89	6		
	TFNG	0.01	5	78–85	81	4		
		0.1	5	73–85	82	6		
	TFNA	0.01	5	75–87	80	7		
		0.1	5	80–97	90	7		
	TFNA-AM	0.01	5	69–75	72	4		
		0.1	5	76–86	83	5		
Animal Commodities								
Milk	Flonicamid	0.01	5	72–81	76	6	842993	
		0.10	5	74–82	79	4		
	TFNA	0.01	5	79–105	94	11		
		0.10	5	80–93	88	6		
	TFNA-AM	0.01	5	78–91	83	6		
		0.10	5	86–97	92	5		
	OH–TFNA-AM	0.01	5	74–83	80	5		
		0.10	5	74–86	82	7		
	TFNG	0.01	5	79–107	95	12		
		0.10	5	71–79	76	5		
Bovine Muscle	Flonicamid	0.01	5	102–108	107	2	844743	
		0.10	5	84–108	92	11		
	TFNA	0.01	5	85–108	98	8		
		0.10	5	95–108	101	6		
	TFNA-AM	0.01	5	95–101	98	3		
		0.10	5	86–106	92	10		
	OH-TFNA-AM	0.01	5	83–100	94	7		
		0.10	5	87–99	94	6		
	TFNG	0.01	5	86–100	95	6		
		0.10	5	95–106	100	5		
Bovine Liver	Flonicamid	0.01	5	72–80	78	4		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		0.10	5	73–78	75	2		
	TFNA	0.01	5	82–107	91	12		
		0.10	5	82–88	86	3		
	TFNA-AM	0.01	5	79–87	82	4		
		0.10	5	76–82	79	3		
	OH-TFNA-AM	0.01	5	80–94	86	6		
		0.10	5	86–106	95	10		
	TFNG	0.01	5	85–92	87	8		
		0.10	5	94–107	100	6		
Bovine Kidney	Flonicamid	0.01	5	72–101	82	14		
		0.10	5	78–97	87	10		
	TFNA	0.01	5	76–90	83	7		
		0.10	5	89–96	92	4		
	TFNA-AM	0.01	5	90–105	101	6		
		0.10	5	99–106	103	3		
	OH-TFNA-AM	0.01	5	88–105	99	7		
		0.10	5	100–105	102	2		
	TFNG	0.01	5	78–107	91	13		
		0.10	5	88–96	92	4		
Bovine Fat	Flonicamid	0.01	5	108–110	109	1		
		0.10	5	104–108	106	2		
	TFNA	0.01	5	72–108	82	18		
		0.10	5	73–79	75	2		
	TFNA-AM	0.01	5	88–108	96	8		
		0.10	5	89–96	92	3		
	OH-TFNA-AM	0.01	5	71–73	72	1		
		0.10	5	71–83	76	6		
	TFNG	0.01	5	88–99	93	4		
		0.10	5	99–108	104	4		
Poultry Egg	Flonicamid	0.01	5	81–88	85	3		
		0.10	5	92–101	96	4		
	TFNA	0.01	5	70–76	72	3		
		0.10	5	81–98	90	8		
	TFNA-AM	0.01	5	78–86	82	4		
		0.10	5	87–99	93	6		
	OH-TFNA-AM	0.01	5	81–91	87	5		
		0.10	5	94–106	99	5		
	TFNG	0.01	5	74–85	78	5		
		0.10	5	94–110	100	6		
Poultry Muscle	Flonicamid	0.01	2	109–110	110	NA		
		0.10	2	105	105	NA		
		1.0	2	98–99	98	NA		
	TFNA	0.01	2	88	88	NA		
		0.10	2	88–93	90	NA		
		1.0	2	97–101	99	NA		
	TFNA-AM	0.01	2	108–109	108	NA		
		0.10	2	103–106	104	NA		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%]			Method	Reference
				Range	Mean	RSD		
		1.0	2	98–99	98	NA		
	OH-TFNA-AM	0.01	2	101–105	103	NA		
		0.10	2	104–110	107	NA		
		1.0	2	100–102	101	NA		
	TFNG	0.01	2	89–107	98	NA		
		0.10	2	98–105	101	NA		
		1.0	2	97–98	98	NA		
Poultry Liver	Flonicamid	0.01	2	106–107	106	NA		
		0.10	2	95–97	96	NA		
		1.0	2	96–100	98	NA		
	TFNA	0.01	2	80–94	88	NA		
		0.10	2	90–94	92	NA		
		1.0	2	95–97	96	NA		
	TFNA-AM	0.01	2	107–109	108	NA		
		0.10	2	97–99	98	NA		
		1.0	2	97–98	98	NA		
	OH-TFNA-AM	0.01	2	80–89	84	NA		
		0.10	2	95–97	96	NA		
		1.0	2	92–94	93	NA		
	TFNG	0.01	2	83–90	86	NA		
		0.10	2	93–109	101	NA		
		1.0	2	99–101	100	NA		
Poultry Fat	Flonicamid	0.01	2	107–108	108	NA		
		0.10	2	107–108	107	NA		
		1.0	2	104–110	107	NA		
	TFNA	0.01	2	72–89	81	NA		
		0.10	2	70–76	73	NA		
		1.0	2	72–74	73	NA		
	TFNA-AM	0.01	2	92–104	98	NA		
		0.10	2	96–99	97	NA		
		1.0	2	95–101	98	NA		
	OH-TFNA-AM	0.01	2	75–78	76	NA		
		0.10	2	74–81	78	NA		
		1.0	2	83	83	NA		
	TFNG	0.01	2	100–105	102	NA		
		0.10	2	106–108	107	NA		
		1.0	2	97–105	101	NA		
Milk	Flonicamid	0.01	5	88–92	90	2	AGR/MOA/IKI-5	
		0.10	5	89–92	91	2		
	TFNA-AM	0.01	5	86–90	88	2		
		0.10	5	88–92	90	2		
Eggs	Flonicamid	0.01	5	87–93	90	3		
		0.10	5	92–98	95	3		
	TFNA-AM	0.01	5	91–93	92	2		
		0.10	5	93–96	94	1		
Bovine Muscle	Flonicami	0.01	5	79–87	84	4		

Matrix	Compound	Fortification Levels [mg/kg]	No. of samples	Recovery [%] Range	Mean	RSD	Method	Reference
	d							
		0.10	5	81–88	84	4		
	TFNA-AM	0.01	5	83–88	85	2		
		0.10	5	85–91	87	3		
Bovine Fat	Flonicamid	0.01	5	91–93	92	1		
		0.10	5	88–95	91	3		
	TFNA-AM	0.01	5	91–94	92	1		
		0.10	5	91–96	93	2		
Bovine Liver	Flonicamid	0.01	5	79–84	82	3		
		0.10	5	77–84	81	3		
	TFNA-AM	0.01	5	81–88	83	4		
		0.10	5	80–86	83	3		

Stability of residues in stored analytical samples

Information was received on the freezer storage stability of flonicamid and its metabolites in plant commodities. The storage stability of flonicamid and its metabolites TFNA, TFNA-AM and TFNG are described as follows. The results are shown in Table 42.

Wheat (grain, forage, straw, bran, middling, germ), cottonseed (seed, hulls, meal, refined oil), spinach, potato tuber, apple juice and tomato

Report: P-3570

Study No. 178CSS02R1

Method: P-3561

Description: Untreated control samples were fortified with flonicamid and its metabolites TFNA, TFNA-AM and TFNG at a concentration of 0.5 mg/kg per analyte and then frozen below –17 °C. Samples were analysed immediately after fortification (0 day) and after storage intervals up to 2 years (23 months). At each interval, three stored samples were analysed, with one or more procedural recovery samples (control samples spiked just before analysis at 0.5 mg/kg).

Table 42 Storage Stability of Flonicamid, TFNA, TFNA-AM and TFNG in wheat, cotton, potato, apple and tomato

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
Wheat Grain					
0	0.50, 0.47, 0.53	0.5	100%	76	
3	0.42, 0.37, 0.40	0.4	80%	89	
6	0.39, 0.38, 0.46	0.41	82%	103	
9	0.40, 0.40, 0.45	0.42	84%	86	
15	0.50, 0.49, 0.44	0.48	96%	79	
23	0.48, 0.52, 0.54	0.51	102%	82	
Wheat Forage					
0	0.57, 0.52, 0.53	0.54	100%	79	

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
3	0.52, 0.48, 0.53	0.51	94%	96	
6	0.22, 0.29, 0.68	0.40	74%	72	
9	0.49, 0.48, 0.52	0.50	93%	98	
15	0.43, 0.41, 0.46	0.43	80%	95	
23	0.48, 0.57, 0.55	0.53	98%	100	
Wheat Straw					
0	0.55, 0.58, 0.53	0.55	100%	93	
3	0.52, 0.51, 0.48	0.5	91%	96	
6	0.44, 0.46, 0.40	0.44	80%	75	
9	0.48, 0.50, 0.46	0.48	87%	75	
15	0.44, 0.47, 0.46	0.46	84%	71	
23	0.52, 0.53, 0.53	0.52	95%	75	
Wheat Bran					
0	0.53, 0.38, 0.48	0.46	100%	76	
3	0.49, 0.44, 0.47	0.47	102%	83	
6	0.37, 0.40, 0.35	0.37	80%	91	
9	0.43, 0.45, 0.40	0.43	93%	100	
15	0.47, 0.45, 0.49	0.47	102%	98	
23	0.51, 0.57, 0.53	0.54	117%	89	
Wheat Germ					
0	0.55, 0.50, 0.54	0.53	100%	99	
3	0.59, 0.51, 0.42	0.51	96%	110	
6	0.52, 0.46, 0.53	0.50	94%	80	
9	0.47, 0.45, 0.45	0.46	87%	99	
15	0.39, 0.47, 0.37	0.41	77%	91	
23	0.42, 0.41, 0.44	0.42	79%	90	
Wheat Middling					
0	0.30, 0.34, 0.51	0.38	100%	79	
3	0.24, 0.39, 0.43	0.35	92%	81	
6	0.58, 0.36, 0.58	0.51	134%	76, 89	83
9	0.54, 0.50, 0.53	0.53	139%	77, 89	83
15	0.51, 0.53, 0.60	0.55	145%	82, 77	80
23	0.45, 0.48, 0.50	0.48	126%	114, 77	96
Spinach					
0	0.48, 0.48, 0.50	0.49	100%	89	
3	0.43, 0.44, 0.46	0.44	90%	117	
6	0.62, 0.65, 0.59	0.62	127%	101	
9	0.46, 0.52, 0.47	0.48	98%	88	
15	0.49, 0.41, 0.41	0.44	90%	94	
23	0.45, 0.46, 0.44	0.45	92%	107	
Cottonseed					
0	0.56, 0.55, 0.59	0.57	100%	78	
3	0.46, 0.48, 0.54	0.43	75%	96	
6	0.42, 0.39, 0.38	0.40	70%	86, 106	96
9	0.35, 0.38, 0.38	0.37	65%	97, 105	101
15	0.48, 0.45, 0.47	0.47	82%	80, 84	82
23	0.43, 0.48, 0.53	0.48	84%	84, 90	87
Cotton Hulls					
0	0.30, 0.53, 0.56	0.46	100%	100	
3	0.51, 0.53, 0.56	0.53	92%	125	

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
6	0.56, 0.55, 0.57	0.56	122%	72	
9	0.55, 0.45, 0.54	0.51	111%	93	
15	0.55, 0.58, 0.62	0.58	126%	82	
23	0.61, 0.36, 0.56	0.51	111%	93	
Cotton Meal					
0	0.40, 0.42, 0.41	0.41	100%	100	
3	0.59, 0.50, 0.48	0.52	127%	99	
6	0.58, 0.58, 0.46	0.54	132%	73	
9	0.45, 0.34, 0.44	0.41	100%	103	
15	0.49, 0.48, 0.50	0.49	120%	90	
23	0.44, 0.44, 0.40	0.43	86%	122	
Cotton Refined oil					
0	0.48, 0.45, 0.47	0.47	100%	125	
3	0.49, 0.43, 0.47	0.46	98%	83	
6	0.59, 0.53, 0.47	0.53	113%	103, 110	107
9	0.54, 0.53, 0.55	0.54	115%	86, 83	85
15	0.52, 0.49, 0.54	0.52	111%	90, 92	86
23	0.41, 0.47, 0.42	0.43	91%	104, 107	106
Apple juice					
0	0.52, 0.54, 0.47	0.51	100%	113	
3	0.48, 0.47, 0.43	0.48	75%	125	
6	0.41, 0.47, 0.50	0.46	90%	91	
9	0.46, 0.45, 0.47	0.46	90%	95	
15	0.47, 0.48, 0.56	0.50	98%	92	
23	0.58, 0.55, 0.48	0.54	106%	90	
Tomato					
0	0.52, 0.55, 0.57	0.55	100%	102	
3	0.56, 0.54, 0.52	0.54	98%	111	
6	0.48, 0.43, 0.54	0.48	87%	91	
9	0.45, 0.46, 0.51	0.47	85%	96	
15	0.55, 0.52, 0.51	0.53	96%	82	
23	0.59, 0.55, 0.61	0.58	105%	86	
Potato Tuber					
0	0.46, 0.41, 0.45	0.44	100%	111	
3	0.41, 0.44, 0.44	0.43	79%	124	
6	0.38, 0.40, 0.40	0.39	89%	105, 123	114
9	0.51, 0.50, 0.62	0.54	123%	92 ,97	95
15	0.45, 0.52, 0.42	0.46	105%	99, 97	98
23	0.56, 0.53, 0.49	0.53	120%	112, 92	102
TFNG					
Wheat grain					
0	0.51, 0.49, 0.58	0.53	100%	64	
3	0.46, 0.48, 0.49	0.48	91%	78	
6	0.39, 0.43, 0.46	0.43	81%	93	
9	0.45, 0.51, 0.53	0.49	92%	76	
15	0.58, 0.53, 0.50	0.54	102%	76	
23	0.50, 0.52, 0.50	0.51	96%	82	
Wheat Forage					
0	0.54, 0.52, 0.52	0.53	100%	80	
3	0.53, 0.48, 0.51	0.51	96%	91	
6	0.42, 0.41, 0.41	0.41	77%	91	

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
9	0.56, 0.59, 0.56	0.57	108%	94	
15	0.48, 0.47, 0.47	0.47	89%	88	
23	0.49, 0.56, 0.56	0.54	102%	84	
Wheat Straw					
0	0.63, 0.65, 0.63	0.63	100%	76	
3	0.56, 0.53, 0.53	0.54	114%	75	
6	0.46, 0.49, 0.42	0.46	82%	89	
9	0.47, 0.41, 0.48	0.45	90%	79	
15	0.49, 0.53, 0.50	0.51	100%	81	
23	0.49, 0.55, 0.56	0.53	111%	76	
Wheat Bran					
0	0.53, 0.38, 0.47	0.46	100%	71	
3	0.52, 0.45, 0.46	0.47	102%	74	
6	0.36, 0.41, 0.37	0.38	83%	96	
9	0.45, 0.53, 0.48	0.48	104%	80	
15	0.48, 0.47, 0.50	0.48	104%	85	
23	0.50, 0.46, 0.48	0.51	111%	81	
Wheat Germ					
0	0.52, 0.54, 0.53	0.53	100%	80	
3	0.63, 0.58, 0.59	0.6	113%	92	
6	0.50, 0.47, 0.47	0.48	91%	70	
9	0.56, 0.54, 0.57	0.56	106%	78	
15	0.44, 0.49, 0.51	0.48	91%	87	
23	0.53, 0.60, 0.62	0.58	109%	71	
Wheat Middling					
0	0.48, 0.52, 0.49	0.5	100%	91	
3	0.55, 0.57, 0.55	0.56	112%	90	
6	0.58, 0.40, 0.59	0.52	104%	100, 96	98
9	0.49, 0.56, 0.47	0.51	102%	99, 87	93
15	0.53, 0.55, 0.60	0.56	112%	105, 118	112
23	0.56, 0.53, 0.56	0.55	110%	99, 94	97
Spinach					
0	0.50, 0.50, 0.54	0.52	100%	81	
3	0.43, 0.45, 0.44	0.44	85%	101	
6	0.40, 0.43, 0.39	0.41	79%	96	
9	0.44, 0.48, 0.47	0.46	88%	68	
15	0.55, 0.47, 0.47	0.5	96%	96	
23	0.46, 0.47, 0.46	0.46	88%	99	
Cottonseed					
0	0.56, 0.57, 0.56	0.56	100%	71	
3	0.48, 0.53, 0.53	0.51	91%	87	
6	0.43, 0.38, 0.37	0.39	70%	84, 92	88
9	0.46, 0.42, 0.43	0.44	79%	83, 111	97
15	0.50, 0.47, 0.47	0.48	86%	81, 77	79
23	0.48, 0.50, 0.54	0.51	91%	91, 95	93
Cotton Hulls					
0	0.57, 0.61, 0.61	0.6	100%	92	
3	0.65, 0.63, 0.60	0.63	105%	106	
6	0.46, 0.48, 0.48	0.47	78%	84	
9	0.39, 0.54, 0.41	0.45	75%	80	
15	0.54, 0.55, 0.58	0.56	93%	73	
23	0.59, 0.60, 0.55	0.58	97%	82	
Cotton Meal					
0	0.43, 0.45, 0.43	0.43	100%	85	
3	0.56, 0.54, 0.54	0.55	128%	81	
6	0.46, 0.50, 0.44	0.47	109%	75	
9	0.49, 0.42, 0.50	0.47	158%	69	

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
15	0.48, 0.50, 0.52	0.5	116%	84	
23	0.55, 0.52, 0.52	0.53	123%	82	
Cotton Refined Oil					
0	0.42, 0.37, 0.43	0.41	100%	83	
3	0.39, 0.37, 0.42	0.39	151%	63	
6	0.47, 0.60, 0.49	0.52	127%	79, 73	76
9	0.53, 0.56, 0.58	0.56	137%	64, 76	70
15	0.53, 0.48, 0.54	0.52	127%	81, 80	81
23	0.44, 0.51, 0.45	0.47	115%	114, 100	107
Apple Juice					
0	0.55, 0.51, 0.48	0.51	100%	115	
3	0.47, 0.48, 0.49	0.48	71%	133	
6	0.42, 0.46, 0.46	0.45	88%	82	
9	0.41, 0.47, 0.50	0.46	90%	82	
15	0.50, 0.49, 0.57	0.52	102%	88	
23	0.59, 0.62, 0.50	0.57	112%	65	
Tomato					
0	0.59, 0.49, 0.52	0.53	100%	112	
3	0.51, 0.51, 0.51	0.51	96%	120	
6	0.46, 0.45, 0.44	0.45	85%	87	
9	0.48, 0.50, 0.51	0.5	94%	82	
15	0.51, 0.51, 0.49	0.5	94%	88	
23	0.54, 0.60, 0.64	0.59	111%	82	
Potato Tuber					
0	0.48, 0.43, 0.49	0.47	100%	90	
3	0.45, 0.48, 0.46	0.46	98%	104	
6	0.42, 0.40, 0.41	0.41	87%	97, 102	100
9	0.58, 0.48, 0.60	0.53	113%	78, 90	84
15	0.48, 0.54, 0.48	0.5	106%	88, 86	87
23	0.56, 0.54, 0.55	0.55	117%	102, 93	98
TFNA					
Wheat Grain					
0	0.43, 0.47, 0.48	0.46	100%	73	
3	0.47, 0.42, 0.44	0.44	96%	93	
6	0.37, 0.37, 0.42	0.39	85%	105	
9	0.46, 0.51, 0.54	0.5	109%	80	
15	0.53, 0.52, 0.47	0.51	111%	83	
23	0.52, 0.48, 0.53	0.51	111%	81	
Wheat Forage					
0	0.53, 0.48, 0.48	0.49	100%	79	
3	0.52, 0.45, 0.46	0.48	98%	96	
6	0.49, 0.44, 0.39	0.44	90%	72	
9	0.50, 0.53, 0.56	0.53	108%	98	
15	0.40, 0.39, 0.44	0.47	96%	95	
23	0.47, 0.67, 0.62	0.59	120%	100	
Wheat Straw					
0	0.66, 0.67, 0.66	0.66	100%	83	
3	0.57, 0.56, 0.54	0.56	102%	83	
6	0.38, 0.40, 0.39	0.39	70%	84	
9	0.44, 0.42, 0.45	0.44	77%	87	
15	0.45, 0.49, 0.45	0.46	82%	85	
23	0.49, 0.49, 0.50	0.49	105%	71	
Wheat Bran					
0	0.48, 0.40, 0.47	0.45	100%	63	
3	0.47, 0.48, 0.29	0.41	91%	80	
6	0.37, 0.43, 0.36	0.39	87%	99	
9	0.46, 0.53, 0.46	0.48	107%	89	

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
15	0.46, 0.44, 0.46	0.45	100%	96	
23	0.44, 0.53, 0.46	0.48	107%	76	
Wheat Germ					
0	0.59, 0.68, 0.65	0.64	100%	66	
3	0.76, 0.62, 0.72	0.7	109%	71	
6	0.62, 0.54, 0.57	0.58	91%	70	
9	0.50, 0.49, 0.52	0.5	78%	86	
15	0.41, 0.44, 0.46	0.44	69%	92	
23	0.53, 0.53, 0.52	0.53	83%	75	
Wheat Middling					
0	0.49, 0.52, 0.49	0.5	100%	91	
3	0.52, 0.57, 0.54	0.55	110%	89	
6	0.60, 0.38, 0.62	0.53	106%	84, 88	86
9	0.47, 0.58, 0.52	0.52	104%	86, 75	81
15	0.50, 0.53, 0.54	0.52	104%	93, 104	99
23	0.48, 0.51, 0.57	0.52	104%	94, 82	88
Spinach					
0	0.51, 0.48, 0.51	0.5	100%	94	
3	0.42, 0.45, 0.44	0.43	86%	118	
6	0.40, 0.49, 0.45	0.44	88%	85	
9	0.47, 0.54, 0.50	0.5	100%	80	
15	0.51, 0.42, 0.42	0.45	90%	117	
23	0.45, 0.54, 0.51	0.5	100%	77	
Cottonseed					
0	0.56, 0.57, 0.57	0.56	100%	92	
3	0.45, 0.47, 0.51	0.48	86%	108	
6	0.42, 0.36, 0.35	0.38	68%	95, 108	102
9	0.41, 0.39, 0.40	0.4	71%	92, 120	106
15	0.48, 0.47, 0.44	0.46	82%	86, 92	89
23	0.41, 0.46, 0.51	0.46	82%	81, 95	88
Cotton Hulls					
0	0.55, 0.58, 0.57	0.57	100%	121	
3	0.62, 0.59, 0.59	0.6	79%	133	
6	0.53, 0.51, 0.52	0.52	91%	97	
9	0.42, 0.56, 0.40	0.46	81%	90	
15	0.55, 0.55, 0.60	0.57	100%	89	
23	0.60, 0.61, 0.61	0.61	107%	86	
Cotton Meal					
0	0.43, 0.40, 0.48	0.43	100%	76	
3	0.60, 0.57, 0.55	0.58	135%	77	
6	0.56, 0.59, 0.52	0.56	130%	84	
9	0.44, 0.43, 0.44	0.44	102%	77	
15	0.49, 0.51, 0.50	0.5	116%	99	
23	0.50, 0.54, 0.50	0.51	119%	80	
Cottonseed Refined Oil					
0	0.46, 0.45, 0.45	0.45	100%	93	
3	0.41, 0.46, 0.39	0.42	137%	68	
6	0.51, 0.64, 0.55	0.57	127%	82, 77	80
9	0.51, 0.54, 0.58	0.54	120%	69, 89	79
15	0.49, 0.48, 0.53	0.5	111%	86, 89	88
23	0.29, 0.35, 0.42	0.35	78%	126, 100	113
Apple Juice					
0	0.61, 0.56, 0.51	0.56	100%	105	
3	0.49, 0.55, 0.52	0.52	68%	136	
6	0.40, 0.43, 0.46	0.43	77%	101	
9	0.44, 0.44, 0.50	0.46	82%	90	
15	0.45, 0.46, 0.54	0.48	86%	97	

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
23	0.55, 0.53, 0.47	0.52	93%	89	
Tomato					
0	0.53, 0.49, 0.57	0.53	100%	102	
3	0.51, 0.50, 0.47	0.49	76%	122	
6	0.43, 0.43, 0.41	0.43	81%	110	
9	0.48, 0.50, 0.55	0.51	96%	89	
15	0.48, 0.47, 0.46	0.47	89%	95	
23	0.54, 0.54, 0.62	0.57	108%	93	
Potato Tuber					
0	0.46, 0.39, 0.47	0.44	100%	105	
3	0.41, 0.43, 0.44	0.43	79%	124	
6	0.43, 0.39, 0.47	0.43	98%	104, 98	101
9	0.50, 0.47, 0.58	0.52	118%	85, 109	97
15	0.46, 0.49, 0.44	0.46	105%	104, 96	100
23	0.48, 0.50, 0.49	0.49	111%	119, 96	108
TFNA-AM					
Wheat Grain					
0	0.44, 0.48, 0.54	0.48	100%	60	
3	0.40, 0.40, 0.45	0.42	88%	71	
6	0.38, 0.37, 0.44	0.40	83%	80	
9	0.43, 0.43, 0.50	0.45	94%	79	
15	0.55, 0.50, 0.47	0.51	169%	63	
23	0.49, 0.48, 0.50	0.49	102%	78	
Wheat Forage					
0	0.55, 0.51, 0.55	0.54	100%	73	
3	0.51, 0.47, 0.50	0.49	91%	82	
6	0.43, 0.42, 0.39	0.41	76%	91	
9	0.51, 0.53, 0.54	0.52	96%	88	
15	0.45, 0.45, 0.46	0.45	83%	75	
23	0.49, 0.53, 0.55	0.52	96%	74	
Wheat Straw					
0	0.68, 0.70, 0.67	0.68	100%	69	
3	0.60, 0.57, 0.56	0.58	85%	72	
6	0.42, 0.45, 0.39	0.42	62%	77	
9	0.44, 0.44, 0.45	0.44	65%	71	
15	0.46, 0.50, 0.47	0.48	112%	63	
23	0.51, 0.52, 0.51	0.51	121%	62	
Wheat Bran					
0	0.53, 0.42, 0.48	0.48	100%	67	
3	0.50, 0.46, 0.43	0.46	96%	71	
6	0.37, 0.42, 0.38	0.39	81%	89	
9	0.44, 0.47, 0.43	0.45	94%	86	
15	0.45, 0.48, 0.48	0.47	98%	73	
23	0.50, 0.50, 0.48	0.5	104%	71	
Wheat Germ					
0	0.54, 0.55, 0.52	0.54	100%	68	
3	0.62, 0.57, 0.59	0.59	109%	80	
6	0.46, 0.44, 0.45	0.45	123%	68	
9	0.54, 0.52, 0.52	0.53	98%	74	
15	0.40, 0.46, 0.49	0.45	83%	73	
23	0.53, 0.57, 0.56	0.55	152%	67	
Wheat Middling					
0	0.47, 0.53, 0.51	0.5	100%	94	
3	0.53, 0.58, 0.50	0.54	108%	89	
6	0.60, 0.40, 0.58	0.53	106%	93, 87	90
9	0.54, 0.55, 0.57	0.55	110%	81, 82	82
15	0.52, 0.54, 0.57	0.54	108%	91, 97	94

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
23	0.51, 0.54, 0.56	0.54	108%	80, 68	74
Spinach					
0	0.53, 0.53, 0.54	0.53	100%	79	
3	0.43, 0.46, 0.43	0.44	83%	98	
6	0.40, 0.44, 0.37	0.4	75%	93	
9	0.44, 0.49, 0.50	0.48	91%	73	
15	0.55, 0.46, 0.46	0.49	92%	81	
23	0.42, 0.44, 0.47	0.44	83%	90	
Cottonseed					
0	0.56, 0.57, 0.56	0.56	100%	78	
3	0.48, 0.48, 0.52	0.49	88%	96	
6	0.42, 0.38, 0.36	0.38	68%	86, 106	96
9	0.42, 0.39, 0.41	0.41	73%	97, 105	101
15	0.46, 0.43, 0.45	0.45	80%	80, 84	82
23	0.41, 0.47, 0.52	0.47	84%	84, 90	87
Cotton Hulls					
0	0.55, 0.57, 0.59	0.57	100%	89	
3	0.63, 0.58, 0.59	0.6	105%	100	
6	0.46, 0.49, 0.46	0.47	82%	82	
9	0.45, 0.56, 0.43	0.48	84%	76	
15	0.53, 0.57, 0.60	0.57	154%	65	
23	0.65, 0.63, 0.59	0.62	109%	72	
Cotton Meal					
0	0.41, 0.44, 0.42	0.42	100%	78	
3	0.56, 0.54, 0.55	0.55	131%	72	
6	0.49, 0.50, 0.46	0.48	114%	78	
9	0.47, 0.44, 0.45	0.45	160%	67	
15	0.47, 0.48, 0.51	0.49	117%	75	
23	0.55, 0.52, 0.51	0.53	186%	68	
Cottonseed Refined Oil					
0	0.40, 0.39, 0.42	0.4	100%	102	
3	0.41, 0.37, 0.41	0.4	100%	76	
6	0.52, 0.66, 0.57	0.58	145%	77, 71	74
9	0.55, 0.57, 0.59	0.57	207%	65 73	69
15	0.51, 0.46, 0.51	0.49	178%	70, 68	69
23	0.35, 0.48, 0.45	0.43	108%	80, 77	79
Apple Juice					
0	0.55, 0.53, 0.50	0.53	100%	80	
3	0.47, 0.51, 0.49	0.49	92%	97	
6	0.40, 0.44, 0.44	0.42	79%	86	
9	0.44, 0.49, 0.52	0.49	92%	80	
15	0.49, 0.52, 0.57	0.53	100%	75	
23	0.57, 0.61, 0.47	0.55	104%	72	
Tomato					
0	0.60, 0.52, 0.54	0.55	100%	79	
3	0.53, 0.50, 0.53	0.52	95%	89	
6	0.46, 0.42, 0.41	0.43	78%	88	
9	0.51, 0.52, 0.56	0.53	96%	79	
15	0.53, 0.51, 0.51	0.52	95%	73	
23	0.54, 0.57, 0.67	0.59	107%	75	
Potato Tuber					
0	0.48, 0.43, 0.48	0.46	100%	89	
3	0.43, 0.45, 0.45	0.44	96%	101	
6	0.40, 0.38, 0.38	0.39	85%	105, 102	104
9	0.48, 0.48, 0.57	0.51	111%	78, 91	85
15	0.47, 0.53, 0.44	0.48	104%	75, 75	75
23	0.54, 0.54, 0.52	0.53	115%	83, 71	77

Apples, potatoes, wheat grain and wheat straw

Report: Not assigned

Study No. A-22-00-03

Method: "Determination of Residues of IKI-220 and its Metabolites TFNG, TFNA and TFNA-AM in Various Crops—Validation of the Method"

Description: Untreated control samples were fortified with flonicamid and its metabolites TFNA, TFNA-AM and TFNG at a concentration of 0.1 mg/kg per analyte for apple, potato and wheat grain and 0.2 mg/kg for wheat straw and then frozen below–17 °C. Samples were analysed immediately after fortification (0 day) and after storage intervals up to 18 months. At each interval, two stored samples were analysed, with one or more procedural recovery samples (control samples spiked just before analysis).

Table 43 Storage Stability of Flonicamid, TFNA, TFNA-AM and TFNG in apple, potato and wheat

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
Apples					
0		0.093	100%	95, 89, 86	90
3	0.09, 0.09	0.09	97%	93	93
6	0.11, 0.11	0.11	118%	106	106
12	0.11, 0.10	0.11	118%	99	99
18	0.10, 0.09	0.10	108%	89, 87	88
Potatoes					
0		0.09	100%	89, 92, 93	91
3	0.09, 0.10	0.09	100%	107	107
6	0.11, 0.10	0.10	111%	109	109
12	0.10, 0.14	0.12	133%	82	82
18	0.12, 0.10	0.10	111%	113, 96	104
Wheat grain					
0		0.10	100%	93, 92, 98	94
3	0.10, 0.10	0.10	100%	96	96
6	0.08, 0.08	0.08	80%	87	87
12	0.08, 0.10	009	90%	96	96
18	0.09, 0.10	0.10	100%	94, 94	94
Wheat straw					
0		0.19	100%	104, 110, 82	99
3	0.20, 0.18	0.19	100%	90	90
6	0.22, 0.22	0.22	116%	113	113
12	0.21, 0.22	0.21	111%	102	102
18	0.20, 0.23, 0.21	0.22	116%	87, 111	99
TFNG					
Apples					
0	0.09, 0.08	0.09	100%	76, 93, 78	82
3	0.10, 0.09	0.10	111%	91.00	91
6	0.08, 0.09	0.09	100%	74.00	74
12	0.12, 0.11	0.12	133%	56.00	89
18	0.10, 0.10	0.10	111%	94, 102	97
Potatoes					
0		0.08	100%	90, 84, 84	86
3	0.08, 0.09	0.08	100%	102	102
6	0.10, 0.09	0.09	113%	93	93
12	0.09, 0.11	0.10	125%	80	80
18	0.11, 0.10	0.10	125%	106, 91	98
Wheat grain					

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
0		0.08	100%	87, 67, 86	80
3	0.08, 0.08	0.08	100%	78	78
6	0.08, 0.08	0.08	100%	92	92
12	0.17, 0.09	0.13	163%	85	85
18	0.08, 0.08	0.08	100%	83	83
Wheat straw					
0		0.15	100%	79, 84, 87	77
3	0.16, 0.15	0.16	107%	78	78
6	0.18, 0.18	0.18	120%	93	93
12	0.20, 0.20	0.2	133%	100	100
18	0.16, 0.20, 0.23	0.2	133%	68, 124	96
TFNA					
Apples					
0		0.10	100%	90, 81, 83	85
3	0.08, 0.07	0.07	70%	66	66
6	0.08, 0.07	0.07	70%	77	77
12	0.12, 0.11	0.12	120%	83	83
18	0.08, 0.08	0.08	80%	88, 86	87
Potatoes					
0		0.11	100%	115, 106, 113	112
3	0.10, 0.12	0.11	100%	78	78
6	0.08, 0.08	0.08	73%	77	77
12	0.09, 0.11	0.10	91%	80	80
18	0.07, 0.09	0.08	73%	71	71
Wheat grain					
0		0.1	100%	117, 101, 99	106
3	0.07, 0.07	0.07	70%	71	71
6	0.09, 0.08	0.08	80%	89	89
12	0.12, 0.08	0.1	100%	79	79
18	0.07, 0.07	0.07	70%	72	72
Wheat straw					
0		0.18	100%	109, 109, 76	98
3	0.14, 0.13	0.13	72%	72	72
6	0.15, 0.15	0.15	83%	77	77
12	0.19, 0.19	0.19	106%	97	97
18	0.13, 0.15, 0.14	0.14	120%	45, 85	65
TFNA-AM					
Apples					
0		0.08	100%	93, 81, 83	86
3	0.08, 0.09	0.09	113%	97	97
6	0.10, 0.09	0.10	125%	94	94
12	0.11, 0.08	0.10	125%	91	91
18	0.07, 0.10	0.09	113%	83, 100	92
Potatoes					
0		0.08	100%	80, 73, 79	78
3	0.08, 0.09	0.08	100%	89	89
6	0.09, 0.09	0.09	113%	87	87
12	0.08, 0.10	0.09	113%	77	77
18	0.09, 0.10	0.09	113%	78	78
Wheat grain					
0		0.09	100%	93, 84, 99	92
3	0.08, 0.08	0.08	89%	82	82
6	0.08, 0.07	0.08	89%	81	81
12	0.07, 0.08	0.08	89%	76	76
18	0.08, 0.08	0.08	89%	87	87

Time	Flonicamid				
	Individual Stored Sample Residues (mg/kg)	Mean Stored Sample Residue (mg/kg)	Remaining	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)
Wheat straw					
0		0.17	100%	85, 86, 80	84
3	0.17, 0.15	0.16	94%	80	80
6	0.19, 0.19	0.19	112%	99	99
12	0.17, 0.17	0.17	100%	87	86
18	0.16, 0.19, 0.20	0.18	106%	70, 107	89

USE PATTERN

The insecticide flonicamid is registered in Canada, the United States, Slovenia, Cyprus and Australia for control of various insects on a variety of crops. The information available to the Meeting on registered uses on various fruits, vegetables, tree nuts, oilseeds, dried hops, mint and tea is summarized in Table 44. The manufacturer submitted labels for all flonicamid uses.

Table 44 Registered uses of flonicamid

Crop	Country	Form.	Application				PHI, Days
			Method	Rate, kg ai/ha	Spray conc., kg ai/hL	No.	
Pome fruits							
Pome fruits	USA	50WG/50SG	Foliar	0.07–0.1	0.01–0.02	3	21
Apples	AUS	500WG	Foliar	NS	0.005–0.01	3	21
Apples, pears	Cyprus	50WG	Foliar	0.06–0.14	0.006–0.01	3	21
Apples	Slovenia	50WG	Foliar	0.07	0.014	3	21
Stone fruits							
Stone fruits	USA	50WG/50SG	Foliar	0.07–0.1	0.01–0.02	3	14
Peaches	Cyprus	50WG	Foliar	0.06–0.07	0.006–0.007	2	14
Plums							35
Peaches	Slovenia	50WG	Foliar	0.07	0.014	3	14
Berries and other small fruit							
Low growing berries	USA	50SG	Foliar	0.1	0.02–0.10	3	0
Brassica (cole or cabbage) vegetables							
Brassica (cole) leafy vegetables	USA	50WG/50SG	Foliar	0.07–0.1	0.07–0.1	3	0
Fruiting vegetables, cucurbits							
Cucurbits	USA	50WG/50SG	Foliar	0.07–0.1	0.07–0.1	3	0
Greenhouse cucumber	USA	50WG/50SG	Foliar	0.15	0.1–0.15	2	0
			Soil	0.15	NS	2	0
Cucurbits	AUS	50WG	Foliar	0.05–0.1	NS	3	1
Cucurbits (Field and Greenhouse)	Cyprus	50WG	Soil	0.05	0.005	3 (total)	3
			Foliar	0.10	NS		
Cucurbits	Slovenia	50WG	Foliar	0.05	0.005	3	1
Fruiting vegetables, other than cucurbits							
Fruiting vegetables	USA	50WG/50SG	Foliar	0.1	0.1	3	0
				0.15	0.15	2	0
Greenhouse tomatoes	USA	50WG/50SG	Foliar	0.15	0.15	2	0
Tomatoes (field and greenhouse)	Cyprus	50WG	Soil	0.05–0.06	0.005–0.006	3 (total)	3
			Foliar	0.10	NS		
Leafy vegetables (including Brassica leafy vegetables)							
Leafy vegetables	USA	50WG/50SG	Foliar	0.07–0.1	0.07–0.3	3	0

Crop	Country	Form.	Application				PHI, Days
			Method	Rate, kg ai/ha	Spray conc., kg ai/hL	No.	
(except Brassica vegetables)							
Brassica (cole) leafy vegetables	USA	50WG/50SG	Foliar	0.07–0.1	0.07–0.3	3	0
Root and tuber vegetables							
Tuberous and corm vegetables	USA	50WG/50SG	Foliar	0.07–0.1	0.07–0.3	3	7
Root vegetables (except sugar beets)	USA	50WG/50SG	Foliar	0.07–0.1	0.07–0.3	3	3
Potatoes	AUS	500WG	Foliar	0.07–0.1	NS	2	14
Potatoes	Slovenia	50WG	Foliar	0.08	0.016	2	14
Stems and petioles							
Leafy vegetables (except Brassica)	USA	50WG/50SG	Foliar	0.07–0.1	0.07–0.1	3	0
Cereal grains							
Wheat, rye, triticale	Slovenia	50WG	Foliar	0.07	0.014	2	28
Tree Nuts							
Tree nuts	USA	50WG/50SG	Foliar	0.07–0.1	0.01–0.02	3	40
Oilseed							
Cotton	USA	50WG/50SG	Foliar	0.05–0.1	0.02–0.05	3	30
Cotton	AUS	500 WG	Foliar	0.05–0.07	NS	2	7
Rape seed	USA	50WG/50SG	Foliar	0.1	0.1	3	7
Herbs							
Mint	USA	50WG/50SG	Foliar	0.07–0.1	0.04–0.05	3	7
Dried herbs							
Hops	USA	50WG/50SG	Foliar	0.06–0.1	0.01–0.02	3	10
Hops	Slovenia	50WG	Foliar	0.09	0.0225	2	21
Straw, fodder and forage of cereal grains and grasses							
Alfalfa seed	USA	50WG/50SG	Foliar	0.1	0.05	2	14 forage and seed 62 hay
Teas							
Tea	Japan	DF	Foliar	0.1	0.01	1	7

NS Not specified

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised field trials for flonicamid uses that produced residues on the following commodities:

Commodity	Group	Table No.
Apples	FP Pome fruits	45
Pears		46
Peaches	FS Stone fruits	47
Cherries		48
Plums		49
Strawberries	FB Berries and small fruits	50
Broccoli	VB Brassica vegetables	51

Commodity	Group	Table No.
Cabbage		52
Cucumber	VC Fruiting vegetables, Cucurbits	53
Melon		54
Summer squash		55
Tomatoes	VO Fruiting vegetables, other than Cucurbits	56
Bell peppers		57
Non-bell peppers		58
Head lettuce	VL Leafy vegetables (including Brassica leafy vegetables)	59
Leaf lettuce		60
Spinach		61
Radish leaves		62
Mustard greens		63
Potato	VR Root and tuber vegetables	64
Carrot		65
Radish		66
Celery	VS Stem and petioles	67
Wheat	Cereal grains	68
Barley		69
Almonds	TN Tree nuts	70
Pecans		71
Pistachios		72
Rape seed	SO Oilseed	73
Cotton seed		74
Mint	HH Herbs	75
Hops	DH Dried herbs	76
Tea	DT Teas	77
Wheat forage and straw	AS Straw, fodder and forage of cereal grains and grasses	78
Barley forage and straw		79
Alfalfa		80
Almond hulls		81
Cottonseed gin trash		82

In the residue supervised trials tables, where two samples were taken from a single plot, the average value is reported (individual sample results in parentheses). Where results from separate plots with distinguishing characteristics such as different formulations, varieties or treatment schedules were reported, results are listed for each plot. In these cases, the higher residue has been used for calculation purposes. Dates of duration of residue sample storage before analysis were provided.

Residue values from the trials conducted according to the maximum GAP have been used for the estimation of maximum residue levels. Those results included in the calculations by the OECD MRL-calculator are underlined.

Pome fruits

Apple

Twelve independent trials were conducted on apples in the US between 1968 and 1995. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Apples were harvested 14–21 days after last treatment (DALT).

In Australia. fourteen independent trials were conducted on between 1983 and 2009. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days and apples were harvested 21 DALT.

The analytical method P-3568 (based on method P-3561M) was used to analyse samples collected from the US trials while method AATM-R-165 was used to analyse the samples from the Australian trials. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 35 days for the Australian trials and 297 days (ca. 10 months) for the US trials Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 45.

Table 45 Residues of Flonicamid in Apples Following Foliar Spray with Flonicamid WG Formulation in Regions of North America

Location, year (variety)	Application						DALT, days	Residues, mg/kg				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/ SG	0.07 – 0.10	0.01–0.02	100–500	3	7	21					
Lyons, NY, 1998 (Jonigold)	WG	0.10	0.01	756	3	7	0	0.065 (0.063, 0.067)	< 0.01 (< 0.01, < 0.01)	0.034 (0.030, 0.038)	< 0.01 (< 0.01, < 0.01)	IB-2001-MDG-003-00-01
							7	0.055 (0.052, 0.058)	< 0.01 (< 0.01, < 0.01)	0.037 (0.035, 0.039)	< 0.01 (< 0.01, < 0.01)	
							14	0.064 (0.062, 0.065)	< 0.01 (< 0.01, < 0.01)	0.037 (0.040, 0.049)	< 0.01 (< 0.01, < 0.01)	
							21	0.033 (0.032, 0.034)	< 0.01 (< 0.01, < 0.01)	0.039 (0.039, 0.039)	< 0.01 (< 0.01, < 0.01)	
							28	0.047 (0.060, 0.034)	< 0.01 (< 0.01, < 0.01)	0.018 (0.018, 0.017)	< 0.01 (< 0.01, < 0.01)	
Dundee, NY, 1973 (Macoun)	WG	0.10	0.01	941	3	7	21	0.037 (0.032, 0.042)	< 0.01 (< 0.01, < 0.01)	0.021 (0.017, 0.024)	< 0.01 (< 0.01, < 0.01)	
Herford, PA, 1968 (Starkrimson Red Delicious)	WG	0.10	0.02	511	3	7	20	0.037 (0.043, 0.031)	< 0.01 (< 0.01, < 0.01)	0.024 (0.011, 0.013)	< 0.01 (< 0.01, < 0.01)	
Cana, VA, 1994 (Red Delicious)	WG	0.10	0.01	940	3	7	20	0.047 (0.045, 0.048)	< 0.01 (< 0.01, < 0.01)	0.018 (0.016, 0.019)	< 0.01 (< 0.01, < 0.01)	
Conklin, MI, 1993 (Golden Delicious)	WG	0.10	0.01	794	3	7	21	0.097 (0.099, 0.095)	< 0.01 (< 0.01, < 0.01)	0.038 (0.038, 0.038)	< 0.01 (< 0.01, < 0.01)	
Menomonie, WI,	WG	0.10	0.02	468	3	7	21	0.066 (0.067,	< 0.01 (< 0.01,	0.018 (0.017,	< 0.01 (< 0.01,	

Location, year (variety)	Application						DALT, days	Residues, mg/kg				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
1985 (Prairie Sky)								0.064)	< 0.01)	0.018)	< 0.01)	
Eckert, CO, 1984 (Red Delicious)	WG	0.10	0.01	749	3	7	21	0.049 (0.044, 0.054)	< 0.01 (< 0.01, < 0.01)	0.018 (0.016, 0.020)	< 0.01 (< 0.01, < 0.01)	
Fairfield, CA, 1992 (Golden Delicious)	WG	0.10	0.02	637	3	7	14	0.111 (0.104, 0.117)	< 0.01 (< 0.01, < 0.01)	0.041 (0.037, 0.044)	< 0.01 (< 0.01, < 0.01)	
Hood River, OR, 1991 (Red Delicious)	WG	0.10	0.02	665	3	7	20	0.057 (0.055, 0.058)	< 0.01 (< 0.01, < 0.01)	0.016 (0.017, 0.015)	< 0.01 (< 0.01, < 0.01)	
Hood River, OR, 1993 (Jonigold)	WG	0.10	0.01	742	3	7	20	0.023 (0.024, 0.022)	< 0.01 (< 0.01, < 0.01)	0.015 (0.014, 0.016)	< 0.01 (< 0.01, < 0.01)	
Hood River, OR, 1995 (Gala)	WG	0.10	0.01	1032	3	7	21	0.039 (0.038, 0.039)	< 0.01 (< 0.01, < 0.01)	0.014 (0.015, 0.012)	< 0.01 (< 0.01, < 0.01)	
Outlook, WA, 1995 (Red Delicious)	WG	0.10	0.01	960	3	7	21	0.052 (0.053, 0.051)	< 0.01 (< 0.01, < 0.01)	0.019 (0.019, 0.019)	< 0.01 (< 0.01, < 0.01)	
AUS GAP	WG	NS	0.005–0.01	100–1000	3	14	21					
Batlow, New South Wales, 2006 (Sundowner)	WG	NS	0.01	2933–3352	3	7	0 14 21 27	0.34 0.16 0.12 0.093	< 0.01 < 0.01 < 0.01 < 0.01	0.017 0.040 0.049 0.054	< 0.01 < 0.01 < 0.01 < 0.01	
Batlow, New South Wales, 2006 (Sundowner)	WG	NS	0.02	2438–2952	3	7	0 14 21 27	0.86 0.24 0.23 0.17	< 0.01 < 0.01 < 0.01 < 0.01	0.032 0.074 0.11 0.11	< 0.01 < 0.01 < 0.01 < 0.01	
Batlow, New South Wales, 2000 (Pink Lady)	WG	NS	0.01	1856–2022	3	7	21	0.24	< 0.01	0.033	< 0.01	UP L-1002
Batlow, New South Wales, 2000 (Pink Lady)	WG	NS	0.02	1800–2078	3	7	21	0.47	< 0.01	0.067	0.011	
Spreyton, Tasmania, 2009 (Pink Lady)	WG	NS	0.01	201–240	3	7	0 14 21 28	0.20 0.097 0.086 0.010	< 0.01 < 0.01 < 0.01 < 0.01	0.010 0.023 0.034 0.045	< 0.01 < 0.01 < 0.01 0.012	
Grove, Tasmania, 1996 (Fuji)	WG	NS	0.02	1600–1659	3	7	21	0.024	< 0.01	0.1	0.018	
Yering,	WG	NS	0.01	4115	3	7	0	0.43	< 0.01	< 0.01	< 0.01	UP

Location, year (variety)	Application						DALT, days	Residues, mg/kg				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Victoria, 1984 (Fuji)				– 4398			14 21 28	0.16 0.15 0.16	< 0.01 < 0.01 < 0.01	0.023 0.029 0.032	0.011 0.018 0.023	L-1108
Yering, Victoria, 1984 (Fuji)	WG	NS	0.02	3978 – 4374	3	7	0 14 21 28	0.59 0.38 0.29 0.25	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.026 0.039 0.040	< 0.01 0.018 0.028 0.033	
Arding, New South Wales, 1983 (Red Delicious)	WG	NS	0.01	2153 – 2500	3	7	21	0.13	< 0.01	0.036	0.012	
Arding, New South Wales, 1983 (Red Delicious)	WG	NS	0.02	2153 – 2361	3	7	21	0.27	< 0.01	0.063	0.023	
Spreyton, Tasmania, 2010 (Golden Delicious)	WG	NS	0.01	1144 – 1337	3	7	21	0.12	< 0.01	0.069	0.01	
Spreyton, Tasmania, 2010 (Golden Delicious)	WG	NS	0.02	1248 – 1381	3	7	21	0.23	< 0.01	0.099	0.019	
Stanthorpe, Queensland, 1985 (Granny Smith)	WG	NS	0.01	2645 – 3043	3	7	0 14 21 28	0.28 0.19 0.22 0.22	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 0.030 0.035 0.057	< 0.01 0.026 0.033 0.050	
Stanthorpe, Queensland, 1985 (Granny Smith)	WG	NS	0.02	2101 – 3043	3	7	0 14 21 28	0.48 0.38 0.43 0.45	< 0.01 < 0.01 < 0.01 < 0.01	0.011 0.050 0.079 0.13	0.018 0.046 0.071 0.11	

Pear

Six independent trials were conducted on pears in the US between 1962 and 1996. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Pears were harvested 14–21 DALT.

The analytical method P-3568 (based on method P-3561M) was used to analyse the samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 329 days (ca. 11 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 46.

Table 46 Residues of Flonicamid in Pears Following Foliar Spray with Flonicamid 50 WG Formulation in Regions of North America

Location,	Application	DAL	Residues (mg/kg)	Ref

year (variety)	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	T, days	Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/ WS	0.07–0.10	0.01–0.02	100–500	3	7	21					
Lyons, NY, 1968 (Clapps Favorite)	WG	0.10	0.01	936	3	7	21	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.021 (0.021, 0.020)	< 0.01 (< 0.01, < 0.01)	IB-2001_MDG-003-00-01
Fairfield, CA, 1986 (Bartlett)	WG	0.10	0.02	654	3	7	14	0.018 (0.017, 0.019)	< 0.01 (< 0.01, < 0.01)	0.045 (0.048, 0.041)	< 0.01 (< 0.01, < 0.01)	
Isleton, CA, 1972-1996 (Bartlett)	WG	0.10	0.02	650	3	7	14	0.016 (0.013, 0.018)	< 0.01 (< 0.01, < 0.01)	0.038 (0.031, 0.044)	< 0.01 (< 0.01, < 0.01)	
Soap Lake, WA, 1962 (Bartlett)	WG	0.10	0.005	1880	3	7	21	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.023 (0.023, 0.022)	< 0.01 (< 0.01, < 0.01)	
Hood River, OR, 1994 (Starkrimson)	WG	0.10	0.01	861	3	7	0	0.020 (0.019, 0.020)	< 0.01 (< 0.01, < 0.01)	0.037 (0.040, 0.034)	< 0.01 (< 0.01, < 0.01)	
							7	0.014 ((0.015, 0.013)	< 0.01 (< 0.01, < 0.01)	0.033 (0.036, 0.030)	< 0.01 (< 0.01, < 0.01)	
							14	0.010 (0.010, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.022 (0.020, 0.024)	< 0.01 (< 0.01, < 0.01)	
							21	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.026 (0.030, 0.022)	< 0.01 (< 0.01, < 0.01)	
Zillah, WA, 1985 (Bartlett)	WG	0.099	0.011	905	3	7	21	0.020 (0.019, 0.021)	< 0.01 (< 0.01, < 0.01)	0.031 (0.029, 0.033)	< 0.01 (< 0.01, < 0.01)	

Stone Fruit

Peach

Nine independent trials were conducted on peaches in the US between 1976 and 1998. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Peaches were harvested 10–14 DALT.

The analytical method P-3561M was used to analyse the samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 329 days (ca. 11 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 47.

Table 47 Residues of Flonicamid in Peaches Following Foliar Spray with Flonicamid 50 WG Formulation in Regions of North America

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/WS	0.07–0.10	0.01–0.02	100–500	3	7	14					

Lyons, NY, 1998 (Harcrest)	WG	0.10	0.01	754	3	7	0	0.298 (0.284, 0.311)	< 0.01 (< 0.01, < 0.01)	0.026 (0.026, 0.026)	0.015 (0.015, 0.014)	IB-2001-MDG-005-00-01
							3	0.308 (0.289, 0.326)	< 0.01 (< 0.01, < 0.01)	0.024 (0.024, 0.025)	0.014 (0.013, 0.014)	
							7	0.190 (0.179, 0.201)	< 0.01 (< 0.01, < 0.01)	0.032 (0.037, 0.027)	0.014 (0.015, 0.012)	
							14	0.216 (0.225, 0.207)	< 0.01 (< 0.01, < 0.01)	0.038 (0.050, 0.026)	0.024 (0.026, 0.022)	
Covesville, VA, 1985 (Blake)	WG	0.10	0.02	680	3	7	14	0.087 (0.091, 0.082)	< 0.01 (< 0.01, < 0.01)	0.036 (0.043, 0.028)	0.025 (0.032, 0.018)	
Monetta, SC, 1990 (Crest Haven)	WG	0.10	0.01	829	3	7	14	0.086 (0.096, 0.075)	< 0.01 (< 0.01, < 0.01)	0.020 (0.023, 0.017)	0.012 (0.011, 0.012)	
Kinston, NC, 1995 (Legend)	WG	0.10	0.01	938	3	7	14	0.423 (0.400, 0.446)	< 0.01 (< 0.01, < 0.01)	0.020 (0.019, 0.021)	0.015 (0.014, 0.015)	
Conklin, MI, 1995 (Red Haven)	WG	0.10	0.01	979	3	7	14	0.095 (0.100, 0.090)	< 0.01 (< 0.01, < 0.01)	0.011 (< 0.01, 0.012)	0.012 (0.013, < 0.01)	
Waller, TX, 1989 (Idylwild)	WG	0.11	0.005	2120	3	7	10	0.065 (0.055, 0.074)	< 0.01 (< 0.01, < 0.01)	0.017 (0.014, 0.020)	0.011 (< 0.01, 0.012)	
Winters, CA, 1976 (Fay Elberta)	WG	0.11	0.02	521	3	7	14	0.151 (0.184, 0.117)	< 0.01 (< 0.01, < 0.01)	0.054 (0.065, 0.042)	0.023 (0.027, 0.018)	
Berenda, CA, 1988 (Last Chance)	WG	0.10	0.01	935	3	7	14	0.219 (0.218, 0.220)	< 0.01 (< 0.01, < 0.01)	0.051 (0.054, 0.047)	0.057 (0.060, 0.053)	
Selma, CA, 1996 (September Sun)	WG	0.10	0.01	938	3	7	14	0.134 (0.149, 0.119)	< 0.01 (< 0.01, < 0.01)	0.067 (0.070, 0.063)	0.023 (0.026, 0.020)	

Cherry

Six independent trials were conducted on cherries in the US between 1989 and 1995. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Cherries were harvested 14 DALT.

The analytical method P-3561M was used to analyse the samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 329 days (ca. 11 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 48.

Table 48 Residues of Flonicamid in Cherries Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/ WS	0.07–0.10	0.01–0.02	100–500	3	7	14					
Conklin MI, 1995 (Napoleon)	WG	0.10	0.01	945–963	3	7–8	0	0.759 (0.736, 0.782)	< 0.01 (< 0.01, < 0.01)	0.036 (0.031, 0.021)	0.022 (0.022, 0.022)	IB-2001-MDG-005-00-01
							3	0.360 (0.326, 0.394)	< 0.01 (< 0.01, < 0.01)	0.032 (0.027, 0.036)	0.021 (0.019, 0.023)	
							7	0.290 (0.282, 0.297)	< 0.01 (< 0.01, < 0.01)	0.038 (0.042, 0.034)	0.027 (0.026, 0.027)	
							14	<u>0.273</u> (0.292, 0.253)	< 0.01 (< 0.01, < 0.01)	0.042 (0.042, 0.041)	0.042 (0.045, 0.039)	
Conklin MI, 1993 (Mont-morency)	WG	0.10	0.01	935–954	3	7–8	14	<u>0.276</u> (0.289, 0.262)	< 0.01 (< 0.01, < 0.01)	0.028 (0.028, 0.027)	0.026 (0.028, 0.024)	
Fairfield, CA, 1990 (Ranier)	WG	0.10	0.02	655	3	6	14	<u>0.281</u> (0.271, 0.290)	< 0.01 (< 0.01, < 0.01)	0.167 (0.161, 0.172)	0.048 (0.044, 0.052)	
Courtland CA, 1992 (Bing)	WG	0.10	0.02	655–673	3	6	14	<u>0.256</u> (0.238, 0.273)	< 0.01 (< 0.01, < 0.01)	0.044 (0.041, 0.047)	0.030 (0.029, 0.031)	
Parkdale OR, 1994 (Bing)	WG	0.10	0.01	973–1094	3	7	14	<u>0.266</u> (0.302, 0.230)	< 0.01 (< 0.01, < 0.01)	0.037 (0.040, 0.034)	0.035 (0.037, 0.032)	
Granger WA, 1989, (Bing)	WG	0.10	0.01	926–963	3	7	14	<u>0.365</u> (0.387, 0.343)	< 0.01 (< 0.01, < 0.01)	0.065 (0.065, 0.064)	0.062 (0.061, 0.063)	

Plum

Five independent trials were conducted on <u>plums</u> in the US between 1980 and 1995. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Plums were harvested 14 DALT.

The analytical method P-3561M was used to analyse the samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 329 days (ca. 11 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 49.

Table 49 Residues of Flonicamid in Plums Following Foliar Spray with Flonicamid 50 WG Formulation in Regions of North America

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.01–0.02	100–500	3	7	14					
Conklin MI, 1995 (Stanley)	WG	0.10	0.01	954	3	7	14	<u>0.041</u> (0.040, 0.042)	0.012 (0.014, < 0.01)	0.016 (0.017, 0.015)	< 0.01 (< 0.01, < 0.01)	IB-2001-MDG-

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Fairfield [a] CA, 1992 (French)	WG	0.10	0.02	505–514	3	5–8	14	0.044 (0.044, 0.044)	0.025 (0.026, 0.023)	0.011 (0.011, < 0.01)	0.012 (0.011, 0.012)	005-00-01
Fairfield [a] CA, 1992 (French)	WG	0.10	0.02	514	3	6–7	14	0.045 (0.051, 0.038)	0.016 (0.019, 0.013)	0.012 (< 0.01, 0.014)	0.01 (0.012, < 0.011)	
Madera CA, 1990 (Fortune)	WG	0.10	0.01	935–963	3	7	0	0.011 (0.011, 0.011)	< 0.01 (< 0.01, < 0.01)	0.040 (0.044, 0.035)	< 0.01 (< 0.01, < 0.01)	
							3	0.012 (0.012, 0.012)	< 0.01 (< 0.01, < 0.01)	0.040 (0.041, 0.038)	< 0.01 (< 0.01, < 0.01)	
							7	0.012 (0.012, 0.012)	< 0.01 (< 0.01, < 0.01)	0.043 (0.041, 0.044)	< 0.01 (< 0.01, < 0.01)	
							14	0.013 (0.012, 0.014)	< 0.01 (< 0.01, < 0.01)	0.045 (0.043, 0.046)	< 0.01 (< 0.01, < 0.01)	
Selma CA, 1997 (Howard Sun)	WG	0.10	0.01	917–926	3	7	14	0.032 (0.041, 0.023)	0.010 (0.010, < 0.01)	0.027 (0.017, 0.037)	0.010 (0.010, < 0.01)	
Hillsboro OR, 1980 (Italian)	WG	0.10	0.01	823–851	3	7	14	0.023 (0.023, 0.023)	< 0.01 (< 0.01, < 0.01)	0.011 (0.011, 0.010)	< 0.01 (< 0.01, < 0.01)	

[a] The last applications at each trial site were made on the same day and varieties were the same rendering the trials dependent

Berries and other small fruits

Strawberry

Eight independent trials were conducted on strawberries in the US in 2008. In all trials, three foliar spray applications of a SG formulation were made with a re-treatment interval of 6–8 days. Strawberries were harvested 14 DALT.

The analytical method P-3561M was used to analyse the samples. The LOQ was determined to be 0.02 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 498 days (ca. 17 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 50.

Table 50 Residues of Flonicamid in Strawberries Following Foliar Spray with Beleaf 50 SG Formulation in Regions of North America

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	SG	0.10	0.02–0.10	100–500	3	7	0					
Salinas, CA, 2008 (Albion) [a]	SG	0.10	0.02–0.04	253–440	3	7	0	0.47 (0.42, 0.51)	< 0.020 (0.020, < 0.020)	0.041 (0.037, 0.044)	0.038 (0.034, 0.042)	9604
Salinas, CA, 2008	SG	0.10	0.02–0.04	299–496	3	6	0	0.59 (0.52,	< 0.020 (0.020,	0.047 (0.044,	0.033 (0.030,	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
(Albion) [a]								0.66)	< 0.020)	0.049)	0.036)	
Parlier, CA, 2008 (Seascape)	SG	0.10	0.025	402–412	3	7	0	0.54 (0.48, 0.60)	< 0.020 (0.020, < 0.020)	0.10 (0.10, 0.10)	0.056 (0.053, 0.58)	
Balm, FL, 2008 (Festival)	SG	0.10	0.027	374	3	8–11	0	0.27 (0.29, 0.24)	< 0.020 (< 0.020, < 0.020)	0.051 (0.058, 0.044)	0.028 (0.032, 0.024)	
Clinton, NC, 2008 (Chandler)	SG	0.10	0.03	318–327	3	7	0	0.33 (0.34, 0.32)	< 0.020 (< 0.020, < 0.020)	0.021 (0.020, 0.022)	< 0.020 (< 0.020, < 0.020)	
							3	0.23 (0.19, 0.27)	< 0.02 (< 0.02, < 0.02)	0.024 (0.024, 0.025)	< 0.02 (< 0.02,, < 0.02)	
							5	0.16 (0.16, 0.15)	< 0.020 (< 0.020, < 0.020)	0.031 (0.030, 0.032)	< 0.020 (< 0.020, < 0.020)	
							7	0.14 (0.14, 0.14)	< 0.020 (< 0.020, < 0.020)	0.037 (0.036, 0.037)	< 0.020 (< 0.020, < 0.020)	
Penn Yan, NY, 2008 (Honeoye)	SG	0.099–0.1	0.034–0.035	281–290	3	7	0	0.41 (0.35, 0.46)	< 0.020 (< 0.020, < 0.020)	0.046 (0.040, 0.051)	0.087 (0.084, 0.090)	
Salem, OR, 2008 (Totem)	SG	0.104–0.105	0.024	430–440	3	6–10	0	0.13 (0.11, 0.15)	< 0.020 (< 0.020, < 0.020)	0.022 (< 0.02, 0.023)	0.021 (< 0.020, 0.022)	
Arlington, WI, 2008 (Kent)	SG	0.096–0.102	0.029	327–346	3	7–8	0	0.19 (0.20, 0.18)	< 0.020 (< 0.020, < 0.020)	< 0.020 (< 0.020, < 0.020)	< 0.020 (< 0.020, < 0.020)	

[a] The last applications at each site were made 2 months apart, rendering the trials independent

Brassica (Cole or cabbage) vegetables

Broccoli

Six independent trials were conducted on broccoli in the US during the 2003 growing season. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Plants were harvested 0 DALT.

The analytical method P-3561M was used to analyse the samples. The LOQ was determined to be 0.025 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 4 days. Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 51.

Table 51 Residues of Flonicamid in Broccoli Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location,	Application	DALT,	Residues (mg/kg)	Ref

year (variety)	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	days	Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.07–0.10	100	3	7	0					
East Bernard, TX, 2003 (Early Dividend)	WG	0.10	0.10	95–96	3	6–7	0	0.428 (0.484. 0.372)	< 0.025 (< 0.025, < 0.025)	< 0.025 (0.025, < 0.025)	0.077 (0.086, 0.068)	P-3679
Camarillo CA, 2003 (Marathon)	WG	0.11	0.11	93–95	3	5–8	0	0.373 (0.331, 0.435)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.034 (0.031, 0.036)	
							1	0.432 (0.338, 0.525)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.045 (0.041, 0.048)	
							3	0.308 (0.327, 0.288)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.048 (0.046, 0.049)	
							7	0.178 (0.186, 0.170)	< 0.025 (< 0.025, < 0.025)	0.032 (0.033, 0.030)	0.060 (0.062, 0.057)	
Visalia CA, 2003 (Waltham 29)	WG	0.10	0.10	94	3	7	0	0.462 (0.430, 0.493)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.144 (0.127, 0.161)	
Casa Grande AZ, 2003 (Marathon)	WG	0.10	0.11	95	3	6–7	0	0.499 (0.416, 0.581)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	
Yuma AZ, 2003 (Everest)	WG	0.10	0.11	93–96	3	7–9	0	0.250 (0.268, 0.232)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.027 (0.028, < 0.025)	
Hillsboro OR, 2003 (Packman)	WG	0.10	0.06	153–159	3	7	0	0.553 (0.515, 0.590)	< 0.025 (< 0.025, < 0.025)	0.056 (0.059, 0.053)	0.144 (0.150, 0.137)	

Cabbage

Six independent trials were conducted on cabbage in the US during the 2003 growing season. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Cabbage heads were harvested 0 DALT.

The analytical method P-3561M was used to analyse samples. The LOQ was determined to be 0.025 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 180 days (6 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 52.

Table 52 Residues of Flonicamid in Cabbage Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Matrix	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.07–0.10	100	3	7	0						
North Rose NY, 2003 (Early Thunder)	WG	0.10	0.10	94	3	7	0	Cabbage w/wrapper leaves	0.062 (0.066, 0.057)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.032 (0.034, 0.029)	
								Cabbage w/out wrapper leaves	< 0.025 (0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.091 (0.099, 0.082)	0.165 (0.184, 0.145)	
Delmar DE, 2003 (Blue Thunder)	WG	0.10	0.11	93–94	3	6–7	0	Cabbage w/wrapper leaves	0.205 (0.217, 0.193)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.053 (0.055, 0.051)	
								Cabbage w/out wrapper leaves	< 0.025 (0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.085 (0.088, 0.082)	0.141 (0.152, 0.129)	
Jennings FL, 2003 (Bravo)	WG	0.10	0.10	97	3	7	0	Cabbage w/wrapper leaves	1.262 (1.281, 1.243)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.089 (0.087, 0.090)	
								Cabbage w/out wrapper leaves	1.138 (1.067, 1.208)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.072 (0.069, 0.075)	P-3679
East Bernard, TX, 2003 (Early Jersey Wakefield)	WG	0.10	0.10	91–97	3	7–8	0	Cabbage w/wrapper leaves	0.288 (0.311, 0.265)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.037 (0.036, 0.037)	
								Cabbage w/out wrapper leaves	0.055 (0.059, 0.050)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	
Ellendale MN, 2003 (Dannish Ball)	WG	0.10	0.12	88–90	3	7	0	Cabbage w/wrapper leaves	0.025 (0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.074 (0.072, 0.075)	0.127 (0.123, 0.130)	
								Cabbage w/out wrapper leaves	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.070 (0.061, 0.079)	0.110 (0.087, 0.132)	
Visalia CA, 2003 (Copenhagan)	WG	0.10	0.11	94	3	7	0	Cabbage w/wrapper leaves	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025)	0.031 (0.031, 0.031)	
								Cabbage w/out wrapper leaves	< 0.025, (< 0.025 < 0.025)	< 0.025 (< 0.025, < 0.025)	0.035 (0.033, 0.036)	0.067 (0.064, 0.069)	

Fruiting vegetables, cucurbits

Cucumber

Six independent trials were conducted on field cucumbers in the US during the 2001 growing season. In all trials, three foliar spray applications of a WG formulation were made at 0.10 kg ai/ha with a re-treatment interval of 6–7 days. Cucumbers were harvested 0 DALT. Four independent trials were conducted on greenhouse cucumbers in Canada and the US during the 2008 and 2009 growing seasons. In each of the greenhouse trials, one of the plots was treated with two foliar spray applications of a SG formulation at 0.15 kg ai/ha with a re-treatment interval of 6–7 days. The other plot was treated twice via syringe to the rock wool cubes in which the plants were grown. Application rates were determined using an average plant density of 2.4 plants per square meter, regardless of the actual density in the respective trials. The nominal rate was 0.15 kg ai/ha per application for a total range of 0.30 kg ai/ha per season. In all trials, cucumbers were harvested 0 DALT.

Two independent trials were also conducted on field cucumbers in Australia during the 2011 and 2012 growing seasons. In both trials, three foliar spray applications of a WG formulation were made at 0.10 kg ai/ha or 0.20 kg ai/ha with a re-treatment interval of 7 days. Cucumbers were harvested 0, 1, 3 and 7 DALT.

For the North American trials, the analytical method P-3561M was used to analyse all samples. The LOQ for the field cucumbers was determined to be 0.025 mg/kg/analyte while the LOQ for greenhouse cucumbers was 0.02 mg/kg/analyte. In Australia, the analytical method AATM-R-165 was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 340 days (ca. 11.5 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 53.

Table 53 Residues of Flonicamid in Field and Greenhouse Cucumbers Following Foliar Spray with Flonicamid 50 WG (Field) in North American Regions and Australia and Beleaf 50SG (Greenhouse) in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Field Cucumbers												
US GAP	WG	0.07–0.10	0.07–0.10	100	3	7	0					
Cotton GA, 2001 (Cross Country)	WG	0.10	0.05	187	3	7	0	0.065 (0.063, 0.067)	< 0.01 (< 0.01, < 0.01)	0.042 (0.040, 0.044)	< 0.01 (< 0.01, < 0.01)	IB-2001-MDG-007-00-01
Rose Hill NC, 2001 (Poinsett)	WG	0.10	0.05	187	3	7	0	0.081 (0.086, 0.076)	< 0.01 (< 0.01, < 0.01)	0.052 (0.054, 0.050)	0.027 (0.028, 0.01)	
							1	0.116 (0.118, 0.113)	< 0.01 (< 0.01, < 0.01)	0.085 (0.085, 0.084)	0.082 (0.080, 0.084)	
							3	0.102 (0.094, 0.110)	< 0.01 (< 0.01, < 0.01)	0.060 (0.055, 0.064)	0.067 (0.063, 0.071)	
							7	0.049 (0.042, 0.056)	< 0.01 (< 0.01, < 0.01)	0.067 (0.063, 0.070)	0.075 (0.070, 0.079)	
Hobe Sound FL, 2001	WG	0.10	0.05	187	3	6–7	0	0.073 (0.076, 0.069)	< 0.01 (< 0.01, < 0.01)	0.045 (0.046, 0.044)	0.026 (0.027, 0.024)	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
(Speedway)												
Northwood ND, 2001 (Market-more 76)	WG	0.10	0.05	187	3	7	0	0.055 (0.047, 0.063)	< 0.01 (< 0.01, < 0.01)	0.102 (0.099, 0.104)	0.076 (0.069, 0.082)	
Arkansas WI, 2001 (Eureka)	WG	0.10	0.05	187	3	7	0	0.055 (0.052, 0.058)	< 0.01 (< 0.01, < 0.01)	0.155 (0.145, 0.164)	0.105 (0.098, 0.111)	
Eakly OK, 2001 (Boston pickling)	WG	0.10	0.05	178–187	3	6–7	0	0.039 [a] (0.041, 0.040; 0.038, 0.038)	< 0.01[1] (< 0.01, < 0.01; < 0.01, < 0.01)	0.090[1] (0.059, 0.057; 0.123, 0.121)	0.070[1] (0.043, 0.041; 0.108, 0.086)	
AUS GAP	WG	0.05–0.10			3	14	1					
Bowen, Queensland, 2011 (Black Prince)	WG	0.10	0.02	502	3	7	0	0.031	< 0.01	0.045	0.043	UPL-1003
							1	0.03	< 0.01	0.063	0.051	
							3	0.028	< 0.01	0.06	0.054	
							7	0.029	< 0.01	0.071	0.061	
		0.20	0.04	502	3	7	0	0.059	< 0.01	0.073	0.056	
							1	0.042	< 0.01	0.066	0.052	
							3	0.048	< 0.01	0.054	0.043	
							7	0.048	< 0.01	0.12	0.1	
Bowen, Queensland, 2012 (Gremlin)	WG	0.10	0.03	395	3	7	0	0.034	< 0.01	0.065	0.096	UPL-1007
							1	0.027	< 0.01	0.055	0.074	
							3	0.031	< 0.01	0.063	0.12	
							7	0.019	< 0.01	0.077	0.12	
		0.20	0.05	395	3	7	0	0.052	< 0.01	0.086	0.13	
							1	0.044	< 0.01	0.062	0.1	
							3	0.055	< 0.01	0.073	0.12	
							7	0.014	< 0.01	0.032	0.071	
Greenhouse Cucumbers												
US GAP	SG	0.15	0.15	minimum 100	2	7	0					71512-9
Foliar application												
Fort Collins CO, USA, 2009 (DRL 1061 F1)	SG	0.16	0.03	505–561	2	6	0	0.054 (0.046, 0.061)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	0.02 (0.02 0.02)	
Salisbury MD, USA, 2008 (Samir)	SG	0.15	0.03	468	2	7	0	0.14 (0.14, 0.14)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	0.03 (0.03 0.02)	
Crossville TN, USA, 2008 (DRL 1061 F1)	SG	0.16	0.05	290	2	7	0	0.54 (0.69, 0.39)	<0.02 (<0.02 <0.02)	0.03 (0.03, 0.03)	0.02 (<0.02, 0.03)	
Harrow ON, CAN, 2009 (Pyralis)	SG	0.14–0.15	0.012	1162–1197	2	7	0	0.061 (0.059, 0.062)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	
							3	0.053 (0.052,	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
								0.054)				
							5	0.048 (0.046, 0.050)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	0.02 (<0.02 (0.02)	
							7	0.042 (0.038, 0.048)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	0.03 (0.02, 0.04)	
Soil application												
Fort Collins CO, USA, 2009 (DRL 1061 F1)	SG	0.15	N/A		2	6	0	<u>0.13</u> (0.13, 0.12)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	
Salisbury MD, USA, 2008 (Samir)	SG	0.15	N/A		2	7	0	<u>0.20</u> (0.20, 0.20)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	0.11 (0.11, 0.11)	
Crossville TN, USA, 2008 (DRL 1061 F1)	SG	0.15	N/A		2	7	0	<u>0.094</u> (0.094, 0.094)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	
Harrow ON, CAN, 2009 (Pyralis)	SG	0.15	N/A		2	7	0	0.14 (0.13, 0.14)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	
							3	0.15 (0.14, 0.16)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	
							5	0.16 (0.17, 0.15)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	0.02 (0.03, 0.02)	
							7	<u>0.16</u> (0.18, 0.13)	<0.02 (<0.02 <0.02)	<0.02 (<0.02 <0.02)	0.04 (0.05, 0.03)	

[a] Mean of four duplicate samples

N/A = Not applicable as treatment was made via syringe to the growth media

Melons

Six independent trials were conducted on <u>melons</u> in the USA during the 2003 growing season. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Melons were harvested 0 DALT.

The analytical method P-3561M was used to analyse samples. The LOQ was determined to be 0.01 mg/kg/analyte. On average, for all the trials, 12 fruits were sampled from each control and treated plot. In trials 5, 11, 13 and 17, fruits were quartered and each quarter was placed in a plastic bag and stored in a freezer. In Trial 14, melons were cut and 1/8th of each melon was placed into plastic bags. In Trial 15, the study only reported that fruits were placed into plastic bags. Trials 1 through 4, 6 through 10, 12 and 16 were conducted on cucumbers or summer squash.

Five independent trials were conducted on <u>rockmelons</u> (cantaloupe) in Australia during the 2010, 2011 and 2012 growing seasons. In all trials, three foliar spray applications of a WG formulation were made at 0.10 kg ai/ha or 0.20 kg ai/ha with a re-treatment interval of 7 days and DALTs of 0, 1, 3 and 7 days.

The analytical method AATM-R-165 was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte. Five commercially mature fruit weighing > 7 kg were collected by hand from five plants per plot and placed in labelled specimen bags. No specimens were collected from the buffer areas of each plot. Sampling was conducted after the spray solution had dried at the 0-day DALT. Any soil adhering to the fruit was removed by brushing not washing. Gloves were worn and changed between treatments. All specimens were double bagged and labelled in accordance with the specimen list defined in the study plan. No information was provided as to whether the melons were cut prior to bagging.

A total of thirteen independent trials were conducted in Southern Europe (France, Italy and Spain) on field and greenhouse-grown melons during the 2003, 2004 and 2011 growing seasons. In the trials conducted during 2003 and 2004, three foliar spray applications of a WG formulation were made at 0.08 kg ai/ha with re-treatment intervals of 4–10 days. In the trials conducted in 2011, three foliar spray applications of a WG formulation were made at 0.05 kg ai/ha with a re-treatment interval of 7 days. In all trials, melons were harvested 0, 1, 2, 3 and/or 7 DALT.

For the 2003 and 2004 trials, the analytical method was based on the method A22-00-02 and adapted to melon peel by changing the C_{18} clean-up. For the 2011 trials, the LC-MS/MS method AGR/MOA/IKI220-1 was used to analyse all samples. For both methods, the LOQ was determined to be 0.01 mg/kg/analyte for each peel and pulp. In general, each harvested fruit was cut in minimum of two slices. From each retained slice, the peel was separated from the pulp.

In total, the maximum period of sample storage at –20 °C was up to 340 days (11.5 months). Storage stability data on water content commodities show that residues are stable for at least 23 months. The results are summarized in Table 54.

Table 54 Residues of Flonicamid in Whole Melons Following Foliar Spray with a 50 WG Formulation of Flonicamid in North American Regions, Australia and Southern EU

Location, year (variety)	Trial No.	Application						DALT, days	Residues (mg/kg)				Ref
		Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP		WG	0.07–0.10	0.07–0.10	100	3	7	0					
Rose Hill NC, 2001 (Hales Best Jumbo)	5	WG	0.10	0.05	187	3	7	0	0.086 (0.089, 0.082)	< 0.01 (< 0.01, < 0.01)	0.107 (0.107, 0.107)	0.044 (0.046, 0.042)	IB-2001-MDG-007-00-01
Arkansaw WI, 2001 (Hybrid Primo)	11	WG	0.10	0.05	187	3	7	0	0.037 (0.036, 0.037)	< 0.01 (< 0.01, < 0.01)	0.054 (0.057, 0.050)	0.028 (0.029, 0.026)	
East Bernard TX, 2001 (Hales Best 36)	13	WG	0.10	0.08	131	3	7	0	0.056 (0.054, 0.058)	< 0.01 (< 0.01, < 0.01)	0.074 (0.076, 0.072)	0.023 (0.023, 0.022)	
Arbuckle CA, 2001 (Tendral Amaraillo Tandio)	14	WG	0.10	0.04	234	3	7	0	0.050 (0.045, 0.055)	< 0.01 (< 0.01, < 0.01)	0.044 (0.041, 0.046)	0.026 (0.023, 0.028)	
Maricopa AZ, 2001 (Olympic Gold)	15	WG	0.10	0.05	178–187	3	7	0	0.019 (0.021, 0.017)	< 0.01 (< 0.01, < 0.01)	0.077 (0.075, 0.079)	0.047 (0.047, 0.046)	
Fresno CA, 2001 (Top Mark)	17	WG	0.10	0.05	187–196	3	7	0	0.031 (0.029, 0.033)	< 0.01 (< 0.01, < 0.01)	0.082 (0.081, 0.083)	0.053 (0.053, 0.052)	
								1	0.019 (0.021, 0.017)	< 0.01 (< 0.01, < 0.01)	0.092 (0.113, 0.071)	0.059 (0.071, 0.046)	

Location, year (variety)	Trial No.	Application						DALT, days	Residues (mg/kg)				Ref
		Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
								3	0.024 (0.024, 0.023)	< 0.01 (< 0.01, < 0.01)	0.113 (0.102, 0.124)	0.082 (0.075, 0.088)	
								7	0.020 (0.014, 0.026)	< 0.01 (< 0.01, < 0.01)	0.153 (0.153, 0.153)	0.125 (0.116, 0.134)	
AUS GAP		WG	0.05–0.10	NS	NS	3		1					
Bowen, Queensland, 2011 (Hotshot)	NA	WG	0.10	0.02	499	3	7	0	0.091	< 0.01	0.065	0.023	UPL-1003
								1	0.17	< 0.01	0.063	0.026	
								3	0.076	< 0.01	0.047	0.017	
								7	0.031	< 0.01	0.049	0.023	
			0.20	0.04	499	3	7	0	0.25	< 0.01	0.14	0.055	
								1	0.25	< 0.01	0.13	0.046	
								3	0.18	< 0.01	0.13	0.06	
								7	0.098	< 0.01	0.17	0.071	
Caversham, Western Australia, 2010 (Sienna)	NA	WG	0.10	0.017	582	3	7	0	0.078	< 0.01	0.034	< 0.01	
								1	0.092	< 0.01	0.049	< 0.01	
								3	0.043	< 0.01	0.04	< 0.01	
								7	0.034	< 0.01	0.12	0.026	
			0.20	0.034	582	3	7	0	0.18	< 0.01	0.053	0.013	
								1	0.14	< 0.01	0.053	0.013	
								3	0.13	< 0.01	0.091	0.021	
								7	0.092	< 0.01	0.13	0.039	
Wallaville, Queensland, 2012 (Caribbean Queen)	NA	WG	0.10	0.02	636	3	7	0	0.038	< 0.01	0.039	0.016	UPL-1007
								1	0.05	< 0.01	0.033	0.012	
								3	0.031	< 0.01	0.048	0.019	
								7	0.05	< 0.01	0.11	0.03	
			0.20	0.03	636	3	7	0	0.058	< 0.01	0.04	0.018	
								1	0.12	< 0.01	0.051	0.026	
								3	0.084	< 0.01	0.052	0.025	
								7	0.12	< 0.01	0.19	0.089	
Caversham, Western Australia, 2011 (Sienna)	NA	WG	0.10	0.017	576	3	7	0	0.13	< 0.01	0.091	0.054	
								1	0.047	< 0.01	0.055	0.038	
								3	0.032	< 0.01	0.074	0.04	
								7	0.036	< 0.01	0.084	0.053	
			0.20	0.03	576	3	7	0	0.083	< 0.01	0.08	0.048	
								1	0.11	< 0.01	0.13	0.083	
								3	0.039	< 0.01	0.13	0.083	
								7	0.066	< 0.01	0.16	0.099	
Whitton, New South Wales, 2011 (Dubloon)	NA	WG	0.10	0.03	317	3	7	0	0.05	< 0.01	0.049	0.015	
								1	0.028	< 0.01	0.04	0.015	
								3	< 0.01	< 0.01	0.042	0.018	
								7	0.021	< 0.01	0.04	0.017	
			0.20	0.06	317	3	7	0	0.11	< 0.01	0.063	0.024	
								1	0.025	< 0.01	0.059	0.02	
								3	0.026	< 0.01	0.046	0.023	
								7	0.044	< 0.01	0.067	0.033	

Location, year (variety)	Application							Commodity	Residues (mg/kg)				Ref.
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Slovenia GAP	WG	0.05	0.005	1000	3	7	1						
Greenhouse melons													
Languedoc Le Cailar, South France, 2003	WG	0.080	0.010	788–802	3	8–9	0	Peel	0.13	< 0.01	0.07	0.05	FA-22-03-
								Pulp	0.01	< 0.01	0.04	0.02	
								Whole	0.06	< 0.01	0.05	0.03	

Location, year (variety)	Application							Commodity	Residues (mg/kg)				Ref.
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Slovenia GAP	WG	0.05	0.005	1000	3	7	1						
(Arpege) [a]								fruit					01/01
							1	Peel	0.08	< 0.01	0.06	0.07	
								Pulp	0.02	< 0.01	0.06	0.02	
								Whole fruit	0.04	< 0.01	0.06	0.04	
							2	Peel	0.09	< 0.01	0.07	0.04	
								Pulp	0.01	< 0.01	0.07	0.04	
								Whole fruit	0.04	< 0.01	0.07	0.04	
							3	Peel	0.08	< 0.01	0.06	0.05	
								Pulp	0.01	< 0.01	0.07	0.02	
								Whole fruit	0.04	< 0.01	0.07	0.03	
							7	Peel	0.06	< 0.01	0.04	0.06	
								Pulp	0.01	< 0.01	0.08	0.06	
								Whole fruit	0.03	< 0.01	0.06	0.06	
Almeria, Spain, 2003 (Cantarino) [b]	WG	0.080	0.085	900–930	3	6–8	0	Peel	0.05	< 0.01	0.02	0.08	FA-22-03-02/01
								Pulp	< 0.01	< 0.01	< 0.01	< 0.01	
								Whole fruit	0.03	< 0.01	0.01	0.04	
							1	Peel	0.07	< 0.01	0.03	0.09	
								Pulp	< 0.01	< 0.01	< 0.01	< 0.01	
								Whole fruit	0.04	< 0.01	0.02	0.05	
							2	Peel	0.03	< 0.01	0.03	0.10	
								Pulp	< 0.01	< 0.01	< 0.01	0.04	
								Whole fruit	0.02	< 0.01	0.02	0.07	
							3	Peel	0.04	< 0.01	< 0.01	0.05	
								Pulp	< 0.01	< 0.01	< 0.01	0.02	
								Whole fruit	0.02	< 0.01	< 0.01	0.04	
							7	Peel	0.01	< 0.01	0.06	0.16	
								Pulp	< 0.01	< 0.01	< 0.01	< 0.01	
								Whole fruit	0.01	< 0.01	0.03	0.08	
Veneto, Italy, 2003 (Tazio) [c]	WG	0.080–0.082	0.010	801–819	3	6–7	0	Peel	0.07	< 0.01	0.05	0.05	FA-22-03-03/01
								Pulp	< 0.01	< 0.01	0.03	0.02	
								Whole fruit	0.02	< 0.01	0.04	0.03	
							1	Peel	0.10	< 0.01	0.06	0.05	
								Pulp	< 0.01	< 0.01	0.03	0.03	
								Whole fruit	0.03	< 0.01	0.04	0.05	
							2	Peel	0.09	< 0.01	0.06	0.08	
								Pulp	< 0.01	< 0.01	0.04	0.04	
								Whole fruit	0.03	< 0.01	0.05	0.05	
							3	Peel	0.05	< 0.01	0.08	0.10	
								Pulp	0.01	< 0.01	0.04	0.06	
								Whole fruit	0.02	< 0.01	0.05	0.07	
							7	Peel	0.08	< 0.01	0.13	0.17	
								Pulp	< 0.01	< 0.01	0.04	0.05	
								Whole fruit	0.02	< 0.01	0.07	0.09	
Murcia, Spain,	WG	0.079–	0.083–	944–	3	8	1	Peel	0.04	< 0.01	0.09	0.19	FA-

Location, year (variety)	Application							Commodity	Residues (mg/kg)				Ref.
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Slovenia GAP	WG	0.05	0.005	1000	3	7	1						
2004 (Cantagrillo) [e]		0.081	0.084	964				Pulp	< 0.01	< 0.01	0.02	0.02	22-04-02
								Whole fruit	0.02	< 0.01	0.05	0.09	
Aquitaine, South France, 2004 (Amigo)	WG	0.079–0.081	0.010	788–813	3	6–9	1	Peel	0.05	< 0.01	0.14	0.09	FA-22-04-04/02
								Pulp	< 0.01	< 0.01	0.02	0.01	
								Whole fruit	0.02	< 0.01	0.06	0.03	
Field Melons													
Valencia, Spain, 2003 (Piel Sapo) [b]	WG	0.080	0.0084	932–960	3	6–8	1	Peel	0.07	< 0.01	0.03	0.10	FA-22-03-02/02
								Pulp	< 0.01	< 0.01	< 0.01	< 0.01	
								Whole fruit	0.04	< 0.01	0.02	0.05	
Emilia Romagna, Italy, 2003 (Bingo) [c]	WG	0.078–0.083	0.013	584–620	3	4–10	1	Peel	0.10	< 0.01	0.06	0.09	FA-22-03-03/02
								Pulp	< 0.01	< 0.01	0.03	0.04	
								Whole fruit	0.03	< 0.01	0.02	0.03	
Emilia Romagna, Italy, 2004 (Colorado) [d]	WG	0.080–0.082	0.013	599–618	3	7	0	Peel	0.10	< 0.01	0.06	0.05	FA-22-04-02
								Pulp	< 0.01	< 0.01	0.03	0.02	
								Whole fruit	0.04	< 0.01	0.04	0.03	
							1	Peel	0.03	< 0.01	0.07	0.05	
								Pulp	0.01	< 0.01	0.03	0.02	
								Whole fruit	0.02	< 0.01	0.05	0.03	
							2	Peel	0.05	< 0.01	0.06	0.05	
								Pulp	0.01	< 0.01	0.03	0.02	
								Whole fruit	0.03	< 0.01	0.04	0.03	
							3	Peel	0.02	< 0.01	0.07	0.05	
								Pulp	< 0.01	< 0.01	0.02	0.01	
								Whole fruit	0.01	< 0.01	0.03	0.03	
							7	Peel	0.04	< 0.01	0.10	0.07	
								Pulp	0.01	< 0.01	0.04	0.02	
								Whole fruit	0.02	< 0.01	0.06	0.04	
Poitou-Charentes, South France, 2004 (Cezanne)	WG	0.075–0.084	0.013	564–631	3	7–8	0	Peel	0.09	< 0.01	0.01	0.01	FA-22-04-04/01
								Pulp	< 0.01	< 0.01	0.01	< 0.01	
								Whole fruit	0.04	< 0.01	0.01	< 0.01	
							1	Peel	0.10	< 0.01	0.02	0.02	
								Pulp	< 0.01	< 0.01	0.01	< 0.01	
								Whole fruit	0.05	< 0.01	0.02	0.01	
							2	Peel	0.04	0.01	0.02	0.01	
								Pulp	< 0.01	< 0.01	0.01	< 0.01	
								Whole fruit	0.02	< 0.01	0.02	< 0.01	
							3	Peel	0.05	< 0.01	0.02	0.02	
								Pulp	< 0.01	< 0.01	0.01	0.01	
								Whole fruit	0.02	< 0.01	0.02	0.01	
							7	Peel	0.04	0.01	0.03	0.03	
								Pulp	< 0.01	< 0.01	0.02	0.01	
								Whole fruit	0.02	< 0.01	0.03	0.02	
Pyrenees	WG	0.049–	0.008	592–	3	7	0	Peel	0.03	Not	0.17	0.09	S-11-

Location, year (variety)	Application							Commodity	Residues (mg/kg)				Ref.
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Slovenia GAP	WG	0.05	0.005	1000	3	7	1						
Orientales, France, 2011 (Stellio)		0.050		610				Pulp	< 0.01	analysed	0.04	0.02	02600
								Whole fruit	0.02		0.09	0.05	
							1	Peel	< 0.01		0.14	0.08	
								Pulp	< 0.01		0.02	0.01	
								Whole fruit	< 0.01		0.07	0.04	
							3	Peel	0.03		0.16	0.14	
								Pulp	0.01		0.08	0.04	
								Whole fruit	0.02		0.11	0.08	
Emilia Romagna, Italy, 2011 (Bacir)	WG	0.047–0.049	0.006	780–805	3	7	0	Peel	0.02	Not analysed	0.08	0.02	
								Pulp	< 0.01		0.02	< 0.01	
								Whole fruit	< 0.01		0.05	0.01	
							1	Peel	0.03		0.08	0.03	
								Pulp	< 0.01		0.03	< 0.01	
								Whole fruit	0.01		0.05	0.02	
							3	Peel	0.01		0.11	0.04	
								Pulp	< 0.01		0.03	< 0.01	
								Whole fruit	< 0.01		0.06	0.02	
Castellon, Spain, 2011 (Sancho)	WG	0.048–0.050	0.006	760–805	3	7	0	Peel	0.13	Not analysed	0.11	0.10	
								Pulp	< 0.01		< 0.01	< 0.01	
								Whole fruit	0.07		0.06	0.05	
							1	Peel	0.08		0.13	0.14	
								Pulp	< 0.01		0.01	< 0.01	
								Whole fruit	0.04		0.07	0.08	
							3	Peel	0.05		0.13	0.14	
								Pulp	< 0.01		0.02	0.01	
								Whole fruit	0.02		0.07	0.07	
Albacete, Spain, 2011 (Piel de Sapo)	WG	0.050	0.006	798–806	3	7	0	Peel	0.01	Not analysed	0.09	0.03	
								Pulp	< 0.01		0.02	< 0.01	
								Whole fruit	0.04		0.05	0.02	
							1	Peel	0.10		0.02	0.05	
								Pulp	< 0.01		0.14	< 0.01	
								Whole fruit	0.04		0.07	0.02	
							3	Peel	0.02		0.13	0.05	
								Pulp	< 0.01		0.02	< 0.01	
								Whole fruit	< 0.01		0.07	0.03	

The experimental weight percentage ratio between peel and pulp was reported to be:

[a] 38.1% for peel and 61.9% for pulp

[b] 50.5% for peel and 49.5% for pulp

[c] 31.1 % for peel and 68.9% for pulpl

[d] 38.9 % for peel and 61.7% for pulp

[e] 41.6% for peel and 58.4% for pulp

Squash

Five independent trials were conducted on summer squash in the US during the 2001 growing season. In all trials, three foliar spray applications of a WG formulation were made with a re-treatment interval of 6–7 days. Squash was harvested 0 DALT.

The analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

Three independent trials were also conducted on pumpkin in Australia during the 2010 and 2012 growing seasons. In all trials, three foliar spray applications of a WG formulation were made at 0.10 kg ai/ha or 0.20 kg ai/ha with a re-treatment interval of 7 days. Pumpkins were harvested 0, 1, 3 and 7 DALT.

The analytical method AATM-R-165 was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The Meeting also received four independent trials conducted on pumpkin in Hungary during the 2012 growing season. In all trials, two foliar spray applications of a WG formulation were made at 0.08 kg ai/ha with a re-treatment interval of 7 days. Pumpkins were harvested 0, 1, 3 and 7 DALT.

The analytical method SOP R 700 FEJ2 was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C for all trials was up to 351 days (12 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 55.

Table 55 Residues of Flonicamid in Summer Squash Following Foliar Spray with Flonicamid 50 WG Formulation from North American Regions, Australia and Hungary

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG	0.07–0.10	0.07–0.10	100	3	7	0					71512-9
North Rose NY, 2001 (Zucchini Select)	WG	0.10	0.04	234	3	7	0	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	IB-2001-MDG-007-00-01
Rose Hill NC, 2001 (Early Prolific Straight-neck)	WG	0.10	0.05	187	3	7	0	0.031 (0.031, 0.031)	< 0.01 (< 0.01, < 0.01)	0.053 (0.050, 0.055)	0.035 (0.033, 0.036)	
Hobe Sound FL, 2001 (Rogers Hybrid)	WG	0.10	0.05	187–196	3	6–7	0	0.032 (0.032, 0.031)	< 0.01 (< 0.01, < 0.01)	0.063 (0.064, 0.062)	0.039 (0.038, 0.039)	
Arkansaw WI, 2001 (Hybrid Monet)	WG	0.10	0.05	187–196	3	6–7	0	0.042 (0.040, 0.043)	< 0.01 (< 0.01, < 0.01)	0.073 (0.081, 0.065)	0.039 (0.043, 0.035)	
							1	0.025 (0.024, 0.026)	< 0.01 (< 0.01, < 0.01)	0.080 (0.075, 0.084)	0.031 (0.028, 0.033)	
							3	0.026 (0.023, 0.028)	< 0.01 (< 0.01, < 0.01)	0.083 (0.075, 0.091)	0.034 (0.030, 0.037)	
							7	0.016 (0.015, 0.017)	< 0.01 (< 0.01, < 0.01)	0.087 (0.081, 0.092)	0.027 (0.026, 0.028)	
Madera CA, USA, 2001 (Sundance)	WG	0.10	0.04	281	3	7	0	0.031 (0.033, 0.029)	< 0.01 (< 0.01, < 0.01)	0.065 (0.076, 0.053)	0.036 (0.042, 0.030)	
AUS GAP	WG	0.05–0.10	NS	NS	3		1					

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Ballandean, South Queensland, 2010 (Butternut Large)	WG	0.10	0.015	667	3	7	0	0.12	< 0.01	0.039	0.033	UPL-1003
							1	0.079	< 0.01	0.057	0.05	
							3	0.07	< 0.01	0.059	0.05	
							7	0.029	< 0.01	0.04	0.047	
		0.20	0.03	667	3	7	0	0.27	< 0.01	0.082	0.069	
							1	0.1	< 0.01	0.084	0.097	
							3	0.086	< 0.01	0.074	0.093	
							7	0.054	< 0.01	0.12	< 0.01	
Bowen,Queensland, 2012 (Ken's Special)	WG	0.10	0.02	638	3	7	0	0.01	< 0.01	< 0.01	< 0.01	UPL-1107
							1	< 0.01	< 0.01	< 0.01	< 0.01	
							3	< 0.01	< 0.01	< 0.01	< 0.01	
							7	< 0.01	< 0.01	< 0.01	< 0.01	
		0.20	0.03	638	3	7	0	0.039	< 0.01	< 0.01	< 0.01	
							1	< 0.01	< 0.01	< 0.01	< 0.01	
							3	< 0.01	< 0.01	< 0.01	< 0.01	
							7	0.013	< 0.01	< 0.01	< 0.01	
Bowen, Queensland, 2012 (Sunset QHI)	WG	0.10	0.025	401	3	7	0	0.026	< 0.01	< 0.01	0.017	
							1	0.013	< 0.01	0.013	0.021	
							3	0.042	< 0.01	0.012	0.021	
							7	0.017	< 0.01	0.023	0.031	
		0.20	0.05	401	3	7	0	0.068	< 0.01	0.032	0.057	
							1	0.11	< 0.01	0.023	0.03	
							3	0.063	< 0.01	0.022	0.031	
							7	0.043	< 0.01	0.02	0.043	

Location, year (variety)	Application						PHI, days	Residues (mg/kg)			Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA	TFNG	
Slovenia GAP	WG	0.05	0.005–0.012	400–1000	3	7	1				12 ISK AA 0701
Kapolnasnyék, Hungary, 2012 (NS)	WG	0.08	NS		2	7	0	< 0.01	< 0.01	< 0.01	
							1	< 0.01	< 0.01	< 0.01	
							3	< 0.01	< 0.01	< 0.01	
							7	< 0.01	< 0.01	< 0.01	
Füle, Hungary, 2012 (NS)	WG	0.08	NS		2	7	0	< 0.01	< 0.01	< 0.01	
							1	< 0.01	< 0.01	< 0.01	
							3	< 0.01	< 0.01	< 0.01	
							7	< 0.01	< 0.01	< 0.01	
Vereb, Hungary, 2012 (NS)	WG	0.08	NS		2	7	0	< 0.01	< 0.01	< 0.01	
							1	< 0.01	< 0.01	< 0.01	
							3	< 0.01	< 0.01	< 0.01	
							7	< 0.01	< 0.01	< 0.01	
Székesfehérvar-Csala, Hungary, 2012 (NS)	WG	0.08	NS		2	7	0	< 0.01	< 0.01	< 0.01	
							1	< 0.01	< 0.01	< 0.01	
							3	< 0.01	< 0.01	< 0.01	
							7	< 0.01	< 0.01	< 0.01	

Fruiting vegetables, other than cucurbits

Tomatoes

Twenty-six independent trials were conducted on field tomatoes in the US between 2001 and 2010. For 12 of the trials, three foliar spray applications of a WG formulation were made at 0.10 kg ai/ha with re-treatment intervals of 6–12 days. Fourteen additional independent trials on field tomatoes were conducted in the US between 2010 and 2011 where two foliar spray applications of a SG formulation were made at 0.15 kg ai/ha with 6–8 day retreatment intervals. Three independent trials were conducted on greenhouse tomatoes in Canada and the US between 2010 and 2011 where two foliar sprays of a SG formulation were made at 0.15 kg ai/ha with 6–7 day retreatment intervals, In all trials, tomatoes were harvested 0 DALT.

The analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 382 days (19 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 56.

Table 56 Residues of Flonicamid in Field Tomatoes Following Foliar Spray with Flonicamid 50 WG and Beleaf 50SG and in Greenhouse Tomatoes Following Foliar Spray with Beleaf 50 SG in North American Regions

	Application							Residues (mg/kg)				
Location, year (variety)	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days	Flonicamid	TFNA-AM	TFNA	TFNG	Ref
Field tomatoes												
US GAP	WG/SG	0.10	0.10	100	3	7	0					
		0.15	0.15	100	2							
North Rose NY, 2001 (Floradade)	WG	0.10	0.04	234	3	7	0	0.022 (0.024, 0.019)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	IB-2001-MDG-006-00-1
							1	0.013 (0.035, 0.027)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							3	0.033 (0.034, 0.032)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.013 (0.011, 0.014)	
							7	0.021 (0.023, 0.018)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Tifton GA, 2001 (5037)	WG	0.10	0.05	187	3	7	0	0.069 (0.057, 0.081)	< 0.01 (< 0.01, < 0.01)	0.014 (0.013, 0.015)	0.014 (0.010, 0.018)	
Hobe Sound FL, 2001 (Florida 47)	WG	0.10	0.02	496–514	3	7	0	0.048 (0.047, 0.045)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01 < 0.01)	0.011 (0.011, 0.010)	
Winter Garden FL, 2001 (Better Boy)	WG	0.10	0.04	271	3	6–7	0	0.093 (0.08, 0.105)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Northwood ND, 2001 (Sheyenne)	WG	0.10	0.10	187	3	7–12	0	0.056 (0.053, 0.058)	< 0.01 (< 0.01, < 0.01)	0.010 (< 0.01, 0.010)	0.013 (0.012, 0.014)	
Vacaville CA, 2001 (3155)	WG	0.10	0.04	234	3	7	0	0.077 (0.088, 0.066)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Davis CA [a], 2001 (Brigade)	WG	0.10	0.04	234	3	6–8	0	0.082 (0.079, 0.085)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Davis CA [a], 2001 (Brigade)	WG	0.10	0.04	224–243	3	6–8	0	0.086 (0.086, 0.086)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Chowchilla CA,	WG	0.10	0.04	281	3	7	0	0.143	< 0.01	0.013	< 0.01	

	Application							Residues (mg/kg)				
Location, year (variety)	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days	Flonicamid	TFNA-AM	TFNA	TFNG	Ref
2001 (US 99)								(0.154, 0.131)	(< 0.01, < 0.01)	(0.012, 0.013)	(< 0.01, < 0.01)	
Mader[a] CA, 2001 (Celebrity)	WG	0.10	0.04	271–281	3	7	0	0.217 (0.196, 0.238)	< 0.01 (< 0.01, < 0.01)	0.011 (< 0.01, 0.011)	< 0.01 (< 0.01, < 0.01)	
Fresno CA [b], 2001 (Super Roma)	WG	0.10	0.02	187–196	3	7	0	0.088 (0.091, 0.084)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Fresno CA [b], 2001 (Shady Lady)	WG	0.10	0.02	701–711	3	6–8	0	0.232 (0.272, 0.191)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.011 (0.012, 0.010)	
Jennings FL, 2003 (Florida 47)	WG	0.10	0.10	94–95	3	7	0	0.15 (0.15, 0.14)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	P-3695
							1	0.09 (0.08, 0.09)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							3	0.06 (0.06, 0.05)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							7	0.03 (0.03, 0.03)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.02 (0.02, 0.02)	
	SG w/o surfactant						0	0.13 (0.12, 0.014)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							1	0.09 (0.11, 0.07)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							3	0.06 (0.08, 0.04)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							7	0.04 (0.04, 0.04)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.02 (0.02, 0.02)	
	SG with surfactant						0	0.12 (0.10, 0.13)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							1	0.09 (0.08, 0.09)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							3	0.08 (0.09, 0.06)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
							7	0.05 (0.04, 0.05)	< 0.01 (< 0.01,	< 0.01 (< 0.01,	0.03 (0.03, 0.03)	

	Application							Residues (mg/kg)				
Location, year (variety)	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days	Flonicamid	TFNA-AM	TFNA	TFNG	Ref
									< 0.01)	< 0.01)		
Maricopa AZ, 2010 (Round Red)	SG	0.16	0.05	290–299	2	7	0	0.063 (0.037, 0.088)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.01 (0.01, 0.01)	
Davis CA [c], 2010 (Sun 6366)	SG	0.15	0.05	299	2	8	0	0.118 (0.120, 0.115)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Davis CA [c], 2010 (Shady Lady)	SG	0.15	0.05	299	2	7	0	0.083 (0.078, 0.087)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Parlier CA [d], 2010 (H3155)	SG	0.16	0.04–0.08	196–206	2	7	0	0.066 (0.065, 0.067)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.011 (0.012, 0.010)	
Parlier CA [d], 2010 (Cherry Grande)	SG	0.15–0.16	0.04	383–393	2	7	0	0.103 (0.103, 0.103)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.017 (0.017, 0.017)	
Riverside [e] CA, 2010 (Sun 6788)	SG	0.15–0.16	0.04	374–383	2	7	0	0.191 (0.187, 0.194)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.014 (0.014, 0.014)	
Riverside [e] CA, 2010 (Celebrity)	SG	0.15–0.16	0.04	468–477	2	7	0	0.056 (0.057, 0.055)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Holtville CA, 2010 (Shady Lady)	SG	0.15	0.05	299–309	2	6	0	0.117 (0.116, 0.118)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.014 (0.014, 0.013)	IR-4 PR No. 08556
Holtville CA, 2011 (Hypeel 4S)	SG	0.15	0.04	337–346	2	8	0	0.110 (0.110, 0.109)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Citra FL, 2010 (BHN 602)	SG	0.16	0.04	383	2	8	0	0.048 (0.049, 0.047)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Tifton GA, 2010 (Sun Gold F1)	SG	0.15	0.04	393–402	2	6	0	0.102 (0.100, 0.103)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.019 (0.019, 0.019)	
Salisbury MD, 2010 (Sunbrite)	SG	0.15	0.05	309	2	6	0	0.131 (0.131, 0.130)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Clonton NC, 2010 (Supersweet 100)	SG	0.15	0.04	412	2	7	0	0.147 (0.148, 0.145)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.050 (0.050, 0.050)	
Las Cruces [f] NM, 2010 (Roma)	SG	0.15	0.03	468–486	2	6	0	0.078 (0.077, 0.078)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01 < 0.01)	
Las Cruces [f] NM, 2010 (Celebrity)	SG	0.15	0.03	187	2	6	0	0.074 (0.073, 0.074)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Freeville NY, 2010	SG	0.15–	0.03	552–561	2	7	0	0.050	< 0.01	< 0.01	< 0.01	

	Application							Residues (mg/kg)				
Location, year (variety)	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days	Flonicamid	TFNA-AM	TFNA	TFNG	Ref
(Marianna)		0.16						(0.049, 0.050)	(< 0.01, < 0.01)	(< 0.01, < 0.01)	(< 0.01, < 0.01)	
Fremont OH, 2010 (Mountain Pride)	SG	0.15	0.03	421–440	2	7	0	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Arlington OH, 2010 (Better Boy)	SG	0.16	0.08	187	2	7	0	0.070 (0.070, 0.071)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Greenhouse Tomatoes												
GAP	SG	0.10–0.15	0.10–0.15	100	2	7	0					
Parlier CA, 2010 (Trust)	SG	0.15	0.03	458–477	2	7	0	0.058 (0.060, 0.056)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Citra FL, 2011 (BHN 268)	SG	0.15	0.05	281	2	7	0	0.037 (0.037, 0.036)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.025 (0.025, 0.025)	
Harrow ON, CA, 2010 (Macarena)	SG	0.15	0.02	1000–1007	2	7	0	0.049 (0.053, 0.044)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.014 (0.013, 0.014)	
							3	0.050 (0.050, 0.050)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.023 (0.023, 0.022)	
							7	0.040 (0.036, 0.044)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.043 (0.043, 0.043)	
							10	0.041 (0.039, 0.043)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.069 (0.066, 0.071)	

[a] The last applications made at each site were on the same day and the varieties were the same, rendering the trials dependent

[b] The last applications made at each site were 19 days apart, therefore, trials were considered independent

[c] The last applications made at each site were 5 days apart, and varieties were not sufficiently different to render the trials independent

[d] The last applications made at each site were 8 days apart, and the tomato variety H3155 could not be identified, therefore, trials were considered dependent

[e] The last applications made at each site were 9 days apart, and varieties were not sufficiently different to render the trials independent

[f] The last applications were made on the same day and varieties were not sufficiently different to render the trials independent.

Bell peppers

Six independent trials were conducted on field bell peppers in the US in 2001 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–7 days. Bell peppers were harvested 0 DALT.

The analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 382 days (19 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 59.

Table 57 Residues of Flonicamid in Bell Peppers Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.10	0.10	100	3	7	0					
		0.15	0.15	100	2							
Rose Hill NC, 2001 (Jupiter)	WG	0.10	0.05	187	3	7	0	<u>0.058</u> (0.056, 0.059)	< 0.01 (< 0.01, < 0.01)	0.070 (0.071, 0.068)	0.030 (0.029, 0.030)	IB-2001-MDG-006-00-01
Hobe Sound FL, 2001 (Wizard)	WG	0.10	0.02	571–589	3	6–7	0	<u>0.057</u> (0.052, 0.062)	< 0.01 (< 0.01, < 0.01)	0.068 (0.064, 0.072)	0.031 (0.029, 0.032)	
Arkansaw WI, 2001 (Better Bell IMP.)	WG	0.10	0.05	187	3	6–7	0	<u>0.056</u> (0.061, 0.051)	< 0.01 (< 0.01, < 0.01)	0.070 (0.077, 0.062)	0.031 (0.035, 0.027)	
East Bernard TX, 2001 (Capistrano)	WG	0.10	0.08–0.09	112–131	3	7	0	0.055 (0.056, 0.053)	< 0.01 (< 0.01, < 0.01)	0.034 (0.032, 0.035)	0.049 (0.043, 0.055)	
							1	<u>0.113</u> (0.118, 0.108)	< 0.01 (< 0.01, < 0.01)	0.039 (0.040, 0.037)	0.079 (0.083, 0.074)	
							3	0.099 (0.105, 0.093)	< 0.01 (< 0.01, < 0.01)	0.047 (0.050, 0.044)	0.115 (0.122, 0.107)	
							7	0.051 (0.048, 0.054)	< 0.01 (< 0.01, < 0.01)	0.060 (0.054, 0.065)	0.144 (0.135, 0.153)	
Suisun CA, 2001 (variety not available)	WG	0.10	0.04	234	3	7	0	<u>0.104</u> (0.107, 0.101)	< 0.01 (< 0.01, < 0.01)	0.045 (0.049, 0.041)	0.038 (0.039, 0.037)	
Fresno CA, 2001 (Jupiter)	WG	0.10	0.015	683–701	3	7	0	<u>0.107</u> (0.105, 0.108)	< 0.01 (< 0.01, < 0.01)	0.037 (0.036, 0.038)	0.038 (0.037, 0.039)	

Non-bell Peppers

Two independent trials were conducted on field <u>non-bell peppers</u> in the US in 2001 where three foliar spray applications of a WG formulation were made with a re-treatment interval of 7 days. Non-bell peppers were harvested 0 DALT.

The analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 382 days (19 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 58.

Table 58 Residues of Flonicamid in Non-bell Peppers Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/ SG	0.10	0.10	100	3	7	0					
		0.15	0.15	100	2							
East Bernard TX, 2001 (Big Jim)	WG	0.10	0.08	122	3	7	0	0.219 (0.215, 0.223)	< 0.01 (< 0.01, < 0.01)	0.028 (0.029, 0.027)	0.041 (0.041, 0.040)	IB-2001-MDG-006-00-01
Suisun CA [a], 2001 (Anaheim)	WG	0.10	0.04	234	3	7–9	0	0.210 (0.204, 0.215)	< 0.01 (< 0.01, < 0.01)	0.030 (0.030, 0.030)	0.040 (0.039, 0.040)	
Suisun CA [a], 2001 (Anaheim)	WG	0.10	0.04	711–720	3	7	0	0.205 (0.208, 0.202)	< 0.01 (< 0.01, < 0.01)	0.031 (0.030, 0.031)	0.038 (0.036, 0.039)	

[a] The last applications were made on the same day and the varieties were the same, rendering the trials dependent.

Leafy vegetables (including Brassica leafy vegetables)

Head lettuce

Six independent trials were conducted on head lettuce in the US in 2002 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–8 days. Head lettuce was harvested 0 DALT.

Method P-3575, a modified version of analytical method P-3561, was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 147 days (ca. 5 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 59.

Table 59 Residues of Flonicamid in Head Lettuce Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Matrix	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/ SG	0.07–0.10	0.07–0.30	30–100	3	7	0						
Germansville PA, 2002 (Sun Devil)	WG	0.10	0.11	94	3	6–7	0	w/wrapper leaves	0.493 (0.392, 0.593)	< 0.01 (< 0.01, < 0.01)	0.021 (0.024, 0.018)	0.026 (0.026, 0.025)	Buser, J.W. and Chen, A.W., 2003
								w/out wrapper leaves	0.027 (0.028, 0.025)	< 0.01 (< 0.01, < 0.01)	0.012 (0.012, 0.011)	< 0.01 (< 0.01, < 0.01)	
Belle Glade FL, 2002 (Iceberg 35x110)	WG	0.10	0.10	92–98	3	7	0	w/wrapper leaves	0.617 (0.649, 0.584)	0.012 (0.013, < 0.01)	0.022 (0.020, 0.023)	0.025 (0.027, 0.023)	
								w/out wrapper leaves	0.024 (0.029, 0.019)	< 0.01 (< 0.01, < 0.01)	0.012 (0.013, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Lagurta AZ, 2002 (Desert Spring)	WG	0.10	0.11	94–96	3	7	0	w/wrapper leaves	0.518 (0.565, 0.471)	< 0.01 (< 0.01, < 0.01)	0.018 (0.021, 0.015)	0.023 (0.023, 0.022)	
								w/out wrapper	0.027 (0.026,	< 0.01 (< 0.01,	0.012 (0.012,	< 0.01 (< 0.01,	

Location, year (variety)	Application						DALT, days	Matrix	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
								leaves	0.028)	< 0.01)	0.011)	< 0.01)	
Visalia CA, 2002 (Great Lakes 659:)	WG	0.10	0.11	94	3	7–8	0	w/wrapper leaves	0.584 (0.723, 0.445)	< 0.01 (< 0.01, < 0.01)	0.023 (0.027, 0.018)	0.028 (0.031, 0.025)	
							0	w/out wrapper leaves	0.027 (0.028, 0.026)	< 0.01 (< 0.01, < 0.01)	0.014 (0.015, 0.013)	< 0.01 (< 0.01, < 0.01)	
							1	w/wrapper leaves	0.013 (0.038, 0.023)	< 0.01 (< 0.01, < 0.01)	0.028 (0.032, 0.024)	0.010 (0.010, < 0.01)	
							3		0.013 (0.012, 0.014)	< 0.01 (< 0.01, < 0.01)	0.014 (0.012, 0.015)	< 0.01 (< 0.01, < 0.01)	
							7		< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.022 (0.021, 0.023)	< 0.01 (< 0.01, < 0.01)	
Bard CA, 2002 (Green Lightning)	WG	0.10	0.11	93–95	3	7	0	w/wrapper leaves	0.431 (0.509, 0.353)	< 0.01 (< 0.01, < 0.01)	0.022 (0.026, 0.018)	0.026 (0.030, 0.022)	
								w/out wrapper leaves	0.033 (0.030, 0.035)	< 0.01 (< 0.01, < 0.01)	0.013 (0.012, 0.014)	< 0.01 (< 0.01, < 0.01)	
Greenfield CA, 2002 (Big Ben)	WG	0.10	0.11	93–97	3	7	0	w/wrapper leaves	0.394 (0.386, 0.402)	< 0.01 (< 0.01, < 0.01)	0.027 (0.034, 0.020)	0.032 (0.042, 0.021)	
								w/out wrapper leaves	0.028 (0.028, 0.027)	< 0.01 (< 0.01, < 0.01)	0.014 (0.013, 0.014)	< 0.01 (< 0.01, < 0.01)	

Leaf lettuce

Six independent trials were conducted on leaf lettuce in the US in 2002 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–7 days. Leaf lettuce was harvested 0 DALT. Side-by-side trials were also conducted in 2003 on Cos lettuce to compare the WG formulation to the SG formulation (with and without surfactant). The same use pattern was applied as that of the six trials.

A modified version of analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 172 days (ca. 6 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 60.

Table 60 Residues of Flonicamid in Leaf Lettuce Following Foliar Spray with Flonicamid 50 WG or Beleaf 50 SG in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.07–0.30	30–100	3	7	0					
Germansville PA, 2002 (New Fire)	WG	0.10	0.11	94	3	6	0	2.525 (2.741, 2.309)	0.015 (0.017, 0.013)	0.013 (0.014, 0.011)	0.036 (0.038, 0.034)	P-3575
Belle Glade FL, 2002 (Green Leaf Two Star)	WG	0.10	0.10	92–101	3	7	0	3.113 (3.211, 3.014)	0.017 (0.017, 0.016)	0.014 (0.014, 0.014)	0.042 (0.041, 0.042)	
Maricopa	WG	0.10	0.11	94	3	7	0	3.056	0.016	0.014	0.038	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
AZ, 2001 (Ventana)								(3.051, 3.061)	(0.015, 0.017)	(0.014, 0.014)	(0.037, 0.039)	
Visalia CA, 2002 (Salad Bowl)	WG	0.10	0.11	94	3	7	0	1.936 (1.738, 2.134)	0.023 (0.019, 0.026)	0.028 (0.024, 0.032)	0.100 (0.086, 0.113)	
Visalia CA, 2002 (Salad Bowl)	WG	0.10	0.11	94	3	7	1	1.821 (1.764, 1.877)	0.029 (0.028, 0.030)	0.068 (0.087, 0.049)	0.115 (0.134, 0.095)	
Visalia CA, 2002 (Salad Bowl)	WG	0.10	0.11	94	3	7	3	1.211 (1.058, 1.363)	0.028 (0.023, 0.033)	0.042 (0.039, 0.045)	0.065 (0.078, 0.051)	
Visalia CA, 2002 (Salad Bowl)	WG	0.10	0.11	94	3	7	7	0.374 (0.348, 0.399)	0.013 (0.013, 0.013)	0.047 (0.053, 0.040)	0.061 (0.067, 0.054)	
Bard CA, 2001 (Marin)	WG	0.10	0.11	92–95	3	7	0	2.182 (2.713, 1.650)	0.017 (0.017, 0.016)	0.017 (0.021, 0.012)	0.039 (0.047, 0.031)	
Greenfield CA, 2001 (Green Towers)	WG	0.10	0.11	93–96	3	7	0	2.668 (2.257, 3.078)	0.018 (0.018, 0.018)	0.014 (0.016, 0.012)	0.040 (0.040, 0.040)	
Side-by-side trials												
Jennings FL, 2003 (Romain TA-11 Guzman)	WG	0.10	0.10	97	3	7	0	2.59 (2.56, 2.61)	0.04 (0.04, 0.03)	0.05 (0.05, 0.05)	0.06 (0.06, 0.06)	P-3695
Jennings FL, 2003 (Romain TA-11 Guzman)	WG	0.10	0.10	97	3	7	1	2.55 (2.50, 2.59)	0.06 (0.05, 0.06)	0.05 (0.05, 0.05)	0.08 (0.07, 0.08)	P-3695
Jennings FL, 2003 (Romain TA-11 Guzman)	WG	0.10	0.10	97	3	7	3	2.22 (1.90, 2.53)	0.04 (0.04, 0.04)	0.04 (0.03, 0.04)	0.05 (0.05, 0.05)	P-3695
Jennings FL, 2003 (Romain TA-11 Guzman)	WG	0.10	0.10	97	3	7	7	0.70 (0.71, 0.69)	0.08 (0.07, 0.08)	0.06 (0.06, 0.06)	0.14 (0.13, 0.014)	P-3695
Jennings FL, 2003 (Romain TA-11 Guzman)	SG	0.10	0.10	97	3	7	0	2.32 (2.41, 2.22)	0.02 (0.02, 0.02)	0.04 (0.04, 0.04)	0.04 (0.04, 0.04)	P-3695
Jennings FL, 2003 (Romain TA-11 Guzman)	SG	0.10	0.10	97	3	7	1	1.94 (1.92, 1.95)	0.10 (0.10, 0.10)	0.05 (0.05, 0.05)	0.13 (0.13, 0.12)	P-3695
Jennings FL, 2003 (Romain TA-11 Guzman)	SG	0.10	0.10	97	3	7	3	2.07 (1.84, 2.29)	0.04 (0.04, 0.04)	0.04 (0.03, 0.05)	0.07 (0.06, 0.08)	P-3695
Jennings FL, 2003 (Romain TA-11 Guzman)	SG	0.10	0.10	97	3	7	7	0.38 (0.35, 0.41)	0.04 (0.03, 0.04)	0.04 (0.04, 0.04)	0.06 (0.05, 0.07)	P-3695
Jennings FL, 2003 (Romain TA-11 Guzman)	SG (with surfactant)	0.10	0.10	97	3	7	0	2.71 (2.80, 2.61)	0.02 (0.02, 0.02)	0.05 (0.05, 0.04)	0.05 (0.05, 0.05)	P-3695
Jennings FL, 2003 (Romain TA-11 Guzman)	SG (with surfactant)	0.10	0.10	97	3	7	1	1.82 (1.79, 1.84)	0.06 (0.06, 0.06)	0.04 (0.04, 0.04)	0.08 (0.09, 0.09)	P-3695
Jennings FL, 2003 (Romain TA-11 Guzman)	SG (with surfactant)	0.10	0.10	97	3	7	3	1.55 (1.41, 1.68)	0.05 (0.05, 0.05)	0.04 (0.03, 0.04)	0.08 (0.08, 0.08)	P-3695
Jennings FL, 2003 (Romain TA-11 Guzman)	SG (with surfactant)	0.10	0.10	97	3	7	7	0.50 (0.47, 0.53)	0.05 (0.04, 0.05)	0.05 (0.05, 0.05)	0.09 (0.09, 0.09)	P-3695

Spinach

Six independent trials were conducted on spinach in the US in 2001 and 2002 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–9 days. Plants were harvested 0 DALT.

Method P-3575, a modified version of analytical method P-3561M, was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 131 days (ca 4 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 61.

Table 61 Residues of Flonicamid in Spinach Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.07–0.30	30–100	3	7	0					
Baptistown NJ, 2002 (Tyee)	WG	0.10	0.11	94	3	6–9	0	6.967 (7.196, 6.737)	0.139 (0.134, 0.144)	0.402 (0.401, 0.402)	0.251 (0.247, 0.254)	P-3575
							1	3.062 (3.030, 3.094)	0.051 (0.053, 0.048)	0.218 (0.225, 0.210)	0.015 (0.118, 0.112)	
							3	2.049 (2.116, 1.981)	0.088 (0.089, 0.086)	0.314 (0.323, 0.304)	0.154 (0.159, 0.148)	
							7	0.580 (0.645, 0.514)	0.022 (0.028, 0.015)	0.181 (0.204, 0.158)	0.081 (0.092, 0.070)	
Suffolk VA, 2002 (Tyee)	WG	0.10	0.10	95–100	3	7	0	6.586 (6.073, 7.099)	0.150 (0.143, 0.156)	0.357 (0.353, 0.361)	0.262 (0.248, 0.275)	
Raymondville TX, 2002 (Skookum)	WG	0.10	0.11	94	3	7	0	4.820 (4.160, 5.480)	0.128 (0.116, 0.139)	0.296 (0.277, 0.315)	0.221 (0.204, 0.238)	
Wellington CO, 2002 (Unipack)	WG	0.10	0.11	94	3	7	0	4.855 (5.022, 4.687)	0.052 (0.054, 0.050)	0.132 (0.127, 0.136)	0.167 (0.163, 0.170)	
Yuma AZ, 2001 (RSP 6200)	WG	0.10	0.11	94	3	7	0	5.727 (6.000, 5.454)	0.149 (0.138, 0.160)	0.343 (0.332, 0.354)	0.251 (0.243, 0.259)	
San Ardo CA, 2001 (Bolero)	WG	0.10	0.11	94	3	7	0	5.713 (5.461, 5.965)	0.149 (0.149, 0.149)	0.332 (0.314, 0.350)	0.255 (0.280, 0.230)	

Radish leaves

Five independent trials were conducted on radish leaves in the US in 2003 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–9 days. Leaves were harvested 2 DALT.

The analytical method P-3561M was used to analyse all samples of radish roots and radish tops. The LOQ for radish leaves was determined to be 0.05 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 517 days (ca. 17 months) for radish leaves. Concurrent storage stability data show that the residues are stable for up to 464 days (ca. 15 months). The results are summarized in Table 62.

Table 62 Residues of Flonicamid in Radish Leaves Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Matrix	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
GAP	WG	0.07–0.10	0.07–0.10	100	3	7	3						
Salinas CA, 2003 (Altaglobe)	WG	0.10	0.02	533–542	3	6–7	2	Tops	3.1 (3.2, 2.9)	0.068 (0.070, 0.066)	0.051 (0.05, 0.051)	0.20 (0.20, 0.20)	08753
Citra FL [a], 2003 (Cabernet F1)	WG	0.10	0.04	281	3	6–7	2	Tops	8.5 (8.8, 8.2)	0.47 (0.46, 0.48)	0.16 (0.14, 0.18)	0.70 (0.71, 0.68)	
Citra FL [a], 2003 (Cabernet F1)	WG	0.10	0.04	281–290	3	7–8	2	Tops	5.7 (6.2, 5.2)	0.30 (0.35, 0.25)	0.17 (0.22, 0.12)	0.33 (0.36, 0.29)	
Bridgeton NJ, 2003 (Rebel)	WG	0.10	0.04	243–262	3	8–9	2	Tops	5.4 (5.2, 5.6)	0.098 (0.096, 0.10)	< 0.05 (< 0.05, < 0.05)	0.12 (0.12, 0.12)	
Willard OH, 2003 (Cabernet)	WG	0.10	0.02–0.03	402–430	3	6–8	4	Tops	0.21 (0.23, 0.18)	< 0.05 (< 0.05, < 0.05)	< 0.05 (< 0.05, < 0.05)	0.069 (0.074, 0.063)	

[a] The last applications at each trial site were made 21 days apart, rendering the trials independent.

Mustard Greens

Eight trials were conducted on mustard greens in the US in 2003 and 2004 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–7 days. Mustard green leaves were harvested 0 DALT.

A modified version of analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 214 days (7 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 63.

Table 63 Residues of Flonicamid in Mustard Greens Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.07–0.30	100	3	7	0					
Goochland VA, 2003 (Southern Giant)	WG	0.10	0.10	101–107	3	6–8	0	6.873 (6.945, 6.801)	0.047 (0.047, 0.047)	0.411 (0.411, 0.411)	0.907 (0.911, 0.902)	
Senatobia MS, 2003 (Florida Broadleaf)	WG	0.10	0.10	93–94	3	7	0	8.307 (8.097, 8.517)	0.071 (0.064, 0.077)	0.136 (0.131, 0.141)	1.341 (1.304, 1.378)	P-3679
Ellendale MN, 2003 (Southern	WG	0.10	0.11	89–93	3	7	0	2.037 (2.147, 1.926)	< 0.010 (< 0.010, < 0.010)	0.044 (0.051, 0.037)	0.163 (0.182, 0.144)	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Giant Curled)												
Eakly OK, 2003 (Florida Broadleaf)	WG	0.10	0.10	94–95	3	6–7	0	3.965 (3.669, 4.260)	0.046 (0.043, 0.049)	0.184 (0.160, 0.207)	0.401 (0.361, 0.440)	
Visalia CA, 2003 (Florida Broadleaf)	WG	0.10	0.11	91–92	3	7	0	2.209 (1.813, 2.605)	0.031 (0.026, 0.035)	0.070 (0.060, 0.080)	0.418 (0.359, 0.477)	
							1	1.643 (1.598, 1.688)	0.033 (0.030, 0.035)	0.052 (0.049, 0.055)	0.340 (0.307, 0.373)	
							3	1.136 (0.989, 1.283)	0.040 (0.036, 0.044)	0.057 (0.055, 0.059)	0.417 (0.395, 0.438)	
							7	0.369 (0.388, 0.350)	0.018 (0.027, < 0.010)	0.082 (0.078, 0.086)	0.412 (0.425, 0.398)	
Chula GA, 2004 (Broadleaf)	WG	0.10	0.10	96–102	3	7	0	4.401 (4.468, 4.334)	< 0.010 (< 0.010, < 0.010)	0.041 (0.77, < 0.01)	0.448 (0.460, 0.435)	P-3764
Jennings FL, 2004 (Curly Leaf)	WG	0.10	0.10	94–102	3	6–7	0	4.778 (5.123, 4.433)	< 0.010 (< 0.010, < 0.010)	0.069 (0.066, 0.072)	0.416 (0.418, 0.413)	
Visalia CA, 2004 (Broadleaf)	WG	0.10	0.10	95	3	7	0	4.909 (5.042, 4.775)	< 0.010 (< 0.010, < 0.010)	0.084 (0.072, 0.096)	0.482 (0.496, 0.467)	

ND = Not detected

Root and tuber vegetables

Potato tubers

Sixteen independent trials were conducted on potatoes in the US in 2001 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–8 days. Potato tubers were harvested 0 DALT.

In Australia, four independent trials were conducted in 2010 and 2012 where two foliar spray applications of a WG formulation were made at 0.08 kg ai/ha or 0.16 kg ai/ha with 7–9 day re-treatment intervals. Potato tubers were harvested 14 DALT.

The analytical method P-3561M was used to analyse all samples collected from the US trials while method AATM-R-165 was used for the Australian trials. For both methods, the LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 315 days (ca 11 months). Storage stability data on high starch content commodities show that the residues are stable for at least 23 months. The results are summarized in Tables 64.

Table 64 Residues of Flonicamid in Potato Tubers Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions and with UPI-220 500 WG Formulation in Australia

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/ SG	0.07–0.10	0.07–0.30	30–100	3	7	7					
North Rose NY, 2001 (NY-79)	WG	0.12	0.044	234	3	7	0	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.014 (0.012, 0.015)	< 0.01 (< 0.01, < 0.01)	IB-2001-MDG-002-00-01
							1	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.017 (0.016, 0.017)	< 0.01 (< 0.01, < 0.01)	
							3	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.012 (0.012, 0.012)	< 0.01 (< 0.01, < 0.01)	
							7	<u>< 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.033 (0.034, 0.032)	0.059 (0.060, 0.058)	
							14	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.022 (0.019, 0.025)	< 0.01 (< 0.01, < 0.01)	
Germansville PA, 2001 (Andover)	WG	0.10	0.04	281–290	3	7	7	<u>< 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.035 (0.031, 0.039)	0.068 (0.055, 0.080)	
Suffolk VA, 2001 (Superior)	WG	0.10	0.10–0.11	94–103	3	7	7	<u>< 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.026 (0.027, 0.025)	< 0.01 (< 0.01, < 0.01)	
Hobe Sound FL, 2001 (Red LaSoda)	WG	0.10	0.03	346–355	3	7	7	<u>< 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.021 (0.022, 0.019)	0.014 (0.015, 0.013)	
Northwood ND, 2001 (Dark Red Norland)	WG	0.10	0.05	187	3	6–7	7	<u>< 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.019 (0.020, 0.017)	0.015 (0.016, 0.013)	
Bygland MN, 2001 (Dark Red Norland)	WG	0.10	0.05	187–196	3	7	7	<u>< 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.021 (0.020, 0.021)	0.014 (0.014, 0.014)	
Arkansaw WI, 2001 (Russet Burbank)	WG	0.10	0.06	187	3	7	7	<u>< 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.023 (0.024, 0.022)	< 0.01 (< 0.01, < 0.01)	
Theilman MN, 2001 (Russet Norkotah)	WG	0.10	0.05–0.06	187–196	3	7–8	7	<u>0.015</u> (0.020, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.049 (0.049, 0.049)	0.01 (0.01, < 0.01)	
Centre CO, 2001 (Norkotah)	WG	0.10	0.06	187	3	7	7	<u>< 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.046 (0.039, 0.053)	0.010 (< 0.01, 0.010)	
Stockton CA, 2001 (Cal White)	WG	0.10	0.04	234	3	7	7	<u>< 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.028 (0.025, 0.031)	0.016 (0.014, 0.017)	
Ephrata WA, 2001 (Russet Burbank)	WG	0.10–0.11	0.05–0.06	187–196	3	7	7	<u>< 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.028 (0.028, 0.028)	0.016 (0.015, 0.016)	
Moses Lake WA, 2001 (Russet Burbank)	WG	0.10	0.11	94	3	7	7	<u>< 0.01</u> (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.035 (0.034, 0.036)	0.020 (0.020, 0.020)	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
American Falls ID, 2001 (Russet Burbank)	WG	0.10	0.05	196	3	6–7	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.034 (0.033, 0.035)	0.020 (0.020, 0.020)	
Minidoka ID, 2001 (Russet Burbank)	WG	0.10	0.06	159–168	3	7	0	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.013 (0.013, 0.013)	< 0.01 (< 0.01, < 0.01)	
							1	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.019 (0.015, 0.022)	< 0.01 (< 0.01, < 0.01)	
							3	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.013 (0.013, 0.013)	< 0.01 (< 0.01, < 0.01)	
							7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.023 (0.021, 0.025)	< 0.01 (< 0.01, < 0.01)	
							14	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.019 (0.025, 0.012)	< 0.01 (< 0.01, < 0.01)	
Herminston OR, 2001 (Russet Burbank)	WG	0.10	0.04	281–290	3	7	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.037 (0.038, 0.036)	0.016 (0.016, 0.015)	
Jerome ID, 2001 (Russet Burbank)	WG	0.10	0.06	168–178	3	6–8	7	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.047 (0.042, 0.052)	0.023 (0.021, 0.025)	
AUS GAP	WG	0.07–0.10	NS	NS	2	14	14					
Gembrook, Victoria 2010 (Sebago)	WG	0.08	0.02	503–507	2	7	14	< 0.01	< 0.01	< 0.01	< 0.01	UPL-1001 and 1109
Gembrook Victoria, 2010 (Sebago)	WG	0.16	0.03	503–507	2	7	14	< 0.01	< 0.01	0.015	< 0.01	
Morgan South Australia, 2011 (Ruby Loo's)	WG	0.08	0.03	301–307	2	7	14	< 0.01	< 0.01	< 0.01	< 0.01	
Morgan South Australia, 2011 (Ruby Loo's)	WG	0.16	0.05	3 01–307	2	7	14	< 0.01	< 0.01	< 0.01	< 0.01	
Charleston South Carolina, 2011 (Coliban)	WG	0.08	0.02	402–407	2	7	14	< 0.01	< 0.01	< 0.01	< 0.01	
Charleston South Carolina, 2011 (Coliban)	WG	0.16	0.04	402–407	2	7	14	< 0.01	< 0.01	0.013	< 0.01	
Bundaberg Queensland, 2012 (Sebago)	WG	0.08	0.014	562–581	2	7	14	< 0.01	< 0.01	0.017	0.012	
Bundaberg	WG	0.16–0.17	0.03	581–599	2	7	14	< 0.01	< 0.01	0.026	0.016	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Queensland, 2012 (Sebago)												
Boneo Victoria, 2012 (Exton)	WG	0.08	0.01	603–609	2	9	14	< 0.01	< 0.01	< 0.01	< 0.01	
Boneo Victoria, 2012 (Exton)	WG	0.16	0.03	599–618	2	9	14	< 0.01	< 0.01	0.023	< 0.01	

Carrot roots

Eight independent trials were conducted on carrots in the US in 2003 where three foliar spray applications of a WG formulation with re-treatment intervals of 6–8 days. Carrot roots were harvested 6–8 DALT.

The analytical method P-3561M was used to analyse all samples. The LOQ of flonicamid was determined to be 0.02 mg/kg while the LOQ for all metabolites was determined to be 0.05 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 462 days (ca 15 months). Storage stability data on high starch content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 65.

Table 65 Residues of Flonicamid in Carrot Roots Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.07–0.30	30–100	3	7	3					
Salinas CA, 2003 (Mokum-Raw)	WG	0.09–0.10	0.020	449–542	3	7–8	7	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	< 0.050 (< 0.050, < 0.050)	0.060 (0.064, 0.056)	08754
Porterville CA, 2003 (Denver's Half Long 126)	WG	0.10	0.04	224–290	3	6–7	7	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.100 (0.122, 0.077)	< 0.050 (< 0.050, < 0.050)	
Parlier CA, 2003 (Denver's Half Long 126)	WG	0.10	0.04	234–243	3	7	1	0.022 (0.024, 0.020)	< 0.050 (< 0.050, < 0.050)	0.054 (0.052, 0.056)	0.050 (0.050, < 0.050)	
							3	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.071 (0.061, 0.080)	0.052 (< 0.050, 0.054)	
							6	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.092 (0.110, 0.074)	0.052 (0.054, < 0.050)	
							13	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.106 (0.099, 0.112)	0.070 (0.070, 0.070)	
Holtville CA, 2004 (Caropak)	WG	0.10	0.03	355–374	3	7	6	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	< 0.050 (< 0.050, < 0.050)	< 0.050 (< 0.050, < 0.050)	
Citra FL, 2003 (Triple Play 58	WG	0.10	0.04	281–299	3	7	7	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.051 (0.052, < 0.050)	< 0.050 (< 0.050, < 0.050)	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
SMS)												
Willard OH, 2003 (Scarlet Nantes)	WG	0.10	0.02–0.03	374–430	3	7	7	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.059 (0.058, 0.060)	< 0.050 (< 0.050, < 0.050)	
Weslaco TX, 2003 (Six Pence)	WG	0.10	0.04–0.05	206–224	3	6–7	1	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	< 0.05 (< 0.050, < 0.050)	0.086 (0.086, 0.086)	
							3	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.061 (0.059, 0.063)	0.091 (0.095, 0.086)	
							6	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.050 (0.050, < 0.050)	0.163 (0.178, 0.148)	
							13	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	< 0.050 (< 0.050, < 0.050)	0.124 (0.116, 0.132)	
Moxee WA, 2003 (Enterprise)	WG	0.10	0.03	299–327	3	6–7	8	< 0.020 (< 0.020, < 0.020)	< 0.050 (< 0.050, < 0.050)	0.072 (0.066, 0.077)	< 0.050 (< 0.050, < 0.050)	

Radish roots

Five independent trials were conducted on radish roots in the US in 2003 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–9 days. Radish roots were harvested 2 DALT.

The analytical method P-3561M was used to analyse all samples of radish roots. The LOQ for radish roots was determined to be 0.02 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 434 days (ca 14 months) for radish roots. Concurrent storage stability data show that the residues are stable for up to 464 days (ca. 15 months). The results are summarized in Table 66.

Table 66 Residues of Flonicamid in Radish Roots Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Matrix	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG	0.07–0.10	0.07–0.10	100	3	7	3						
Salinas CA, 2003 (Altaglobe)	WG	0.10	0.02	533–542	3	6–7	2	Roots	0.13 (0.13, 0.13)	< 0.02 (< 0.02, < 0.02)	0.042 (0.044, 0.040)	< 0.02 (< 0.02, < 0.02)	08753
Citra FL, 2003 (Cabernet F1) [a]	WG	0.10	0.04	281	3	6–7	2	Roots	0.21 (0.25, 0.17)	< 0.02 (< 0.02, < 0.02)	0.078 (0.067, 0.088)	0.056 (0.066, 0.046)	
Citra FL, 2003 (Cabernet F1) [a]	WG	0.10	0.04	281–290	3	7–8	2	Roots	0.075 (0.080, 0.070)	< 0.02 (< 0.02, < 0.02)	0.034 (0.045, 0.022)	< 0.02 (< 0.02, < 0.02)	
Bridgeton NJ, 2003 (Rebel)	WG	0.10	0.04	243–262	3	8–9	2	Roots	0.099 (0.078, 0.12)	< 0.02 (< 0.02, < 0.02)	0.030 (0.030, 0.030)	< 0.02 (< 0.02, < 0.02)	
Willard OH, 2003 (Cabernet)	WG	0.10	0.02–0.03	402–430	3	6–8	4	Roots	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.022 (0.020, 0.024)	< 0.02 (< 0.02, < 0.02)	

[a] The last applications at each trial site were made 21 days apart, rendering the trials independent.

Celery

Six independent trials were conducted on celery in the US between 2001 and 2002 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 5–8 days. Celery was harvested 0 DALT. Celery stalks were cut at the soil level using hand clippers. Damaged leaves were removed when necessary.

A modified version of analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 198 days (ca. 7 months). Storage stability data on water content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 67.

Table 67 Residues of Flonicamid in Celery Following Foliar Spray with Flonicamid 50 WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.07–0.30	30–100	3	7	0					
Belle Glade FL, 2001 (Walts Pride)	WG	0.10	0.10–0.11	93–97	3	7	0	0.354 (0.391, 0.317)	< 0.01 (< 0.01, < 0.01)	0.013 (0.011, 0.015)	0.029 (0.019, 0.039)	P-3575
Laingsburg MI, 2002 (XP-266)	WG	0.10	0.10–0.11	94–103	3	7	0	0.450 (0.440, 0.459)	< 0.01 (< 0.01, < 0.01)	0.017 (0.015, 0.018)	0.037 (0.034, 0.040)	
Yuma AZ, 2001 (CUF 101)	WG	0.10	0.11	94	3	7–8	0	0.429 (0.459, 0.398)	< 0.01 (< 0.01, < 0.01)	< 0.01 (0.014, < 0.01)	0.026 (0.027, 0.024)	
Visalia CA, 2002 (Tall Utah 52-70)	WG	0.10	0.11	94	3	7	0	0.383 (0.435, 0.330)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.021 (0.025, 0.017)	
							1	0.931 (0.919, 0.942)	< 0.01 (< 0.01, < 0.01)	0.010 (< 0.01, 0.010)	0.032 (0.024, 0.039)	
							3	0.920 (0.956, 0.884)	< 0.01 (< 0.01, < 0.01)	0.010 (0.010, < 0.01)	0.034 (0.037, 0.030)	
							7	0.551 (0.578, 0.524)	< 0.01 (< 0.01, < 0.01)	0.011 (0.011, 0.010)	0.060 (0.057, 0.063)	
King City CA, 2002 (G-20)	WG	0.10	0.11	94–98	3	7	0	0.462 (0.457, 0.466)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.023 (0.025, 0.021)	
Camarillo CA, 2001 (Sonora)	WG	0.10–0.11	0.11	94–100	3	5–7	0	0.444 (0.423, 0.465)	< 0.01 (< 0.01, < 0.01)	0.010 (0.010,	0.029 (0.032,	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
										< 0.01)	0.026)	

Cereal grains

Wheat

The Meeting received information on fifteen independent trials on wheat in Northern and Southern EU between 2000 and 2001 with two foliar spray applications of a WG formulation and a re-treatment intervals of 16–28 days. Wheat grain was harvested 21–30 DALT.

The GC-MS analytical method A-22-00-02 was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte for grain.

The maximum period of sample storage at –20 °C was up to 433 days (ca. 15 months). Storage stability data on high starch content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 68.

Table 68 Residues of Flonicamid in Wheat grain Following Foliar Spray with a 50 WG Formulation of Flonicamid in European Regions

Location, year (variety)	Application						PHI, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Slovenia GAP	WG	0.07	Not specified		2	21	28					
Poggio Renatico, Ferrara, Italy, 2001 (Vayolet)	IBE 3894	0.07	0.018	407–417	2	22	28	< 0.01	< 0.01	0.07	0.7	
Emilia-Romagna, Italy, 2001 (Mieti)	IBE 3894	0.07	0.024	300	2	22	30	0.06 (0.10, < 0.01)	0.05 (0.09, < 0.01)	0.05 (0.09, < 0.01)	0.16 (0.29, 0.03)	
Italy, 2001 (Winter Wheat)	IBE 3894	0.07	0.022–0.023	300	2	22	28	< 0.01	< 0.01	0.05	0.43	
Minaya, Albacete, Spain, 2001 (Gazul) [a]	IBE-3894	0.07	0.02	357–363	2	21	27	< 0.01	< 0.01	0.02	0.09	
Minaya, Albacete, Spain, 2001 (Farak) [a]	IBE-3894	0.07	0.02	360–373	2	21	26	< 0.01	< 0.01	< 0.01	0.07	
Douzonville, North of France, 2001 (Soisson)	IBE-3894	0.07	0.035	205	2	21	21	< 0.01	< 0.01	< 0.01	0.13	
							28	< 0.01	< 0.01	< 0.01	0.12	
Thignonville, North of France, 2001 (Isengrains)	IBE-3894	0.07	0.035	198–200	2	21	28	< 0.01	< 0.01	0.01	0.14	
Rabastens, South of France, 2000 (Gascogne) [b]	IBE 3880	0.07	201	0.035	2	22	27	< 0.01	< 0.01	0.04	0.30	
Rabastens, South of France, 2000 (Gascogne) [b]	IBE 3894	0.07	203–208	0.035	2	22	27	< 0.01	< 0.01	0.06	0.53	
							28	< 0.01	< 0.01	0.03	0.55	
Rabastens, South of France, 2001 (Soisson) [b]	IBE-3894	0.07	0.035	208–211	2	16	28	0.02	< 0.01	0.03	0.16	

Location, year (variety)	Application						PHI, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Puycornet, South of France, 2001 (Soisson)	IBE-3894	0.07	0.035	207	2	19	21	< 0.01	< 0.01	< 0.01	0.07	
							28	< 0.01	< 0.01	< 0.01	0.02	
Stanton, Derbyshire, United Kingdom, 2001 (Consort)	IBE-3894	0.07	0.035	200	2	21	28	< 0.01	< 0.01	< 0.01	0.06	
Meckesheim,Germany, 2001 (Altos) [c] 070-i-d-01	IBE 3894	0.07	0.029	241–249	2	28	21	0.07	0.07	0.06	0.74	
							28	< 0.01	< 0.01	0.05	0.56	
Meckesheim, Germany, 2001 (Monopol) [c]	IBE - 3880	0.073–0.075	0.024	310–316	2	21	28	0.04	< 0.01	0.06	1.10	A-22-01-05
Meckesheim,Germany, 2001 (Bandit) [c]	IBE - 3880	0.066–0.074	278–314	0.024	2	22	28	0.02	< 0.01	0.06	0.28	
Audeville, North of France, 2000 (Tremie) [d]	IBE-3880	0.069	197–198	0.035	2	20	28	< 0.01	< 0.01	0.03	0.55	
Audeville, North of France, 2000 (Tremie) [d]	IBE-3894	0.07	200	0.035	2	20	21	0.01	< 0.01	0.06	0.78	
							28	< 0.01	< 0.01	0.01	0.46	
Puiselet-le-Marais, North of France, 2000 (Altria) [e]	IBE-3880	0.07	200–203	0.035	2	20	28	< 0.01	< 0.01	0.02	0.20	
Puiselet-le-Marais, North of France, 2000 (Altria) [e]	IBE-3894	0.07	204–208	0.035	2	20	28	< 0.01	< 0.01	0.05	0.49	
Meauzac, South of France, 2000 (Aztec) [f]	IBE-3880	0.07	200	0.035	2	20	28	< 0.01	< 0.01	0.07	0.46	
Meauzac, South of France, 2000 (Aztec) [f]	IBE-3894	0.07	198–200	0.035	2	20	21	< 0.01	< 0.01	0.06	0.51	
							28	< 0.01	< 0.01	0.06	0.36	
Hilgersmissen, Germany, 2000 (Brigadier) [g]	IBE 3880	0.07	196–206	0.035	2	22	28	< 0.01	< 0.01	0.01	0.15	A-22-01-10_VP00-1-9
Hilgersmissen,Germany, 2000 (Brigadier) [g]	IBE 3880	0.07	198–200	0.035	2	22	21	0.01	< 0.01	0.01	0.13	
							28	0.01	< 0.01	0.02	0.21	

Note: All trials identified with the same letter were considered dependent as they were conducted at the same location, the last applications were made on the same day at both sites and varieties were not determined to be sufficiently different

Barley

Eight independent trials were conducted on wheat in Germany and Denmark between 2011 and 2012 where a single foliar spray application of a WG formulation was made at 0.07 kg ai/ha. Barley grain was harvested 30–39 DALT.

The LC-MS/MS analytical method AGR/MOA/IKI220-1was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte for grain.

The maximum period of sample storage at –20 °C was up to 111 days (ca. 4 months). Storage stability data on high starch content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 69.

Table 69 Residues of Flonicamid in Barley Grain Following Foliar Spray with a 50 WG Formulation of Flonicamid in Denmark and Germany

<table>
<tr><th rowspan="2">Location, year (variety)</th><th colspan="5">Application</th><th rowspan="2">PHI, days</th><th colspan="3">Residues (mg/kg)</th><th rowspan="2">Ref</th></tr>
<tr><th>Form</th><th>kg ai/ha</th><th>kg ai/hL</th><th>Water, L/ha</th><th>no.</th><th>Flonicamid</th><th>TFNA</th><th>TFNG</th></tr>
<tr><td>Middelfart, Fyn, Denmark, 2011 (Tamtam)</td><td>IBE 3894</td><td>0.069</td><td>0.035</td><td>198</td><td>1</td><td>38</td><td>< 0.01</td><td>0.01</td><td>0.13</td><td rowspan="2">S11-01987</td></tr>
<tr><td>Harndrup, Fyn, Denmark, 2011 (Quench)</td><td>IBE 3894</td><td>0.07</td><td>0.035</td><td>200</td><td>1</td><td>30</td><td>< 0.01</td><td>< 0.01</td><td>0.04</td></tr>
<tr><td>Hygindvej, Ejby, Denmark, 2012 (Simba)</td><td>IBE 3894</td><td>0.067</td><td>0.035</td><td>192</td><td>1</td><td>33</td><td>0.02</td><td>< 0.01</td><td>0.07</td><td rowspan="8">S12-01930</td></tr>
<tr><td>Poppenhausen, Baden, Wurttemberg, Germany, 2012 (Grace)</td><td>IBE 3894</td><td>0.073</td><td>0.035</td><td>210</td><td>1</td><td>31</td><td>< 0.01</td><td>< 0.01</td><td>0.12</td></tr>
<tr><td rowspan="2">Billeshavevej, Middelfart, Denmark, 2012 (Tamtam)</td><td rowspan="2">IBE 3894</td><td>0.073</td><td>0.035</td><td>210</td><td rowspan="2">1</td><td>39</td><td>< 0.01</td><td>0.01</td><td>0.13</td></tr>
<tr><td>0.21</td><td>0.10</td><td>200</td><td>39</td><td>< 0.01</td><td>0.04</td><td>0.52</td></tr>
<tr><td>Tornhoj, Bogense, Denmark, 2012 (Quench)</td><td>IBE 3894</td><td>0.069</td><td>0.035</td><td>197</td><td>1</td><td>38</td><td>< 0.01</td><td>0.01</td><td>0.17</td></tr>
<tr><td>Wiesentheid, Bavaria, Germany, 2012 (Marthe)</td><td>IBE 3894</td><td>0.071</td><td>0.035</td><td>203</td><td>1</td><td>31</td><td>0.01</td><td>< 0.01</td><td>0.08</td></tr>
<tr><td>Main, Bavaria, Germany, 2012 (Quench)</td><td>IBE 3894</td><td>0.071</td><td>0.035</td><td>204</td><td>1</td><td>31</td><td>0.02</td><td>0.01</td><td>0.12</td></tr>
</table>

Tree Nuts

Almonds

Five independent trials were conducted on almonds in the US between 1996 and 2008 where three foliar spray applications of a SG formulation were made with re-treatment intervals of 6–8 days. Almonds were harvested 39–42 DALT.

A modified version of analytical method P-3822 was used to analyse all almond nutmeat samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 196 days (ca. 7 months). Storage stability data on oil content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 70.

Table 70 Residues of Flonicamid in Almond Nutmeats Following Foliar Spray with Beleaf 50 SG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	SG	0.07–	0.01–	100–	3	7	40					IB-

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
		0.10	0.10	500								2011-JLW-014-01-01
Chico, CA, 2008 (Non-pareil)	SG	0.10	0.01	1029–1038	3	7–8	40	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.013 (0.014, 0.011)	< 0.01 (< 0.01, < 0.01)	
Orland, CA, 2004 (Non pareil)	SG	0.1	0.01	1169	3	7	20	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.011 (0.011, 0.010)	< 0.01 (< 0.01, < 0.01)	
							30	0.01 (0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.013 (0.014, 0.012)	< 0.01 (< 0.01, < 0.01)	
							40	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.022 (0.024, 0.020)	< 0.01 (< 0.01, < 0.01)	
							50	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.014 (0.014, 0.014)	< 0.01 (< 0.01, < 0.01)	
Wasco, CA, 1996 (Fritz)	SG	0.10	0.007	1459–1543	3	6–8	39	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Coalinga, CA, 2006 (Non-pareil)	SG	0.10	0.006–0.007	1534–1702	3	7	39	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Turlock, CA, 2007 (Butte)	SG	0.10	0.006–0.007	1487–1721	3	7	42	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.036 (0.034, 0.037)	< 0.01 (< 0.01, < 0.01)	

Pecans

Five independent trials were conducted on pecans in the US between 1983 and 2008 where three foliar spray applications of a SG formulation were made with re-treatment intervals of 7–8 days. Pecans were harvested 20–40 DALT.

A modified version of analytical method P-3822 was used to analyse all almond nutmeat and hulls samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 196 days (ca. 7 months). Storage stability data on oil content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 71.

Table 71 Residues of Flonicamid in Pecans Following Foliar Spray with Beleaf 50 SG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg) (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	SG	0.07-0.10	0.01-0.10	100-500	3	7	40					IB-2011-JLW-014-01-
Anton, TX, 1995	SG	0.10	0.01	1010-1038	3	7	40	< 0.01 (< 0.0	< 0.01 (< 0.0	< 0.01 (< 0.0	< 0.01 (< 0.0	

Location, year (variety)	Application						DALT, days	Residues (mg/kg) (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
(Western Schley)								1, < 0.01)	1, < 0.01)	1, < 0.01)	1, < 0.01)	01
Pearsall, TX, 1983 (Cheyenne)	SG	0.10	0.007	1360-1375	3	7	39	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Opelousas, LA, 2000 (Native)	SG	0.10	0.009	1113-1141	3	7	39	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Bailey, NC, 1989 (Stuart)	SG	0.10	0.007-0.010	1048-1534	3	7	20	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
Girard, GA, 1998 (Desirables)	SG	0.10	0.009	1150-1160	3	7-8	39	< 0.01 (< 0.01, < 0.01)	0.011 (0.011, 0.011)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	

Pistachios

Two independent trials were conducted on pistachios in the US in 2014 where three foliar spray applications of a SG formulation were made with re-treatment intervals of 6–7 days. Pistachios were harvested 40 DALT.

Analytical method P-3822 was used to analyse all samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 24 days. As pistachio samples were analysed within 30 days of sampling, freezer storage stability information was not required. The results are summarized in Table 72.

Table 72 Residues of Flonicamid in Pistachios Following Foliar Spray with Beleaf 50 SG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg) (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	SG	0.07–0.10	0.01–0.10	100–500	3	7	40					IB-2014-JLW-015-01-01
Madera, CA, 2014 (Kerman)	SG	0.10	0.01	1219–1237	3	7	40	0.042 (0.042, 0.041)	< 0.01 (< 0.01, < 0.01)	0.064 (0.063, 0.065)	0.079 (0.080, 0.078)	
Terra Bella, CA, 2014 (Kerman)	SG	0.10	0.01	941–1393	3	6	40	0.018 (0.018, 0.017)	< 0.01 (< 0.01, < 0.01)	0.042 (0.043, 0.040)	0.069 (0.072, 0.066)	

Rape seed

Eight independent trials were conducted on canola in the US in 2007 where three foliar spray applications of a SG formulation were made with re-treatment intervals of 6–8 days. Canola seeds were harvested 6–8 DALT.

Analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.02 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 755 days (ca. 25 months). Concurrent storage stability data on canola seed showed that the residues of flonicamid and its associated metabolites are stable for 735 days (ca. 24 months). The results are summarized in Table 73.

Table 73 Residues of Flonicamid in Rape Seed Following Foliar Spray with Beleaf 50 SG Formulation in North American Regions

Location, year (variety)	Application							Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days	Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.10	0.1–0.3	30–100	3	6–8	7					
Kimberly, ID, 2007 (Sunrise Spring)	SG	0.10	0.07	140	3	6–7	6	0.083 (0.096, 0.070)	< 0.02 (< 0.02, < 0.02)	0.037 (0.039, 0.034)	0.084 (0.087, 0.081)	9783
Minot, ND, 2007 (5630)	SG	0.10	0.11	94	3	7–8	7	0.333 (0.339, 0.326)	0.033 (0.035, 0.031)	0.086 (0.087 0.086)	0.338 (0.385, 0.291)	
Velva, ND, 2007 (5550)	SG	0.10	0.07–0.08	131–140	3	7	7	0.021 (0.022, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.049 (0.052, 0.045)	0.032 (0.032, 0.031)	
Bridgeton, NJ, 2007 (Sunrise)	SG	0.10	0.08	122	3	7	7	0.024 (0.025, 0.022)	< 0.02 (< 0.02, < 0.02)	0.021 (0.021, 0.020)	0.030 (0.039, 0.020)	
Brookings, SD, 2007 (Crosby)	SG	0.10	0.06	178	3	7–8	7	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.029 (0.026, 0.031)	0.042 (0.035, 0.048)	
Aurora, SD, 2007 (Crosby RR)	SG	0.10	0.06	168	3	6–8	6–7	0.092 (0.135, 0.048)	< 0.02 (< 0.02, < 0.02)	0.063 (0.050, 0.077)	0.161 (0.158, 0.164)	
Brookings, SD, 2008 (Hyclass 601)	SG	0.10	0.10	103	3	6	6	0.169 (0.087, 0.251)	0.068 (0.052, 0.084)	0.066 (0.049, 0.082)	0.136 (0.029, 0.243)	
Prosser, WA, 2007 (Raper)	SG	0.10	0.05–0.07	131–187	3	6–7	8	0.022 (0.023, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
Prosser, WA,	SG	0.1	0.08	122	3	6–8	6	0.045	< 0.02	< 0.02	< 0.02	

Location, year (variety)	Application							Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	DALT, days	Flonicamid	TFNA-AM	TFNA	TFNG	
2008 (Raper)		0						(0.034, 0.056)	(< 0.02, < 0.02)	(< 0.02, < 0.02)	(< 0.02, < 0.02)	

Cotton

Twelve independent trials were conducted on cotton in the US in 2001 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–9 days. Seeds were collected 29–32 DALT, dried and cleaned followed by a stick extraction to remove the gin trash. The lint cotton was saw ginned to remove the majority of the lint from the cottonseed.

In Australia, ten independent trials were conducted on cotton in 2012 where one or two foliar spray applications were made at 0.10 kg ai/ha or 0.20 kg ai/ha at re-treatment intervals of 14–15 days. Cotton was picked from bolls 7–43 DALT and ginned to separate the fuzz (undelinted).

Method P-3567, a modified version of analytical method P-3561M was used to analyse all samples collected from the US trials while method AATM-R-165 was used to analyse all samples from the Australian trials. The LOQ was determined to be 0.02 mg/kg/analyte for P-3567. For method AATM-R-165, the LOQ was 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 470 days (ca. 16 months). Storage stability data on oil content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 74.

Table 74 Residues of Flonicamid in Undelinted Cottonseeds Following Foliar Spray with Flonicamid 50WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/ SG	0.05–0.10	0.02–0.05	30–50	3	7	30					
Elko, SC, 2001 (Delta Pine 451 B/RR)	WG	0.10	0.06	168	3	7	29	<u>0.040</u> (0.042, 0.038)	< 0.02 (< 0.02, < 0.02)	0.050 (0.054, 0.046)	0.024 (0.028, 0.020)	IB-2001-MDG-004-00-01
West Memphis, AR, 2001 (Suregrow)	WG	0.10	0.07	150	3	7	0	0.104 (0.105, 0.102)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							10	0.029 (0.031, 0.026)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							21	0.028 (0.028, 0.027)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							30	<u>0.042</u> (0.039, 0.045)	< 0.02 (< 0.02, < 0.02)	0.055 (0.051, 0.059)	0.024 (0.026, 0.021)	
							40	0.025 (0.026, 0.024)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							10	0.029 (0.025, 0.032)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							21	0.027 (0.028, 0.026)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
							30	0.036 (0.035, 0.036)	< 0.02 (< 0.02, < 0.02)	0.066 (0.063, 0.069)	0.026 (0.025, 0.027)	
							40	0.026 (0.022, 0.029)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
Tillar, AR, 2001 (Pay-master)	WG	0.10	0.10	94–103	3	6–9	30	<u>0.031</u> (0.029, 0.033)	< 0.02 (< 0.02, < 0.02)	0.048 (0.048, 0.048)	0.021 (< 0.02, 0.021)	
Senatobia, MS, 2001 (DPL 451 Bt/RR)	WG	0.10	0.05	187–196	3	7	29	<u>0.034</u> (0.032, 0.035)	< 0.02 (< 0.02, < 0.02)	0.049 (0.049, 0.048)	0.023 (0.023, 0.023)	
Eakly, OK, 2001 (PM 2280)	WG	0.10	0.05	187	3	6–8	30	<u>0.035</u> (0.035, 0.035)	< 0.02 (< 0.02, < 0.02)	0.057 (0.057, 0.056)	0.026 (0.025, 0.027)	
Dill City, OK, 2001 (Pay-master 2326)	WG	0.10	0.05–0.06	168–206	3	7	31	<u>0.048</u> (0.036, 0.059)	< 0.02 (< 0.02, < 0.02)	0.110 (0.113, 0.107)	0.094 (0.080, 0.107)	
Levelland, TX, 2001 (PM 2326 B6/RR)	WG	0.10	0.07	140	3	6–8	29	<u>0.055</u> (0.050, 0.060)	< 0.02 (< 0.02, < 0.02)	0.117 (0.101, 0.133)	0.105 (0.094, 0.116)	
Uvalde, TX, 2001 (PM 2326 RR)	WG90	0.10	0.05	187–196	3	7	30	<u>0.046</u> (0.057, 0.034)	< 0.02 (< 0.02, < 0.02)	0.124 (0.105, 0.143)	0.094 (0.079, 0.109)	
Edmonson, TX, 2001 (Pay-master HS 250)	WG	0.10	0.06–0.07	150–187	3	7–8	0	0.120 (0.143, 0.097)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							11	0.028 (0.028, 0.028)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							20	0.028 (0.026, 0.030)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							32	<u>0.043</u> (0.050, 0.035)	< 0.02 (< 0.02, < 0.02)	0.120 (0.122, 0.118)	0.070 (0.071, 0.068)	
							43	0.025 (0.024, 0.026)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							11	0.033 (0.029, 0.037)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							20	0.038 (0.035, 0.040)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							32	0.035 (0.041, 0.028)	< 0.02 (< 0.02, < 0.02)	0.261 (0.305, 0.217)	0.149 (0.179, 0.118)	
							43	0.030 (0.032, 0.028)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
Stanfield, AZ, 2001 (DP458 B1RR)	WG	0.10	0.06	187	3	7	29	<u>0.041</u> (0.041, 0.040)	< 0.02 (< 0.02, < 0.02)	0.125 (0.127, 0.123)	0.073 (0.074, 0.071)	
Mariopa, AZ, 2001 (DP451 B1RR)	WG	0.10	0.05	187	3	7	30	<u>0.085</u> (0.083, 0.087)	< 0.02 (< 0.02, < 0.02)	0.133 (0.146, 0.119)	0.084 (0.087, 0.080)	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
Madera, CA, 2001 (Acala Riata RR)	WG	0.10–0.11	0.04	281–290	3	7	29	0.085 (0.084, 0.086)	< 0.02 (< 0.02, < 0.02)	0.121 (0.133, 0.109)	0.108 (0.126, 0.089)	
AUS GAP	WG	0.07	NS	NS	2	NS	7					
Mywybilla, Queensland, (Sicot 71BRF)	WG	0.10	0.120	84	2	14	7	0.035	< 0.01	< 0.01	< 0.01	UPL GLP-10-07
		0.10	0.127	81			27	0.012	< 0.01	0.041	0.01	
		0.20	0.248	82			7	0.064	< 0.01	0.014	0.013	
		0.20	0.253	80			27	0.016	< 0.01	0.07	0.019	
		0.10	0.125	79		15	7	0.01 (< 0.01, 0.01)	0.01 (< 0.01, 0.01)	0.013 (0.015, 0.01)	0.01 (< 0.01, 0.01)	
		0.10	0.118	82		14	15	< 0.01	< 0.01	0.018	< 0.01	
		0.10	0.129	77		14	22	< 0.01	< 0.01	0.048	0.016	
		0.10	0.123	79		14	29	< 0.01	< 0.01	0.068	< 0.01	
		0.10	0.114	88		14	36	< 0.01	< 0.01	0.13	0.022	
		0.10	0.106	93		14	43	< 0.01	< 0.01	0.17	0.021	
		0.20	0.255	78		15	7	0.014	< 0.01	< 0.01	< 0.01	
		0.20	0.260	78		14	29	< 0.01	< 0.01	0.08	0.017	
Boggabilla, New South Wales (Sicot 71BRF)	WG	0.10	0.11	92	2	14	7	0.13	< 0.01	0.01	0.015	
		0.10	0.11	92		15	28	0.018	< 0.01	0.038	0.033	
		0.20	0.218	92		14	7	0.31	0.018	0.021	0.046	
		0.20	0.22	92		15	28	0.045	0.021	0.076	0.091	
Narrabi, New South Wales (Sicot 71BRF)	WG	0.10	0.112	89	2	14	7	0.094	< 0.01	< 0.01	< 0.01	
		0.10	0.11	92		15	13	0.074	< 0.01	< 0.01	< 0.01	
		0.10	0.109	92		14	21	0.016	< 0.01	0.05	< 0.01	
		0.10	0.111	91		13	28	0.024	< 0.01	0.12	0.023	
		0.10	0.114	88		14	35	< 0.01	< 0.01	0.1	0.014	
		0.10	0.105	97		14	41	0.012 (0.012, 0.011)	< 0.01 (< 0.01, < 0.01)	0.43 (0.47, 0.38)	0.069 (0.066, 0.071)	
		0.20	0.228	89		14	7	0.18	< 0.01	< 0.01	< 0.01	
		0.20	0.224	90		13	28	0.041	0.012	0.14	0.04	
Chinchilla, Queensland (Sicot 71BRF)	WG	0.10	0.10	105	2	14	7	0.022	< 0.01	< 0.01	< 0.01	UPL GLP 12 01-1
		0.10	0.10	110		14	28	0.011	< 0.01	< 0.01	0.011	
		0.10	0.10	101		14	49	< 0.01	< 0.01	0.01	0.015	
		0.10	0.10	102		14	63	< 0.01	< 0.01	0.063	0.04	
		0.20	0.19	106		14	7	0.085	< 0.01	< 0.01	< 0.01	
		0.20	0.18	110		14	28	0.025	< 0.01	< 0.01	0.018	
		0.20	0.20	101		14	49	< 0.01	< 0.01	0.021	0.026	
		0.20	0.20	102		14	63	< 0.01	< 0.01	0.09	0.077	
Condamine Plains, Queensland (Sicot71BRF)		0.10	0.09	110	2	14	7	< 0.01	< 0.01	< 0.01	< 0.01	
		0.10	0.10	100		15	20	< 0.01	< 0.01	0.037	< 0.01	
		0.10	0.10	101		14	27	0.012	< 0.01	0.056	0.014	
		0.10	0.10	103	1	NA	27	< 0.01	< 0.01	0.016	< 0.01	
		0.05	0.05	103	2	14	35	< 0.01	< 0.01	0.026	< 0.01	
	WG	0.10	0.10	101		14	41	< 0.01	< 0.01	0.096	0.027	
		0.10	0.09	106		13	49	< 0.01	< 0.01	0.11	0.014	
		0.10	0.09	109		14	55	< 0.01	< 0.01	0.19	0.06	
		0.20	0.18	111		13	7	< 0.01	< 0.01	< 0.01	< 0.01	
		0.20	0.20	102		14	27	0.02	< 0.01	0.056	0.022	
		0.20	0.20	101		14	41	0.014	< 0.01	0.16	0.053	
		0.20	0.18	110		14	55	< 0.01	< 0.01	0.34	0.099	
Narrabri, New South Wales (Sicot 71BRF)	WG	0.10	0.11	90–92	2	14	8	0.34	0.071	0.025	0.048	UPL GLP 12 01-1
		0.10	0.11	89–90	2	14	15	0.067	0.033	0.019	0.054	
		0.10	0.11	91–92	2	14	22	0.11	0.047	0.043	0.12	
		0.10	0.11	89–90	2	14	29	0.13	0.069	0.051	0.16	
		0.10	0.11	91	2	14	36	0.088	0.076	0.075	0.23	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
		0.10	0.11	88–91	2	14	43	0.032	0.029	0.064	0.12	
		0.10	0.11	90	2	14	50	0.025	0.028	0.098	0.16	
		0.10	0.11–0.12	84–86	2	14	57	< 0.01	< 0.01	0.1	0.12	
		0.23	0.22	92–93	2	14	8	0.48	0.072	0.026	0.049	
		0.21	0.22	92	2	14	29	0.11	0.082	0.053	0.17	
		0.20	0.22–0.24	85–92	2	14	43	0.096	0.097	0.13	0.33	
		0.20	0.24	85–86	2	14	57	0.022	0.025	0.12	0.19	
Narromine, New South Wales (Sicot 71BRF)	WG	0.10	0.08–0.09	114–125	2	14	7	0.16 (0.17, 0.14)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
		0.10	0.08–0.09	116–119	2	14	28	0.046	< 0.01	0.14	0.036	
		0.10	0.08	123–124	2	11	42	< 0.01	< 0.01	0.091	0.017	
		0.10	0.08–0.09	117–123	2	15	53	< 0.01	< 0.01	0.11	0.026	
		0.20	0.17–0.18	114–119	2	14	7	0.09 (0.088, 0.092)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	
		0.21	0.17	122–124	2	14	28	0.056	0.1	0.13	0.034	
		0.21	0.17	119–123	2	11	42	0.012	< 0.01	0.17	0.037	
		0.21	0.17–0.18	118–123	2	15	53	< 0.01	< 0.01	0.18	0.045	

Mint

Three independent trials were conducted on fresh mint in the US in 2011 where three foliar spray applications of a SG formulation were made with re-treatment intervals of 13–15 days. Mint leaves were harvested 7 DALT.

Analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.02 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 372 days (ca. 12 months). Concurrent storage stability data on mint tops show that the residues are stable for at least 364 days (ca. 12 months). The results are summarized in Table 75.

Table 75 Residues of Flonicamid in Fresh Mint Tops Following Foliar Spray with Beleaf 50 SG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.07–0.10	0.04–0.05	100–200	3	14	7					
Bruneau, ID, 2011 (Black Mitcham Perppermint)	SG	0.10	0.04	271–281	3	14	7	2.36 (2.31, 2.41)	0.377 (0.376, 0.377)	0.105 (0.086, 0.125)	0.104 (0.146, 0.133)	9358
Prosser, WA, 2011 (Peppermint)	SG	0.10	0.04	243–262	3	13–14	7	1.70 (1.67, 1.73)	0.456 (0.451, 0.461)	0.234 (0.214, 0.254)	0.229 (0.222, 0.235)	
Endeavour,	SG	0.10	0.02	552–	3	13–	6	1.92	0.036	0.166	0.208	

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
WI, 2011 (Scotch Spearmint)				561		15		(1.90, 1.93)	(0.337, 0.356)	(0.167, 0.164)	(0.211, 0.204)	

Dried hops

Four independent trials were conducted on hops in the US in 2003 and 2015 where three foliar spray applications of a WG or SG formulation were made with re-treatment intervals of 7–8 days. Green hop cones were sampled 9–11 DALT and dried to 8–10% moisture in a forced hot air dryer. Drying temperature was about 120–140 °F (49–64 °C).

The analytical methods P-3561M (2003 trial) or P-3822 (2015 trial) were used to analyse all samples. The LOQ for dried hop cones was determined to be 0.02 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 329 days (ca 11 months). Concurrent storage stability data on hops show that the residues are stable for at least 299 days (10 months). The results are summarized in Table 76.

Table 76 Residues of Flonicamid in Dried Hop Cones Following Foliar Spray with Flonicamid 50 WG and Beleaf 50SG Formulations in Northern America

Location, year (variety)	Application						DALT, days	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/SG	0.06–0.10	0.01–0.02	500	3	7	10					
Parma ID, 2003 (Nugget)	WG	0.10	0.01	907–917	3	7–8	9	2.82 (2.85, 2.78)	0.717 (0.177, 0.165)	0.307 (0.312, 0.302)	0.104 (0.110, 0.098)	08706
Hubbard OR, 2003 (Nugget)	WG	0.10	0.01	795–945	3	7	9	1.15 (1.10, 1.20)	0.146 (0.139, 0.153)	0.456 (0.470, 0.442)	0.204 (0.204, 0.204)	
Prosser WA, 2003 (Nugget)	WG	0.10	0.01	1272–1347	3	7–8	11	0.563 (0.561, 0.565)	0.038 (0.038, 0.038)	0.335 (0.335, 0.334)	0.162 (0.156, 0.168)	
Ephrata WA, 2015 Cascade)	SG	0.10	0.01	945	3	7	10	9.33 (10.6, 8.06)	0.226 (0.269, 0.184)	0.727 (0.660, 0.794)	0.074 (0.076, 0.072)	IB-2014-JLW-014-01-01

Tea

Two independent trials were conducted on tea in Japan in 2001 where a single foliar spray application of a WG formulation was made. Leaves were harvested 7 DALT. On the day of harvest, leaves were processed according to standard procedure (steaming, cooling, primary drying and rolling, rolling, secondary drying and rolling, final drying and rolling and drying) prior to analysis

The analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.02 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 9 months. Storage stability data on various commodities show that the residues are stable for at least 23 months. The results are summarized in Table 77.

Table 77 Residues of Flonicamid in Tea Following Foliar Spray with Flonicamid 50 WG Formulation in Japan

Location, year (variety)	Application						DALT, days	Residues (mg/kg)			Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days		Flonicamid	TFNA	TFNG	
Japan GAP	DF	0.1	0.01	2000–4000	1	NA	7				
Tsukui, Kanagawa, Japan, 2001 (Yabukita)	DF	0.2	0.01	2000	1	NA	7	20.1 (22.7, 21.8, 18.0, 17.8)	0.31 (0.31, 0.26, 0.35, 0.32)	3.03 (3.32, 3.10, 2.85, 2.82)	Report No 13-79
Uji, Kyoto, Japan, 2001 (Yakibuta)	DF	0.2	0.01	2000	1	NA	7	15.7 (16.9, 16.5, 15.0, 14.5)	0.18 (0.18, 0.16, 0.20, 0.18)	1.82 (1.98, 1.97, 1.67, 1.64)	

Animal feeds

Wheat forage and straw

Fifteen independent supervised trials were conducted in EU on wheat in 2000 and 2001, where two foliar spray applications of a WG formulation were made with re-treatment intervals of 16–21 days. Green forage (green plant, rest of plant) was sampled 0–7 DALT, stems and ears 14–21 DALT and straw 21–30 DALT.

The analytical methods A-22-02 was used to analyse all samples. The LOQ for green forage and straw was 0.02 mg/kg/analyte and 0.01 mg/kg/analyte for stems and ears.

The maximum period of sample storage at –20 °C was up to days (ca. 14 months). Storage stability data on starch content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 78.

Table 78 Residues of Flonicamid in Wheat Forage (green plant, rest of plant), Straw, Ears and Stems Following Foliar Spray with 50 WG Formulations in Northern and Southern EU

Location, year (variety)	Application						PHI, days	Commodity	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
Slovenia GAP	WG	0.07			2	21	28						
Poggio Renatico, Ferrara, Italy, 2001 (Vayolet)	IBE 3894	0.07	0.018	407–417	2	22	28	Straw	< 0.02	< 0.02	< 0.02	0.17	
Emilia-Romagna, Italy, 2001 (Mieti)	IBE 3894	0.07	0.024	300	2	22	30	Straw	0.08	< 0.02	0.02	0.41	
Italy, 2001 (Winter Wheat)	IBE 3894	0.07	0.022–0.023	300	2	22	28	Straw	0.04	< 0.02	0.03	0.36	

Location, year (variety)	Application						PHI, days	Commodity	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
Minaya, Albacete, Spain, 2001 (Gazul)[1]	IBE-3894	0.07	0.02	357–363	2	21	27	Straw	0.11	< 0.02	< 0.02	0.15	
Minaya, Albacete, Spain, 2001 (Farak)[1]	IBE-3894	0.07	0.02	360–373	2	21	26	Straw	0.03	< 0.02	< 0.02	0.14	
Douzonville, North of France, 2001 (Soisson)	IBE-3894	0.07	0.035	205	2	21	0	Green Plant	0.66	< 0.02	< 0.02	0.14	
							7	Green Plant	0.99	< 0.02	< 0.02	0.21	
							14	Ears	0.03	< 0.02	< 0.02	0.03	
							14	Stem	0.06	< 0.02	< 0.02	0.04	
							21	Straw	< 0.02	< 0.02	< 0.02	< 0.02	
							28	Straw	< 0.02	< 0.02	< 0.02	< 0.02	
Thignonville, North of France, 2001 (Isengrains)	IBE-3894	0.07	0.035	198–200	2	21	28	Straw	< 0.02	< 0.02	< 0.02		
Rabastens, South of France, 2000 (Gascogne)[2]	IBE 3880	0.07	201	0.035	2	22	27	Straw	0.03	< 0.02	< 0.02	0.06	
Rabastens, South of France, 2000 (Gascogne)[2]	IBE 3894	0.07	203–208	0.035	2	22	27	Straw	0.02	< 0.02	< 0.02	0.06	
							28	Straw	0.05	< 0.02	< 0.02	0.10	
Rabastens, South of France, 2001 (Soisson)	IBE-3894	0.07	0.035	208–211	2	16	28	Straw	0.02	< 0.02	< 0.02	< 0.02	
Puycornet, South of France, 2001 (Soisson)	IBE-3894	0.07	0.035	207	2	19	0	Green Plant	0.48	< 0.02	< 0.02	0.19	
							7	Green Plant	0.99	< 0.02	< 0.02	0.23	
							14	Ears	0.04	< 0.02	< 0.02	< 0.02	
							14	Stem	0.06	< 0.02	< 0.02	0.05	
							21	Straw	0.07	< 0.02	< 0.02	0.04	
							28	Straw	< 0.02	< 0.02	< 0.02	< 0.02	
Stanton, Derbyshire, United Kingdom, 2001 (Consort)	IBE-3894	0.07	0.035	200	2	21	28	Straw	0.05	< 0.02	< 0.02	0.08	
Meckesheim, Germany, 2001 (Altos)[3]	IBE 3894	0.07	0.029	241–249	2	28	0	Green Plant	0.32	< 0.02	< 0.02	0.11	
							7	Green Plant	0.15	< 0.02	0.02	0.10	
							14	Ears	< 0.02	< 0.02	0.04	0.16	
							14	Stem	< 0.02	< 0.02	< 0.02	0.16	
							21	Straw	< 0.02	< 0.02	< 0.02	0.21	
							28	Straw	< 0.02	< 0.02	< 0.02	0.15	
Meckesheim, Germany, 2001 (Monopol)[3]	IBE - 3880	0.073–0.075	0.024	310–316	2	21	28	Straw	0.23	< 0.02	0.03	0.17	A-22-01-05
Meckesheim, Germany, 2001 (Bandit)[3]	IBE - 3880	0.066–0.074	278–314	0.024	2	22	0	Rest of Plant	0.88	< 0.02	< 0.02	0.16	
							7	Rest of Plant	0.47	< 0.02	< 0.02	0.13	

Location, year (variety)	Application: Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days	PHI, days	Commodity	Residues (mg/kg): Flonicamid	TFNA-AM	TFNA	TFNG	Ref
							14	Ears	0.21	< 0.02	< 0.02	0.04	
							14	Stem	0.51	< 0.02	< 0.02	0.04	
							28	Straw	0.39	< 0.02	< 0.02	0.07	
Audeville, North of France, 2000 (Tremie)[4]	IBE-3880	0.069	197–198	0.035	2	20	0	Green Plant	0.53	< 0.02	< 0.02	0.07	
							28	Straw	0.05	< 0.02	< 0.02	0.10	
Audeville, North of France, 2000 (Tremie)[4]	IBE-3894	0.07	200	0.035	2	20	0	Green Plant	0.64	< 0.02	< 0.02	0.12	
							7	Green Plant	0.20	< 0.02	0.02	0.37	
							14	Ears	0.13	< 0.02	0.04	0.53	
							14	Stem	0.33	< 0.02	< 0.02	0.65	
							21	Straw	0.19	< 0.02	< 0.02	0.33	
							28	Straw	< 0.02	< 0.02	< 0.02	< 0.02	
Puiselet-le-Marais, North of France, 2000 (Altria)[5]	IBE-3880	0.07	200–203	0.035	2	20	28	Straw	0.04	< 0.02	< 0.02	0.05	
Puiselet-le-Marais, North of France, 2000 (Altria)[5]	IBE-3894	0.07	204–208	0.035	2	20	28	Straw	0.03	< 0.02	< 0.02	0.02	
Meauzac, South of France, 2000 (Aztec)[6]	IBE-3880	0.07	200	0.035	2	20	0	Green Plant	0.55	< 0.02	< 0.02	0.15	
							28	Straw	< 0.02	< 0.02	< 0.02	0.10	
Meauzac, South of France, 2000 (Aztec)[6]	IBE-3894	0.07	198–200	0.035	2	20	0	Green Plant	0.69	< 0.02	< 0.02	0.14	
							7	Green Plant	0.02	< 0.02	< 0.02	0.13	
							14	Ears	0.04	< 0.02	0.02	0.18	
							14	Stem	0.03	< 0.02	< 0.02	0.29	
							21	Straw	< 0.02	< 0.02	< 0.02	0.22	
							28	Straw	< 0.02	< 0.02	< 0.02	0.12	
Hilgersmissen ,Germany, 2000 (Brigadier)[7]	IBE 3880	0.07	196–206	0.035	2	22	0	Rest of Plant	0.83	< 0.02	< 0.02	0.08	A-22-01-10_VP00-1-9
							28	Straw	0.09	< 0.02	< 0.02	0.02	
Hilgersmissen ,Germany, 2000 (Brigadier)[7]	IBE 3880	0.07	198–200	0.035	2	22	0	Rest of Plant	0.67	< 0.02	< 0.02	0.06	
							7	Rest of Plant	0.65	< 0.02	< 0.02	0.06	
							14	Ears	0.79	< 0.02	< 0.02	0.08	
							14	Stem	1.58	0.03	0.05	0.30	
							21	Straw	0.11	< 0.02	< 0.02	0.03	
							28	Straw	0.07	< 0.02	< 0.02	0.02	

Barley ears

Four independent supervised trials were conducted in Germany and Denmark on barley ears in 2012, where a single foliar spray application of a WG formulation was made at 0.07 kg ai/ha and where ears were sampled 0–22 DALT.

The LC-MS/MS analytical method AGR/MOA/IKI220-1was used to analyse all samples. The LOQ for green forage and straw was 0.01 mg/kg/analyte for ears.

The maximum period of sample storage at –20 °C was up to 111 days (ca. 4 months). Storage stability data on high starch content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 79.

Table 79 Residues of Flonicamid in Barley Ears Following Foliar Spray with 50 WG Formulations in Denmark and Germany.

Location, year (variety)	Application					PHI, days	Residues (mg/kg)			
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.		Flonicamid	TFNA [a]	TFNG [a]	
Billeshavevej, Middelfart, Denmark, 2012 (Tamtam)	IBE 3894	0.073	0.035	210	1	0	1.18	< 0.01	0.02	S12-01930
						7	0.02	< 0.01	0.17	
						14	< 0.01	< 0.01	0.09	
						21	< 0.01	< 0.01	0.05	
Tornhoj, Bogense, Denmark, 2012 (Quench)	IBE 3894	0.069	0.035	197	1	0	0.49	< 0.01	0.02	
						6	0.06	0.01	0.18	
						14	0.01	0.01	0.15	
						21	< 0.01	< 0.01	0.09	
Wiesentheid, Bavaria, Germany, 2012 (Marthe)	IBE 3894	0.071	0.035	203	1	0	0.96	< 0.01	< 0.01	
						7	0.12	0.01	0.15	
						13	0.06	< 0.01	0.11	
						20	0.07	0.01	0.12	
Main, Bavaria, Germany, 2012 (Quench)	IBE 3894	0.071	0.035	204	1	0	1.0	< 0.01	0.02	
						8	0.14	0.01	0.13	
						15	0.05	< 0.01	0.11	
						22	0.04	0.01	0.12	

[a] Reported in flonicamid equivalents.

Alfalfa forage, seed and hay

Four independent trials were conducted on alfalfa in the US in 2009 where two foliar spray applications of a SG formulation were made with re-treatment intervals of 7–8 days. Because alfalfa is harvested differently in California compared to the Pacific Northwest, sample collection times varied between these two regions. In the Idaho and Washington trials, seed samples were collected 13–14 DALT in the summer, and forage and hay samples were harvested the following year, 265–293 DALT.

Analytical method P-3561M was used to analyse all samples. The LOQ was determined to be 0.02 mg/kg/analyte.

The maximum period of sample storage at –20 °C was 432 days (ca. 14 months) for alfalfa seed, 490 days (ca. 16 months) for forage and 496 days (ca. 16 months) for hay. Concurrent storage stability data show that the residues of flonicamid and its metabolites are stable for 490 days in forage, 518 days in hay and 462 days in seed. The results are summarized in Table 80.

Table 80 Residues of Flonicamid in Alfalfa Following Foliar Spray with Beleaf 50 SG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Matrix	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
GAP (West of the US Rockies)	WG/SG	0.10	0.10–0.50	100–200	2	7	14 14 62	Seed Forage Hay					
Holtville, CA, 2009 (CUF 101)	SG	0.10	0.06–0.07	299–318	2	7	5	Forage	5.97 (5.76, 6.18)	0.074 (0.062, 0.085)	0.491 (0.479, 0.503)	2.012 (2.029, 1.996)	9943
					2	7	10		2.99 (2.57, 3.41)	0.046 (0.035, 0.057)	0.368 (0.371, 0.365)	1.725 (1.581, 1.868)	
					2	7	11		0.319 (0.303, 0.335)	< 0.02 (< 0.02, < 0.02)	0.077 (0.071, 0.083)	0.434 (0.417, 0.450)	
					2	7	19		0.256 (0.265,	< 0.02 (< 0.02,	0.032 (0.039,	0.247 (0.286,	

Location, year (variety)	Application						DALT, days	Matrix	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
									0.247)	< 0.02)	0.026)	0.208)	
					2	7	24		0.323 (0.321, 0.324)	< 0.02 (< 0.02, < 0.02)	0.108 (0.130, 0.085)	0.240 (0.258, 0.221)	
					2	7	62		< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.02 (< 0.02, 0.020)	
					2	7	62	Hay	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.021 (0.022, 0.020)	
					2	7	11	Seed	0.141 (0.131, 0.151)	0.03 (< 0.02, 0.030)	0.155 (0.116, 0.194)	0.011 (0.084, 0.127)	
Holtville, CA, 2009 (CUF 101)	SG	0.10	0.09	234	2	7	14	Forage	1.30 (0.981, 1.620)	0.077 (0.058, 0.096)	0.247 (0.197, 0.297)	1.495 (1.183, 1.806)	
					2	7	65		< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.035 (0.039, 0.031)	
					2	7	65	Hay	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.062 (0.062, 0.061)	
					2	7	14	Seed	0.106 (0.103, 0.108)	< 0.02 (< 0.02, < 0.02)	0.050 (0.040, 0.060)	0.031 (0.020, 0.041)	
Jerome, ID, 2009 (Rampage)	SG	0.10	0.07	281	2	7	293	Forage	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
					2	7	293	Hay	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
					2	7	13	Seed	0.138 (0.134, 0.142)	< 0.02 (< 0.02, < 0.02)	0.355 (0.357, 0.373)	0.045 (0.038, 0.051)	
Touchet, WA, 2009 (Forage Genetics 43M120)	SG	0.10	0.07	271–281	2	7	265	Forage	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	0.022 (< 0.02, 0.023)	< 0.02 (< 0.02, < 0.02)	
					2	7	282	Hay	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
					2	7	14	Seed	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	

Almond Hulls

Five independent trials were conducted on almonds in the US between 1996 and 2008 where three foliar spray applications of a SG formulation were made with re-treatment intervals of 6–8 days. Almonds were harvested 39–42 DALT.

A modified version of analytical method P-3822 was used to analyse all almond nutmeat samples. The LOQ was determined to be 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 196 days (ca. 7 months). Storage stability data on oil content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 81.

Table 81 Residues of Flonicamid in Almond Hulls Following Foliar Spray with Beleaf 50 SG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Commodity[1]	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	SG	0.07–0.10	0.01–0.10	100–500	3	7	40						
Chico, CA, 2008 (Non-pareil)	SG	0.10	0.01	1029–1038	3	7–8	40	Hulls (9.1%)	4.302 (4.411, 4.193)	0.053 (0.053, 0.053)	0.187 (0.199, 0.174)	0.257 (0.248, 0.265)	IB-2011-JLW-014-01-01
							40	Hulls (dry weight)	4.731 (4.874, 4.588)	0.058 (0.058, 0.058)	0.205 (0.219, 0.191)	0.282 (0.273, 0.290)	
Orland, CA, 2004 (Non-pareil)	SG	0.10	0.01	1169	3	7	20	Hulls (11.1–75.2%)	4.107 (4.047, 4.167)	0.051 (0.052, 0.050)	0.133 (0.139, 0.126)	0.406 (0.402, 0.410)	
							30		0.734 (0.851, 0.616)	< 0.01 (< 0.01, < 0.01)	0.062 (0.067, 0.056)	0.143 (0.165, 0.120)	
							40		0.906 (0.916, 0.896)	0.01 (0.01, 0.01)	0.071 (0.063, 0.078)	0.140 (0.137, 0.143)	
							50		0.686 (0.613, 0.758)	0.01 (< 0.01, 0.013)	0.069 (0.070, 0.068)	0.120 (0.109, 0.130)	
							20	Hulls (dry weight)	16.58 (15.995, 17.147)	0.206 (0.207, 0.205)	0.535 (0.549, 0.520)	1.639 (1.589, 1.688)	
							30		2.032 (2.618, 1.896)	0.022 (0.025, 0.018)	0.190 (0.206, 0.173)	0.439 (0.508, 0.370)	
							40		1.089 (1.094, 1.083)	0.012 (0.012, 0.012)	0.085 (0.075, 0.094)	0.169 (0.164, 0.173)	
							50		0.771 (0.690, 0.851)	0.013 (0.010, 0.015)	0.078 (0.079, 0.076)	0.134 (0.122, 0.146)	
Wasco, CA, 1996 (Fritz)	SG	0.10	0.007	1459–1543	3	6–8	39	Hulls (20.2%)	1.442 (1.807, 1.076)	0.033 (0.034, 0.031)	0.032 (0.034, 0.029)	0.120 (0.133, 0.107)	
							39	Hulls (dry weight)	1.813 (2.297, 1.329)	0.041 (0.043, 0.039)	0.040 (0.044, 0.036)	0.151 (0.169, 0.133)	
Coalinga, CA, 2006 (Non-pareil)	SG	0.10	0.006–0.007	1534–1702	3	7	39	Hulls (24.1%)	0.700 (0.734, 0.665)	0.014 (0.015, 0.013)	0.079 (0.080, 0.078)	0.113 (0.123, 0.102)	
							39	Hulls (dry weight)	0.922 (0.981, 0.863)	0.019 (0.020, 0.017)	0.105 (0.107, 0.102)	0.148 (0.164, 0.132)	
Turlock, CA, 2007 (Butte)	SG	0.10	0.006–0.007	1487–1721	3	7	42	Hulls (60.0%)	1.095 (1.159, 1.030)	0.091 (0.093, 0.089)	0.305 (0.302, 0.308)	0.166 (0.167, 0.164)	
							42	Hulls (dry weight)	2.750 (2.912, 2.587)	0.229 (0.233, 0.225)	0.767 (0.759, 0.774)	0.415 (0.419, 0.411)	

Cotton seed by-products

Twelve independent trials were conducted on cotton in the US in 2001 where three foliar spray applications of a WG formulation were made with re-treatment intervals of 6–9 days. Seeds were collected 29–32 DALT, dried and cleaned followed by a stick extraction to remove the gin trash. The lint cotton was saw ginned to remove the majority of the lint from the cottonseed while the ginned seed was saw delinted to remove most of the remaining linters.

In Australia, ten independent trials were conducted on cotton in 2012 where one or two foliar spray applications were made at 0.10 kg ai/ha or 0.20 kg ai/ha at re-treatment intervals of 14–15 days. Cotton was picked from bolls 7–43 DALT and ginned to separate the fuzzy seed and lint. The simulated gin trash consisted of ground parts of the cotton plant including bracts, stems, leaves, immature or mummified bolls, flowers and raw cotton.

Method P-3567, a modified version of analytical method P-3561M was used to analyse all samples collected from the US trials while method AATM-R-165 was used to analyse all samples from the Australian trials. The LOQ was determined to be 0.02 mg/kg/analyte for P-3567. For method AATM-R-165, the LOQ was 0.01 mg/kg/analyte.

The maximum period of sample storage at –20 °C was up to 470 days (ca. 16 months). Storage stability data on oil content commodities show that the residues are stable for at least 23 months. The results are summarized in Table 82.

Table 82 Residues of Flonicamid in Delinted Seeds and Gin Trash Following Foliar Spray with Flonicamid 50WG Formulation in North American Regions

Location, year (variety)	Application						DALT, days	Commodity	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
US GAP	WG/ SG	0.05–0.10	0.02–0.05	30–50	3	7	30						
Elko, SC, 2001 (Delta Pine 451 B/RR)	WG	0.10	0.06	168	3	7	29	Delinted cottonseed	0.050 (0.054, 0.046)	< 0.02 (< 0.02, < 0.02)	0.063 (0.064, 0.062)	0.030 (0.032, 0.028)	
West Memphis, AR, 2001 (Suregrow)	WG	0.10	0.07	150	3	7	0	Delinted cottonseed	0.077 (0.063, 0.090)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							10		0.029 (0.025, 0.032)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							21		0.027 (0.028, 0.026)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							30		0.036 (0.035, 0.036)	< 0.02 (< 0.02, < 0.02)	0.066 (0.063, 0.069)	0.026 (0.025, 0.027)	
							40		0.026 (0.022, 0.029)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
Tillar, AR, 2001 (Pay-master)	WG	0.10	0.10	94–103	3	6–9	30	Delinted cottonseed	0.049 (0.047, 0.051)	< 0.02 (< 0.02, < 0.02)	0.070 (0.066, 0.074)	0.032 (0.031, 0.032)	
								Gin trash	1.200 (1.048, 1.352)	0.343 (0.321, 0.364)	0.478 (0.453, 0.502)	1.258 (1.138, 1.377)	
Senatobia, MS, 2001 (DPL 451 Bt/RR)	WG	0.10	0.05	187–196	3	7	29	Delinted cottonseed	0.049 (0.050, 0.048)	< 0.02 (< 0.02, < 0.02)	0.059 (0.059, 0.059)	0.027 (0.027, 0.027)	
Eakly, OK, 2001 (PM 2280)	WG	0.10	0.05	187	3	6–8	30	Delinted cottonseed	0.050 (0.044, 0.055)	< 0.02 (< 0.02, < 0.02)	0.077 (0.075, 0.078)	0.031 (0.029, 0.032)	
Dill City, OK, 2001 (Pay-master 2326)	WG	0.10	0.05–0.06	168–206	3	7	31	Delinted cottonseed	0.027 (0.026, 0.027)	< 0.02 (< 0.02, < 0.02)	0.213 (0.226, 0.199)	0.125 (0.126, 0.123)	
								Gin trash	2.537 (2.550, 2.523)	0.470 (0.468, 0.471)	0.591 (0.620, 0.562)	1.297 (1.363, 1.230)	

Location, year (variety)	Application						DALT, days	Commodity	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
Levelland, TX, 2001 (PM 2326 B6/RR)	WG	0.10	0.07	140	3	6–8	29	Delinted cottonseed	0.026 (0.032, 0.020)	< 0.02 (< 0.02, < 0.02)	0.252 (0.244, 0.260)	0.144 (0.138, 0.149)	
								Gin trash	1.878 (2.093, 1.663)	0.231 (0.275, 0.186)	0.370 (0.446, 0.293)	0.726 (0.881, 0.570)	
Uvalde, TX, 2001 (PM 2326 RR)	WG	0.10	0.05	187–196	3	7	30	Delinted cottonseed	0.024 (< 0.02, 0.027)	< 0.02 (< 0.02, < 0.02)	0.160 (0.146, 0.174)	0.089 (0.084, 0.094)	
Edmonson, TX, 2001 (Pay-master HS 250)	WG	0.10	0.06–0.07	150–187	3	7–8	0	Delinted cottonseed	0.114 (0.112, 0.115)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							11		0.033 (0.029, 0.037)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							20		0.038 (0.035, 0.040)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							32		0.035 (0.041, 0.028)	< 0.02 (< 0.02, < 0.02)	0.261 (0.305, 0.217)	0.149 (0.179, 0.118)	
							43		0.030 (0.032, 0.028)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	
							32	Gin trash	2.191 (2.411, 1.970)	0.327 (0.370, 0.283)	0.498 (0.604, 0.392)	1.039 (1.220, 0.857)	
Stanfield, AZ, 2001 (DP458 B1RR)	WG	0.10	0.06	187	3	7	29	Delinted cottonseed	0.038 (0.030, 0.045)	< 0.02 (< 0.02, < 0.02)	0.249 (0.265, 0.232)	0.179 (0.212, 0.146)	
Mariopa, AZ, 2001 (DP451 B1RR)	WG	0.10	0.05	187	3	7	30	Delinted cottonseed	0.072 (0.073, 0.070)	< 0.02 (< 0.02, < 0.02)	0.262 (0.260, 0.264)	0.195 (0.204, 0.185)	
								Gin trash	1.223 (1.241, 1.204)	0.331 (0.334, 0.327)	0.464 (0.466, 0.461)	1.169 (1.196, 1.141)	
Madera, CA, 2001 (Acala Riata RR)	WG	0.10–0.11	0.04	281–290	3	7	29	Delinted cottonseed	0.089 (0.115, 0.063)	< 0.02 (< 0.02, < 0.02)	0.227 (0.202, 0.251)	0.149 (0.126, 0.171)	
								Gin trash	1.224 (1.212, 1.235)	0.325 (0.338, 0.312)	0.461 (0.505, 0.416)	1.171 (1.204, 1.137)	
AUS GAP	WG	0.07	NS	NS	2	NS	7						
Mywybilla, Queensland, (Sicot 71BRF)	WG	0.10	0.111	91	2	14	7	Gin trash	2.3	0.2	0.19	0.35	
		0.10	0.121	85		14	27	Gin trash	0.21	< 0.05	0.38	0.43	
		0.20	0.216	94		14	7	Gin trash	8.39	0.46	1.13	2.43	
		0.20	0.246	82		14	27	Gin trash	0.38	0.07	0.83	1.2	
		0.10	0.134	74		15	7	Gin trash	1.33	0.15	0.19	0.33	
		0.10	0.128	76		14	15	Gin trash	0.32	0.069	0.38	0.37	
		0.10	0.130	76		14	22	Gin trash	0.41	0.07	0.35	0.53	
		0.10	0.131	74		14	29	Gin trash	0.37 (0.51, 0.23)	0.108 (0.16, 0.055)	0.54 (0.59, 0.49)	0.83 (1.13, 0.53)	
		0.10	0.130	77		14	36	Gin trash	0.15	< 0.01	0.61	0.9	
		0.10	0.129	77		14	43	Gin trash	0.086	< 0.05	0.47	1.22	
		0.20	0.265	75		15	7	Gin trash	1.02	0.059	0.086	0.11	
		0.20	0.264	77		14	29	Gin trash	0.40 (0.36, 0.44)	0.08 (0.061, 0.10)	0.35 (0.31, 0.38)	0.53 (0.47, 0.59)	
Boggabilla	WG	0.10	0.111	91	2	14	7	Gin trash	2.75	0.068	0.069	0.12	

Location, year (variety)	Application						DALT, days	Commodity	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
, New South Wales (Sicot 71BRF)		0.10	0.112	90		15	28	Gin trash	0.42	0.089	0.43	0.45	
		0.20	0.223	90		14	7	Gin trash	5.19	0.19	0.16	0.31	
		0.20	0.224	90		15	28	Gin trash	1.42	0.28	0.7	0.95	
Narrabi, New South Wales (Sicot 71BRF)	WG	0.10	0.111	90	2	14	7	Gin trash	3.72	0.057	0.086	0.086	
		0.10	0.113	89		15	13	Gin trash	2.42	0.15	0.18	0.11	
		0.10	0.112	89		14	21	Gin trash	0.68	0.12	0.78	0.24	
		0.10	0.11	92		13	28	Gin trash	0.56	0.17	1.06	0.61	
		0.10	0.108	93		14	35	Gin trash	0.16	< 0.01	0.94	0.43	
		0.10	0.113	90		14	41	Gin trash	0.2	0.06	2.66	1.97	
		0.20	0.221	92		14	7	Gin trash	8.23	0.19	0.16	0.15	
		0.20	0.22	92		13	28	Gin trash	0.99	0.27	2.41	1.16	
Chinchilla, Queensland (Sicot 71BRF)	WG	0.10	0.10	98	2	14	7	Gin trash	3.0	0.13	0.23	0.36	UPL GLP 12 01-1
		0.10	0.10	103		14	28	Gin trash	0.18	< 0.01	0.75	0.61	
		0.10	0.10	103		14	49	Gin trash	< 0.05	< 0.05	0.7	0.65	
		0.10	0.10	102		14	63	Gin trash	0.05	< 0.05	0.49	0.89	
		0.20	0.20	98		14	7	Gin trash	6.8	0.24	0.38	0.53	
		0.20	0.20	99		14	28	Gin trash	0.67	< 0.05	0.82	0.92	
		0.20	0.19	104		14	49	Gin trash	0.14	< 0.05	1.1	1.4	
		0.20	0.20	102		14	63	Gin trash	< 0.05	< 0.05	1.4	1.5	
Condamine Plains, Queensland (Sicot71BRF)	WG	0.10	0.10	100	2	14	7	Gin trash	1.7	0.47	0.23	0.55	
		0.10	0.09	111		15	20	Gin trash	1.1	0.29	0.36	0.52	
		0.10	0.10	103		14	27	Gin trash	1.1	0.16	0.29	0.38	
		0.10	0.10	103	1	NA	27	Gin trash	1.7	0.24	0.12	0.22	
		0.10	0.10	102	2	14	35	Gin trash	0.35	0.12	0.22	0.33	
		0.10	0.10	103		14	41	Gin trash	0.27	0.15	0.74	1.2	
		0.05	0.05	103		13	49	Gin trash	0.05	< 0.05	0.4	0.48	
		0.10	0.10	102		14	55	Gin trash	0.11	< 0.05	0.91	2.2	
		0.20	0.20	100		13	7	Gin trash	1.4	0.25	0.11	0.27	
		0.20	0.19	103		14	27	Gin trash	1.3	0.36	0.57	0.9	
		0.20	0.20	103		14	41	Gin trash	0.63	0.12	1.1	1.7	
		0.20	0.20	102		14	55	Gin trash	0.17	0.14	1.3	3.3	
Moree, New South Wales (Sicot 71BRF)	WG	0.10	0.11	89–91	2	14	7	Gin trash	1.2	< 0.05	< 0.05	< 0.05	
		0.10	0.11	91		14	14	Gin trash	1.3	0.08	0.05	0.07	
		0.10	0.11	91–92		14	21	Gin trash	0.74	< 0.05	0.19	0.22	
		0.10	0.11	90–94		14	28	Gin trash	0.56	< 0.05	0.45	0.27	
		0.10	0.11	92		14	35	Gin trash	0.71	0.06	1.4	1.2	
		0.10	0.11	92–93		14	42	Gin trash	0.6	< 0.05	0.44	0.97	
		0.10	0.11	89–92		14	49	Gin trash	0.33	0.05	0.76	1.8	
		0.10	0.11	90–93		14	56	Gin trash	0.07	< 0.05	0.28	0.72	
		0.20	0.22–0.23	89–93		14	7	Gin trash	1.7	< 0.05	< 0.05	< 0.05	
		0.20	0.22	90–91		14	28	Gin trash	1.5	0.05	0.21	0.16	
		0.20	0.22–0.23	90–93		14	42	Gin trash	0.98	0.07	0.75	1.5	
		0.20	0.22–0.23	90–93		14	56	Gin trash	0.22	< 0.05	1	2.4	

Location, year (variety)	Application						DALT, days	Commodity	Residues (mg/kg)				Ref
	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	RTI, days			Flonicamid	TFNA-AM	TFNA	TFNG	
Narrabri, New South Wales (Sicot 71BRF)	WG	0.10	0.11	90–92	2	14	8	Gin trash	1.6	0.07	0.05	0.07	UPL GLP 12 01-1
		0.10	0.11	89-90	2	14	15	Gin trash	0.82	0.06	0.13	0.13	
		0.10	0.11	91–92	2	14	22	Gin trash	0.89	0.05	0.14	0.23	
		0.10	0.11	89–90	2	14	29	Gin trash	0.66	0.07	0.34	0.57	
		0.10	0.11	91	2	14	36	Gin trash	0.58	0.07	0.54	1.1	
		0.10	0.11	88–91	2	14	43	Gin trash	0.31	0.05	0.37	0.73	
		0.10	0.11	90	2	14	50	Gin trash	0.36	0.08	0.8	1.7	
		0.10	0.11–0.12	84–86	2	14	57	Gin trash	0.09	< 0.05	0.67	1.6	
		0.23	0.22	92–93	2	14	8	Gin trash	2.1	0.2	0.12	0.18	
		0.21	0.22	92	2	14	29	Gin trash	1.1	0.11	0.48	1	
		0.20	0.22–0.24	85–92	2	14	43	Gin trash	0.42	0.07	0.96	1.8	
		0.20	0.24	85–86	2	14	57	Gin trash	0.15	< 0.05	0.91	1.9	
Narromine, New South Wales (Sicot 71BRF)	WG	0.10	0.08–0.09	114–125	2	14	7	Gin trash	0.66	< 0.05	< 0.05	< 0.05	
		0.10	0.08–0.09	116–119	2	14	28	Gin trash	0.15	< 0.05	1.2	0.62	
		0.10	0.08	123–124	2	11	42	Gin trash	< 0.05	< 0.05	0.44	0.24	
		0.10	0.08–0.09	117–123	2	15	53	Gin trash	< 0.05	< 0.05	1	0.49	
		0.20	0.17–0.18	114–119	2	14	7	Gin trash	1.6	< 0.05	0.13	0.07	
		0.21	0.17	122–124	2	14	28	Gin trash	1.2	0.14	1.1	1	
		0.21	0.17	119–123	2	11	42	Gin trash	< 0.05	< 0.05	0.87	0.41	
		0.21	0.17–0.18	118–123	2	15	53	Gin trash	< 0.05	< 0.05	0.58	0.27	

FATE OF RESIDUES DURING PROCESSING

In processing-nature of residues

Hydrolysis of flonicamid, radio-labelled in the pyridine ring (specific activity 9.08 MBq/mg), at 1.0 mg ai/L, was investigated in aqueous buffer solutions (0.1 M sodium citrate-citric acid), at 90 °C and pH4 for 20 min (simulating pasteurisation), at 100 °C and pH 5 for 60 min (simulating baking, brewing and boiling), and at 120 °C and pH 6 for 20 min (simulating sterilisation).

Quantitative measurement of the radioactivity was carried out by LSC. Further analysis to quantify and identify the radio-labelled degradation products present in the test solutions was conducted using HPLC and TLC. Flonicamid was identified by HPLC co-chromatography with a certified standard. Selected samples were analysed by TLC to confirm the presence of flonicamid.

Table 83 Degradation of flonicamid under various hydrolysis conditions

Condition	Sampling Regime	Flonicamid [% AR]	Total Others [% AR]	Total Recovery [% AR]
pH 4, 90 °C	Heated	100.28	0.49	100.77
	Control	99.21	0.59	99.80
pH 5, 100 °C	Heated	96.87	0.98	97.85
	Control	97.19	0.57	97.76
pH 6, 120 °C	Heated	96.58	1.72	98.30

Condition	Sampling Regime	Flonicamid [% AR]	Total Others [% AR]	Total Recovery [% AR]
	Control	96.47	0.92	97.39

Overall good recovery of radioactivity was achieved for each of the processing conditions, ranging from 94.6 to 101.1% of the applied radioactivity (AR).

In all cases, flonicamid accounted for at least 96.5% of AR. Therefore, very limited degradation of flonicamid was observed in aqueous buffer solutions under all the conditions tested with no significant degradation product being formed.

In processing-effect on the residue level

The Meeting received information on the fate of flonicamid residues and its metabolites TFNA-AM, TFNA and TFNG during the processing of raw agricultural commodities (RAC) in apples to juice; peaches to canned peaches, juice, jam and puree; plums to dried prunes; tomatoes to paste; potatoes to chips and flakes; rape seed and cotton to refined oil and meal and mint to oil.

Processing of apples

One study was conducted in 2002 in Lyons, New York where apple trees were treated with three foliar spray applications, where the first two treatments were at 0.103 kg ai/ha and the third treatment was at 0.516 kg ai/ha, for a total of 0.722 kg ai/ha. The fruit was harvested 21 days after the last application and transported to the lab for processing into juice and pomace. The results and the calculated processing factors (residue in processed commodity/residue in RAC) for MRL setting and dietary intake purposes are presented in Tables 84 and 93. To make juice and wet pomace, apples were ground in a hammer-mill. The resulting wet mash was loaded in one or more cloth stacks on the hydraulic press. The cloth stacks were pressed for 5 minutes at 2200–3000 psi to remove the apple juice. The wet pomace sample was then taken from the cloth stacks and bagged.

Table 84 Residues of flonicamid in apples (RAC and processed fractions)

RAC/ Processed commodity	Residues (mg/kg)				Processing Factor				Reference
	Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Apple fruit	0.032, 0.036 (0.034)	< 0.01	0.038, 0.041 (0.040)	< 0.01	–	–	–	–	IB-2001-MDG-003-00-01
Wet pomace	0.091, 0.101 (0.096)	< 0.01	0.049, 0.053 (0.051)	0.008, 0.008 (0.008)	2.82	NA	1.28	NA	
Juice	0.122, 0.127 (0.125)	< 0.01	0.139, 0.139 (0.139)	0.011, 0.011 (0.011)	3.67	NA	3.48	NA	

NA = Not applicable

Processing of peaches

Four processing trials were conducted in 2001 in Italy (two), Spain and Southern France where peach trees were treated with two applications of a WG formulation at a rate of 0.07 kg ai/ha/application for a total of 0.140 kg ai/ha. The peaches were harvested 14 days following the last application and processed into canned peaches, juice, jam and puree. The results and the calculated processing factors (residue in processed commodity/residue in RAC) for MRL setting and dietary intake purposes are presented in Tables 85 and 93. The information submitted on processing procedures is summarized as follows.

For each processed commodity, peaches were dipped in boiling water for a few minutes, peeled and stones removed.

Canned peaches

The fruits were cut in halves and placed in glass containers. Peaches were then covered with a 400 g/L sucrose solution. The containers were sealed and sterilized for 15 mins in a boiling water bath. The canned peaches were then cooled.

Juice

The fruits were cut into small pieces and weighed. The pulp was pressed through a sieve of 1 mm mesh size. The mixture was then centrifuged at 7500 rpm and filtered through a filter paper. The final volume of juice and sucrose content was measured and reported. Juice was transferred into a glass container, which was sealed and sterilized for 15 mins at 100 °C.

Jam

The fruits were cut in small pieces and weighed. A syrup solution was prepared by adding 50 mL of water to the same weight of sucrose as the quantity of peaches involved. The solution was cooked until complete dissolution of sucrose. The peaches were added to the syrup and cooked a few minutes before crushing. Pectin and citric acid were added to the mixture, corresponding to 0.5% and 0.6% in weight of sucrose added, respectively. The mixture was pressed through a sieve and the jam was cooked and controlled for sucrose concentration using a refractometer. Cooking was stopped as soon as 61% of sucrose concentration was achieved. The jam was transferred into a glass container, which was sealed and sterilized for 30 mins at 100 °C.

Purée

The fruits were cut into small pieces, weighed and transferred into a glass container. Sucrose was added equivalent to 10% of the weight of the peaches. The container was sealed and heated at 100 °C for 30 minutes. The syrup generated was removed and the volume was reported. The peaches were then crushed through a sieve. The weight of the resulting purée was reported and the sugar concentration was measured using a refractometer. The final sugar content was adjusted to 28% using sucrose. The purée was transferred into a glass container, which was sealed and sterilized for 15 min at 100 °C

Table 85 Residues of flonicamid in peaches (RAC and processed fractions)

Country, Year	RAC/Processed Commodity	Residues (mg/kg)				Processing Factor				Reference
		Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Italy, 2001	Peaches	0.03	< 0.01	< 0.01	< 0.01	–	–	–	–	P-22-01-02
	Canned peaches	< 0.01	< 0.01	< 0.01	< 0.01	0.33	NA	NA	NA	
	Fruit juice	0.03	< 0.01	0.01	0.01	1	NA	NA	NA	
	Jam	0.01	< 0.01	0.02	0.04	0.33	1	2	4	
	Purée	0.02	< 0.01	< 0.01	< 0.01	0.67	NA	NA	NA	
	Peel	0.01	< 0.01	< 0.01	< 0.01	0.33	NA	NA	NA	
	Waste material out of purée	0.02	< 0.01	< 0.01	< 0.01	0.67	NA	NA	NA	
	Blanching water	0.03	< 0.01	0.01	0.01	1	NA	NA	NA	
	Waste material out of juice	0.02	< 0.01	< 0.01	< 0.01	0.67	NA	NA	NA	
Italy, 2001	Peaches	0.02	< 0.01	< 0.01	< 0.01	–	–	–	–	
	Canned peaches	< 0.01	< 0.01	< 0.01	< 0.01	0.5	NA	NA	NA	
	Fruit juice	0.02	< 0.01	< 0.01	< 0.01	1	NA	NA	NA	
	Jam	0.02	< 0.01	0.01	< 0.01	1	NA	NA	NA	

Country, Year	RAC/Processed Commodity	Residues (mg/kg)				Processing Factor				Reference
		Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
	Purée	0.02	< 0.01	< 0.01	< 0.01	1	NA	NA	NA	
Spain, 2001	Peaches	0.03	< 0.01	< 0.01	< 0.01	–	–	–	–	
	Canned peaches	0.01	< 0.01	< 0.01	< 0.01	0.33	NA	NA	NA	
	Fruit juice	0.01	< 0.01	< 0.01	< 0.01	0.33	NA	NA	NA	
	Jam	0.03	< 0.01	< 0.01	< 0.01	1	NA	NA	NA	
	Purée	0.03	< 0.01	< 0.01	< 0.01	1	NA	NA	NA	
South of France, 2001	Peaches	0.06	< 0.01	0.02	0.01	–	–	–	–	
	Canned peaches	0.10	< 0.01	0.02	0.01	1.67	NA	1	1	
	Fruit juice	0.03	< 0.01	0.03	< 0.01	0.5	NA	1.5	NA	
	Jam	0.01	< 0.01	0.03	< 0.01	0.17	NA	1.5	NA	
	Purée	0.05	< 0.01	0.03	< 0.01	0.83	NA	1.5	NA	

Processing of plums

One processing trial was conducted in 1992 in Fairfield, California where plum trees were treated with three foliar spray applications where the first two treatments were at 0.103 kg ai/ha and the third treatment was at 0.516 kg ai/ha, for a total of 0.722 kg ai/ha. The fruit was harvested 14 days after the last application and dried. The results and the calculated processing factors (residue in processed commodity/residue in RAC) for MRL setting and dietary intake purposes are presented in Tables 86 and 93. The information submitted on processing procedures is summarized as follows.

Dried prune

The plums were washed for five minutes in a tub of cold water. The washed plums were spread single layer on trays and dehydrated in a tray air dryer at 68–79 °C for 18–36 hours to reduce the moisture content to the desired range (19–29%).

Table 86 Residues of flonicamid in plums (RAC and processed fractions)

RAC/ Processed commodity	Residues (mg/kg)				Processing Factor				Reference
	Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Plum	0.280 (0.275, 0.284)	0.024 (0.025, 0.023)	0.016, (0.016 0.016)	0.032 (0.033, 0.031)	–	–	–	–	IB-2001_MDG-004-00-01
Dried prune	0.278 (0.264, 0.287)	0.018 (0.017, 0.018)	0.024 (0.026, 0.021)	0.036 (0.038, 0.034)	1	0.75	1.5	1.13	

Processing of tomato

One study was conducted in 2001 in Davis, California where tomato plants were treated with three foliar spray applications where the first two treatments were at 0.102 kg ai/ha and the third treatment was at 0.506 kg ai/ha, for a total of 0.710 kg ai/ha. The fruit was harvested immediately after the last application and transported to the lab for processing into paste. The results and the calculated processing factors (residue in processed commodity/residue in RAC) for MRL setting and dietary intake purposes are presented in Table 93. The information submitted on processing procedures is summarized as follows.

Paste

The tomato fruit were batch rinsed using a high-pressure spray rinse at approximately 70–75 °C for 30 seconds per batch. The fruit was hand fed into the hammermill assembly of the Suntech Fruit Press for crushing. The crushed tomatoes were transferred to the Hubbert Steam Jacketed Kettle and rapidly heated to approximately 80–85 °C and held for 25–30 seconds. The hot break juice was hand fed into the Pulper Finisher for the separation of pomace and juice. The wet pomace recovered was pressed using the Suntech Fruit Press. The pressed wet pomace was discarded and the recovered press juice was returned to the finished juice.

The juice was then transferred to the Groen Vacuum Evaporator. The puree was removed from the evaporator when the desired Brix range was achieved. A portion of the puree was transferred to the 7.5 L Scrape Surface Vacuum Evaporator. The paste was removed from the evaporator when the desired Brix range was achieved. A portion of the paste was removed and 1% salt was added to adjust the Brix to the desired range of 24.0–30.0 °C. The paste was heated to 82–88 °C. The heated paste was packed in 3303 cans and sealed using the Dixie Electric Can Sealer. The sealed cans were then processed using an Open Atmospheric Water Bath Kettle for 15–20 minutes at 96–100 °C and then cooled under running tap water. A representative sample of the cooled canned puree was removed, packaged, labelled and placed in the freezer for the required sample fraction. The excess evaporated puree and paste was discarded.

Table 87 Residues of flonicamid in tomato (RAC and processed fractions)

RAC/ Processed commodity	Residues (mg/kg)				Processing Factor				Reference
	Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Tomato	0.031 (0.029, 0.031, 0.033, 0.031)	< 0.01 (< 0.01, < 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01, < 0.01)	–	–	–	–	IB-2001-MDG-006-00-1
Paste	0.499 (0.494, 0.503)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.028, 0.029 (0.029)	16.1	NA	NA	2.8	

Processing of potato

One study was conducted in 2001 in Ephrata, Washington where potato plants were treated with three foliar spray applications where the first two treatments were at 0.10 kg ai/ha and the third treatment was at 1.0 kg ai/ha, for a total of 1.22 kg ai/ha. The fruit was harvested immediately after the last application and transported to the lab for processing into chips and flakes. The results and the calculated processing factors (residue in processed commodity / residue in RAC) for MRL setting and dietary intake purposes are presented in Tables 88 and 93. The information submitted on processing procedures is summarized as follows.

Chips

The potatoes are peeled for 25–35 seconds in batches using an abrasive peeler. A certain amount of peel is left on the tuber to produce a natural appearance to the finished product. The peel collected is weighed and discarded. The peeled potatoes are individually inspected and hand trimmed. The potatoes are cut using a restaurant style food cutter/slicer. The slices are placed in a tub of hot tap water to rinse the free starch from the surfaces of the slices. The slices are deep fried in a restaurant style deep fat fryer at approximately 163–191 °C for 60–90 seconds. Free oil is drained from the chips using a draining tray and salted by hand. The chips are inspected and undesirable chips are removed.

Flakes

Washed potatoes are sorted and scrubbed in batches using a restaurant style peeler fitted with a rubber scrubber for approximately 25–35 seconds. The peel is then hydraulically pressed to increase the solids content. Potatoes are then individually inspected, hand trimmed and cut into slabs using a restaurant style food cutter/slicer with a cutting blade set to approximately 1–1.3 cm. The potato slices are spray washed for approximately 30 seconds in cold water to rinse the free starch from the surface of the slices. The potato slices are precooked at approximately 70–77 °C for 202–22 minutes using a steam jacketed kettle and subsequently cooled down to less than 32 °C using cold running tap water in a 150 L steam jacketed kettle for 20–22 minutes. The cooled slices are steam cooked at 94–100 °C for 40–42 minutes using an atmospherically flowing steam batch style steam cooker and mashed using a restaurant style meat grinder without the grinding attachment. The mashed potatoes are placed in a bakery style mixer where an emulsion containing the additives are poured into the mashed potatoes and mixed for approximately 60 seconds. The potato mash is hand fed onto a laboratory single drum dryer where the potato mash is dried into a thin sheet. The dried potato sheet is broken into flakes. The large flakes are then hand fed into a hammermill for uniform sizing of the finished flakes. If moisture content of the potato flakes exceeds 9%, the flakes are dried on the fluidized bed dryer to less than or equal to 9% moisture.

Table 88 Residues of flonicamid in potatoes (RAC and processed fractions)

RAC/ Processed commodity	Residues (mg/kg)				Processing Factor				Reference
	Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Potatoes	0.022 (0.022, 0.022)	< 0.01 (< 0.01, < 0.01)	0.041 (0.040, 0.041)	0.029 (0.030, 0.028)	–	–	–	–	IB-2001-MDG-002-00-01
Wet Peel	0.011 (0.010, 0.011)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.50	NA	0.24	0.3	
Chips	0.021 (0.021, 0.021)	< 0.01 (< 0.01, < 0.01)	0.072 (0.071, 0.072)	0.051 (0.049, 0.053)	0.95	NA	1.8	1.8	
Flakes	0.060 (0.059, 0.060)	< 0.01 (< 0.01, < 0.01)	0.122 (0.117, 0.126)	0.092 (0.089, 0.094)	2.73	NA	2.98	3.17	

The best estimates of the processing factors for parent residues (for MRL setting in case of residue increasing) and for the sum of flonicamid, TFNA-AM, TFNA AND TFNG (for dietary intake) are summarized in Table 93.

Rape seed

One study was conducted in 2007 in Prosser, Washington where rape seed plants were treated with three foliar spray applications of 0.30 kg ai/ha for a total of 0.90 kg ai/ha. The seeds were harvested 8 days following the last application and transported to the lab for processing into refined oil and meal. The results and the calculated processing factors (residue in processed commodity/residue in RAC) for MRL setting and dietary intake purposes are presented in Table 93. The information submitted on processing procedures is summarized as follows.

Refined Oil

Canola seeds were flaked in a flaking roll and flakes were heated to 85–100 °C and held for 10 to 15 minutes in the temperature range. Flakes were pressed (expelled) in an expeller to mechanically remove a portion of the crude oil. Residual crude oil remaining in the solid material (presscake) exiting the expeller was extracted with the hexane. The miscella (crude oil and hexane) was passed through a laboratory recovery unit to separate the crude oil and hexane. Crude oil was heated to 90–96 °C for hexane removal. Crude oil samples recovered from the expeller and solvent extraction were filtered and combined. Percent free fatty acid (FFA) for the crude oil was determined. Crude canola oil was placed in a water bath and pre-treated with 85% phosphoric acid. Oil was mixed for 29–31 minutes at 40–45 °C. After the pre-treatment, an amount of 12°Baume sodium hydroxide was added to the oil. The samples were mixed for 19–21 minutes at 40–45 °C and then for 9–11 minutes at 65–70 °C. The neutralized oil was then centrifuged to separate the refined oil and soapstock. The refined oil was decanted and filtered. Soapstock was discarded. Resulting fraction of alkali refined oil was collected and frozen.

Meal

Presscake was placed in stainless steel batch extractors and submerged in 50–60 °C solvent (hexane). After 30 minutes, the hexane was drained and fresh hexane added to repeat the cycle two more times. The final two washes were for 15–30 minutes each. After the final draining, the extracted presscake (meal) was desolventized using warm air forced through the extracted presscake. Resulting fraction, canola meal was collected and placed into frozen storage.

Table 89 Residues of flonicamid in rape seed (RAC and processed fractions)

RAC/ Processed commodity	Residues (mg/kg)				Processing Factor				Reference
	Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Whole seed	0.232	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	–	–	–	–	9783
Meal	< 0.02	< 0.02	< 0.02	< 0.02	< 0.1	NA	NA	NA	
Refined oil	< 0.02	< 0.02	< 0.02	< 0.02	< 0.1	NA	NA	NA	

Processing of cotton

Study 1: US

One processing trial was conducted in 2001 in Uvalde, Texas, where cotton was treated with three applications of a WG formulation where the first two treatments were made at a rate 0.10 kg ai/ha/application and the third treatment was made at a rate of 1.0 kg ai/ha/application for a total of 1.2 kg ai/ha. The undelinted cottonseed was harvested 30 days following the last application and processed into meal and oil. The results and the calculated processing factors (residue in processed commodity/residue in RAC) for MRL setting and dietary intake purposes are presented in Tables 90 and 93. The information submitted on processing procedures is summarized as follows.

Cottonseed hulls

Cotton seed was dried and cleaned followed stick extraction to remove the gin trash. The lint cotton was saw ginned to remove 85–89% of the lint from the cottonseed. The ginned seed was saw delinted to remove most of the remaining linters. Approximately 3% of the lint remained with the seed. A mill was used to crack the seed and the hulls were removed from the kernels and sampled for analysis.

Cottonseed oil and meal

The kernels were dried to < 12% water, heated to 80–90 ºC for 30 minutes, and flaked, followed by passage through an expander extruder to form collets. The collets were submerged in hexane at 50–60 ºC for 30 minutes and washed twice with fresh hexane to remove the cottonseed oil. Residual hexane was removed from the meal fractions with warm air and the meal was sampled for analysis. Hexane was removed from the oil with a vacuum extractor, NaOH was added to precipitate the soap stock, the remaining hexane was removed and refined oil was sampled for analysis.

Table 90 Residues of flonicamid in cotton (RAC and processed fractions)—US

RAC/	Residues (mg/kg)	Processing Factor	Reference

Processed commodity	Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Seed at processing	0.084 (0.079, 0.088)	< 0.02 (< 0.02, < 0.02)	0.101 (0.092, 0.110)	0.080 (0.070, 0.090)	–	–	–	–	IB-2001-MDG-004-00-01
Hulls	0.071 (0.072, 0.069)	< 0.02 (< 0.02, < 0.02)	0.353 (0.351, 0.354)	0.210 (0.207, 0.212)	0.84	NA	3.5	2.6	
Meal	0.023 (0.023, 0.023)	< 0.02 (< 0.02, < 0.02)	0.899 (0.894, 0.883)	0.483 (0.489, 0.476)	0.27	NA	8.9	6.0	
Refined oil	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.02 (< 0.02, < 0.02)	< 0.24	NA	0.20	0.25	

Study 2—Australia

One processing trial was conducted in 2012 in Narrabri, New South Wales, where cotton was treated with two applications of a WG formulation at a rate 0.10 kg ai/ha/application or 0.20 kg ai/ha/application for a total of 0.2 kg ai/ha or 0.4 kg ai/ha, respectively. The undelinted cottonseed was harvested 8, 15, 22, 29, 36, 43, 50 and 57 DALA and processed into meal and oil. The results and the calculated processing factors (residue in processed commodity/residue in RAC) for MRL setting and dietary intake purposes are presented in Tables 91 and 93. The information submitted on processing procedures is summarized as follows.

Cottonseed hulls

The fuzzy seed was passed through a hand driven mechanical grinder to crack the hulls. The cracked fuzzy seed was sieved to separate the hulls from the unprocessed meal.

Cottonseed meal and oil

The unprocessed meal was placed in a small bolt apparatus which was screwed together to press the meal and extract the oil via pressure. Oil was collected during pressing from the drain hole on the apparatus using a syringe and collected in a plastic vial. The process was repeated until at least 1 m of oil was collected.

Table 91 Residues of flonicamid in cotton (RAC and processed fractions)—Australia

RAC/Processed Commodity	DALT	Residues (mg/kg)				Processing Factor				Reference
		Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Total Application Rate of 0.20 kg ai/ha										UPL GLP 12 01-1
Seed at processing	8	0.034	0.071	0.025	0.048	–	–	–	–	
Hulls		0.13	< 0.02	< 0.02	< 0.02	3.8	0.3	0.8	0.4	
Meal		0.15	0.03	< 0.02	0.02	4.4	0.4	0.8	0.4	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.6	0.3	0.8	0.4	
Seed at processing	15	0.067	0.033	0.019	0.054	–	–	–	–	
Hulls		0.04	< 0.02	< 0.02	< 0.02	0.6	0.6	1.0	0.4	
Meal		< 0.02	< 0.02	< 0.02	< 0.02	0.3	0.6	1.0	0.4	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.3	0.6	1.0	0.4	
Seed at processing	22	0.11	0.047	0.043	0.12	–	–	–	–	
Hulls		0.05	< 0.02	< 0.02	0.02	0.4	0.4	0.5	0.2	
Meal		0.04	0.03	0.03	0.06	0.4	0.6	0.7	0.5	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.2	0.4	0.5	0.2	
Seed at processing	29	0.13	0.069	0.051	0.16	–	–	–	–	
Hulls		0.04	< 0.02	< 0.02	< 0.02	0.3	0.3	0.4	0.1	

RAC/Processed Commodity	DALT	Residues (mg/kg)				Processing Factor				Reference
		Flonicamid	TFNA-AM	TFNA	TFNG	Flonicamid	TFNA-AM	TFNA	TFNG	
Meal		0.05	0.05	0.08	0.12	0.4	0.7	1.6	0.8	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.2	0.3	0.4	0.1	
Seed at processing	36	0.088	0.076	0.075	0.23	–	–	–	–	
Hulls		0.10	< 0.02	0.04	0.02	1.1	0.3	0.5	0.09	
Meal		0.02	0.02	0.12	0.10	0.2	0.3	1.6	0.4	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.2	0.3	0.3	0.09	
Seed at processing	43	0.032	0.029	0.064	0.12	–	–	–	–	
Hulls		0.04	< 0.02	0.02	< 0.02	1.2	0.7	0.3	0.2	
Meal		< 0.02	0.02	0.06	0.07	0.6	0.7	0.9	0.6	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.6	0.7	0.3	0.2	
Seed at processing	50	0.025	0.028	0.098	0.16	–	–	–	–	
Hulls		0.06	< 0.02	0.04	0.02	2.4	0.7	0.4	0.1	
Meal		0.02	0.03	0.15	0.18	0.8	1.1	1.5	1.1	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.8	0.7	0.2	0.1	
Seed at processing	57	< 0.02	< 0.02	0.10	0.12	–	–	–	–	
Hulls		0.02	< 0.02	0.04	0.02	NA	NA	0.4	0.2	
Meal		< 0.02	< 0.02	0.11	0.09	NA	NA	1.1	0.8	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	NA	NA	0.2	0.2	
Total Application Rate of 0.40 kg ai/ha										
Seed at processing	8	0.48	0.72	0.26	0.049	–	–	–	–	
Hulls		0.26	< 0.02	< 0.02	< 0.02	0.54	0.03	0.08	0.41	
Meal		0.12	0.03	< 0.02	0.02	0.25	0.04	0.08	0.41	
Refined oil		0.02	< 0.02	< 0.02	< 0.02	0.04	0.03	0.08	0.41	
Seed at processing	29	0.11	0.082	0.053	0.17	–	–	–	–	
Hulls		0.08	< 0.02	0.02	< 0.02	0.7	0.2	0.4	0.1	
Meal		0.02	0.02	0.05	0.06	0.2	0.2	0.9	0.4	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.2	0.2	0.4	0.1	
Seed at processing	43	0.096	0.097	0.13	0.33	–	–	–	–	
Hulls		0.09	0.02	0.04	0.02	0.9	0.2	0.3	0.1	
Meal		0.03	0.03	0.13	0.12	0.3	0.3	1.0	0.4	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.2	0.2	0.2	0.1	
Seed at processing	57	0.022	0.025	0.12	0.19	–	–	–	–	
Hulls		0.03	< 0.02	0.05	0.02	1.4	0.8	0.4	0.1	
Meal		< 0.02	0.03	0.19	0.20	0.9	1.2	1.6	1.1	
Refined oil		< 0.02	< 0.02	< 0.02	< 0.02	0.9	0.8	0.2	0.1	

Mint

Two processing trials were conducted in 2011 in Moxee, Washington and Endeavour and Wisconsin. Mint was treated with three applications of a SG formulation at a rate 0.10 kg ai/ha/application for a total of 0.3 kg ai/ha. The mint tops were harvested 7 days following the last application and processed into oil. The results and the calculated processing factors (residue in processed commodity/residue in RAC) for MRL setting and dietary intake purposes are presented in Tables 92 and 93. No information was submitted on processing procedures.

The maximum storage intervals for mint oil was 368 days. Concurrent storage stability samples were fortified with flonicamid and its metabolites at 0.2 ppm soon after the receipt of the samples by the analytical laboratory. The storage stability samples were held in frozen storage under similar conditions to the field generated samples. After 334 days of freezer storage for mint oil, the storage stability samples were analysed for flonicamid. The recoveries for the mint oil storage stability samples were in the ranges 43–46% (flonicamid), 42–49% (TFNA), 46–53% (TFNA-AM), and 42–45% (TFNG). Concurrent recoveries for spikes analysed along with these storage stability samples were 95% (flonicamid), 100% (TFNA), 81% (TFNA-AM), and 89% (TFNG). These data indicate that flonicamid and its metabolites undergo about 50% degradation in mint oil under the conditions which the samples were held between harvest and analysis. However, even when correcting for in-storage dissipation, residues of flonicamid and its metabolites do not concentrate in mint oil.

Table 92 Residues of flonicamid in mint (RAC and processed fractions)

RAC/ Processed commodity	Residues (mg/kg)					Average Processing Factors					Reference
	Flonicamid	TFNA-AM	TFNA	TFNG	Sum	Flonicamid	TFNA-AM	TFNA	TFNG	Sum	
Mint tops	1.57 (1.55, 1.59)	0.339 (0.329, 0.349)	0.171 (0.170, 0.171)	0.193 (0.193, 0.193)	2.273	< 0.03 (< 0.01, < 0.04)	< 0.08 (< 0.06, < 0.09)	< 0.20 (< 0.12, < 0.27)	< 0.14 (< 0.10, < 0.18)	< 0.07 (< 0.04, < 0.09)	9358
Mint oil	< 0.02	< 0.02	< 0.02	< 0.02	< 0.08						
Mint tops	0.502 (0.500, 0.504)	0.222 (0.219, 0.225)	0.074 (0.072, 0.075)	0.108 (0.107, 0.108)	0.906						
Mint oil	< 0.02	< 0.02	< 0.02	< 0.02	< 0.08						

Table 93 Summary of processing factors for flonicamid residues

RAC	Processed Commodity	Calculated processing factors	Best estimate
		Flonicamid	
Apples	Juice	3.7	3.7
	Pomace	2.82	2.82
Peaches	Canned peaches	0.3, 0.5, 0.3, 1.7	0.7 (median)
	Juice	1.0, 1.0, 0.3, 0.5	0.8 (median)
	Jam	0.3, 1.0, 1.0, 0.2	0.7 (median)
	Puree	0.7, 1.0, 1.0, 0.8	0.9 (median)
Plums	Dried prunes	1.0	1.0
Tomato	Paste	16.1	16.1
Potato	Chips	0.95	0.95
	Flakes	2.7	2.7
Canola	Refined oil	< 0.1	0.1
	Meal	< 0.1	0.1
Cotton	Refined oil	< 0.24 (US); 0.6 and 0.04 (AUS)	0.32 (mean; AUS)
	Hulls	0.8 (US); 3.8, 0.5 (AUS)	2.2 (mean; AUS)
	Meal	0.3 (US); 4.4, 0.2 (AUS)	2.3 (mean; AUS)
Mint	Oil	< 0.03	0.03

Residues in animal commodities

Dairy Cattle

One cattle feeding study was conducted where twelve dairy cows (Red Holstein and Simmentaler Fleckvieh, 4–9 years old, 550–770 kg bw) were divided into three groups. Animals were treated twice daily with a 1/1 mixture of flonicamid/TFNG by means of gelatin capsules and using a balling gun. Treatments were made after the morning and evening milking for 28 consecutive days. One group of three cows served as a control group The actual average doses administered were 0.086, 0.252 and 0.839 mg/kg bw. Based on the actual average daily feed intake of 20.1–25.1 kg/day (or 3.0–4.4 kg/day/100 kg bw) during the acclimation period, the actual dosing levels, constituting a 1/1 mixture of flonicamid/TFNG, were 2.50 mg, 6.89 mg and 23.69 mg/kg feed. All cows were sacrificed after 28 days of dosing, within 24 hours after the last dose.

Milk samples were collected on 15 selected days throughout the administration period. All milk samples were frozen at –20 ºC and analysed within 30 days after sampling. Therefore, storage stability data are not necessary. In contrast, all tissue samples were analysed within 12 months of collection. Freezer storage stability studies, conducted concurrently with the feeding studies, demonstrated that flonicamid, TFNA, TFNA-AM, OH-TFNA-AM and TFNG were stable for 374 days in all tissues except fat. For fat, flonicamid and its metabolites were demonstrated to be stable for 315 days.

All samples were analysed for residues of flonicamid, TFNA, TFNA-AM, OH-TFNA-AM and TFNG using validated analytical methods. In general, the samples were homogenised, extracted and the supernatant was purified by means of liquid-liquid partition or gel permeation chromatography. Some of the solid residues were further subjected to acid hydrolysis. The concentration of flonicamid and its metabolites in the purified extracts were determined by HPLC MS/MS. The LOQ for flonicamid and each of its metabolites in milk and fat is 0.01 mg/kg and for muscle the LOQ is 0.025 mg/kg while for liver and kidney the LOQ is dependent on the method used (0.01 or 0.025 mg/kg).

In milk, no quantifiable (< LOQ) residues of flonicamid, TFNG and TFNA were detected in any test group. For TFNA-AM, the average residues increased from < LOQ in the low dose group to 0.02 mg/kg in the mid dose group and to 0.08 mg/kg in the high dose group. OH-TFNA-AM average residues were ≤ LOQ in the low and mid-dose groups and increased to 0.015 mg/kg in the high dose group.

Table 94 Residues in whole milk following 28 days oral administration of flonicamid to dairy cows

Low Dose (2.5 mg/kg feed)			Mid dose (6.89 mg/kg feed)		High dose (23.69 mg/kg feed)	
Day	TFNA-AM (mg/kg)	OH-TFNA-AM (mg/kg)	TFNA-AM (mg/kg)	OH-TFNA-AM (mg/kg)	TFNA-AM (mg/kg)	OH-TFNA-AM (mg/kg)
1	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.032 (0.035, 0.032, 0.027)	< 0.01 (< 0.01, < 0.01, < 0.01)
2	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.018 (0.019, 0.020, 0.014)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.080 (0.091,0.088, 0.060)	0.015 (0.021, 0.011, 0.011)
3	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.022 (0.024, 0.022, 0.018)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.086 (0.111, 0.093, 0.074)	0.016 (0.027, 0.013, 0.014)
4	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.023 0.025, 0.026, 0.019)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.093 (0.111, 0.093, 0.075)	0.018 (0.027, 0.013, 0.014)
5	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.030 (0.027, 0.042, 0.021)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.082 (0.109, 0.094, 0.042)	0.017 (0.027, 0.016, < 0.01)
6	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.026 (0.024, 0.030, 0.022)	0.0101 (0.0102, < 0.01, < 0.01)	0.092 (0.105, 0.093, 0.078)	0.019 (0.026, 0.015, 0.015)
7	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.024 (0.026, 0.027, 0.019)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.085 (0.094, 0.081, 0.081)	0.016 (0.021, 0.013, 0.014)
8	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.020 (0.024, 0.022, 0.012)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.069 (0.085, 0.062, 0.0601)	0.013 (0.018, < 0.01, < 0.01)
10	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.018 (0.016, 0.022, 0.017)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.049 (0.019, 0.069, 0.058)	0.013 (0.016, < 0.01, 0.012)
14	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.018 (0.018, 0.019, 0.016)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.068 (0.074, 0.075, 0.056)	0.013 (0.018, 0.010, 0.011)

Low Dose (2.5 mg/kg feed)			Mid dose (6.89 mg/kg feed)		High dose (23.69 mg/kg feed)	
17	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.017 (0.017, 0.019, 0.014)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.079 (0.077, 0.090, 0.069)	0.014 (0.019, 0.012, 0.010)
21	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.019 (0.019, 0.023, 0.016)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.076 (0.079, 0.092, 0.058)	0.014 (0.020, 0.012, 0.011)
24	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.023 (0.024, 0.024, 0.022)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.089 (0.097, 0.099, 0.071)	0.018 (0.023, 0.014, 0.015)
27	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.022 (0.022, 0.023, 0.020)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.077 (0.090, 0.088, 0.054)	0.013 (0.018, 0.011, 0.010)
29	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.021 (0.020, 0.023, 0.021)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.086 (0.101, 0.088, 0.068)	0.013 (0.018, < 0.01, < 0.01)

In liver, TFNA-AM and OH-TFNA-AM were detected in the mid and high dose groups above the LOQ using two different analytical methods (FMC-P-3580 / RCC 844743) with different LOQ (0.025/0.01 mg/kg). TFNA-AM levels increased from less than LOQ in the low dose group to 0.039/0.015 mg/kg in the mid dose group and 0.113/0.053 mg/kg in the high dose group. OH-TFNA-AM levels increased from levels below LOQ (0.025/0.01 mg/kg) in the low dose group to levels slightly above the LOQ (< 0.025/0.010 mg/kg) in the mid dose group and 0.030/0.037 mg/kg in the high dose group.

In kidney, TFNA and TFNA-AM were detected in the medium and high dose groups above the LOQ using the same analytical methods as those used for kidney. OH-TFNA-AM and TFNG were only detected above the LOQ (0.025/0.01 mg/kg) in the high dose group. TFNA levels increased from levels below LOQ in the low dose group to 0.043/0.038 mg/kg in the mid dose group and 0.142/0.135 mg/kg in the high dose group. TFNA-AM levels increased from levels below LOQ in the low dose group to 0.031/0.023 mg/kg in the mid dose group and 0.105/0.088 mg/kg in the high dose group. OH-TFNA-AM levels increased from levels below LOQ in the low and mid dose group to 0.025/0.027 mg/kg in the high dose group. TFNG levels increased from levels below LOQ in the low and mid dose group to 0.010 mg/kg in the high dose group.

Table 95 Residues in liver and kidney following 28 days oral administration to dairy cows

	Liver				Kidney							
	Solvent extraction		Hydrolysis		Solvent extraction				Hydrolysis			
Dose (mg/kg feed)	TFNA-AM	OH-TFNA-AM	TFNA-AM	OH-TFNA-AM	TFNA-AM	OH-TFNA-AM	TFNG	TFNA	TFNA-AM	OH-TFNA-AM	TFNG	TFNA
2.5	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.025 (< 0.025, < 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025, < 0.025)	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.016 (0.014, 0.019, 0.014)	< 0.025 (< 0.025, < 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025, < 0.025)
6.89	0.015 (0.010, 0.019, 0.015)	0.010 (< 0.01, < 0.01, 0.011)	0.039 (0.042, 0.040, 0.034)	< 0.025 (< 0.025, < 0.025, < 0.025)	0.023 (0.020, 0.024, 0.025)	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.038 (0.032, 0.041, 0.041)	0.031 (0.027, 0.034, 0.032)	< 0.025 (< 0.025, < 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025, < 0.025)	0.043 (0.039, 0.047, 0.045)

	Liver				Kidney							
	Solvent extraction		Hydrolysis		Solvent extraction				Hydrolysis			
23.69	0.053 (0.056, 0.052, 0.051)	0.037 (0.051, 0.034, 0.026)	0.113 (0.124, 0.124, 0.091)	0.028 (0.035, < 0.025, < 0.025)	0.088 (0.112, 0.083, 0.070)	0.027 (0.038, 0.021, 0.022)	0.01 (0.01, < 0.01, < 0.01)	0.135 (0.166, 0.108, 0.132)	0.105 (0.124, 0.092, 0.099)	0..025 (0.025, < 0.025, < 0.025)	< 0.025 (< 0.025, < 0.025, < 0.025)	0.142 (0.173, 0.107, 0.146)

In muscle, only TFNA-AM was found. The level increased from below LOQ (0.025 mg/kg) in the low dose group to 0.027 mg/kg in the mid dose group and 0.088 mg/kg in the high dose group.

Similarly, only TFNA-AM was measurable in fat and only at the high dose level (0.015 mg/kg).

Table 96 TFNA-AM residues in fat and muscle following 28 days oral administration to dairy cows

Dose (mg/kg feed)	TFNA-AM (mg/kg)	
	Fat	Muscle
2.5	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.025 (< 0.025, < 0.025, < 0.025)
6.89	< 0.01 (< 0.01, < 0.01, < 0.01)	0.027 (< 0.025, 0.027, 0.030)
23.69	0.015 (0.021, 0.013, 0.011)	0.057 (0.010, 0.088, 0.072)

Poultry

Flonicamid and TFNG were dosed in a 1:1 mixture to 50, 22 week-old, laying hens (white leghorn hybrids) weighing on average 1.54 kg with an egg production of at least 0.8 eggs per day. The hens were randomly assigned to five groups, one of which served as control group. Each group was separated into three subgroups of three to four animals. After the acclimation period, the test substance was orally administered, once daily in the afternoon by means of gelatin capsules (after egg sampling). for 28 days. Based on the actual average daily feed intake of 0.108–0.116 kg/hen during the 4-week acclimation period, the actual dose levels were equivalent to average potential concentrations in the feed of 0.26, 2.51, 7.47 and 25.83 mg flonicamid/TFNG per kg feed.

Eggs were collected once daily and pooled per subgroup of three or four hens, resulting in three unique samples of eggs for each dose level. The egg pools were stored at –20 °C until analysis. The animals were sacrificed for tissue sampling the day after the last administration, 24 hours after the last dosing. Liver fat (composite of skin fat) and muscle were excised. Tissue samples were rinsed, weighed and labelled. Tissues were pooled per subgroup of three or four hens, homogenised and stored deep-frozen until analysis. Egg and tissue samples were stored for greater than 30 days. Freezer storage stability studies, conducted concurrently with the feeding studies, demonstrated that flonicamid, TFNA, TFNA-AM, OH-TFNA-AM and TFNG were stable for 8–10 months in eggs and tissues.

All samples were analysed for residues of flonicamid, TFNA, TFNA-AM, OH-TFNA-AM and TFNG using a validated analytical method. The samples were homogenised, extracted and the supernatant was purified by means of gel permeation chromatography. The concentration of flonicamid and its metabolites in the purified extracts were determined by MS/MS detection using HPLC for separation.

In eggs, no quantifiable (< LOQ) residues of TFNA, OH-TFNA-AM and TFNG were detected in any test group. For flonicamid, the average residues increased from < LOQ in the very low and low dose groups to 0.02 mg/kg in the mid dose group and to 0.08 mg/kg in the high dose group. TFNA-AM average residues increased from < LOQ in the very low and low dose groups, to 0.27 mg/kg in the mid dose group and 0.95 mg/kg in the high dose group.

Table 97 Residues in eggs following 28 days oral administration to laying hens

Day	Very Low Dose (0.26 mg/kg feed)		Day	Low Dose (2.51 mg/kg feed)		Day	Mid Dose (7.47 mg/kg feed)		Day	High Dose (25.83 mg/kg feed)	
	Flonicamid (mg/kg)	TFNA-AM (mg/kg)		Flonicamid (mg/kg)	TFNA-AM (mg/kg)		Flonicamid (mg/kg)	TFNA-AM (mg/kg)		Flonicamid (mg/kg)	TFNA-AM (mg/kg)
1	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	1	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	1	< 0.01 (< 0.01, < 0.01, < 0.01)	0.014 (0.022, < 0.01, < 0.01)	1	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)
2	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	2	< 0.01 (< 0.01, < 0.01, < 0.01)	0.034 (0.038, 0.028, 0.038)	2	0.013 (< 0.01, 0.013, 0.013)	0.136 (0.165, 0.124, 0.119)	2	0.052 (0.063, 0.041, 0.051)	0.450 (0.429, 0.430, 0.492)
3	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	3	< 0.01 (< 0.01, < 0.01, < 0.01)	0.053 (0.054, 0.044, 0.061)	3	0.014 (0.010, 0.016, 0.014)	0.190 (0.220, 0.180, 0.169)	3	0.056 (0.064, 0.052, 0.055)	0.691 (0.746, 0.564, 0.764)
4	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	4	< 0.01 (< 0.01, < 0.01, < 0.01)	0.083 (0.075, 0.088, 0.087)	4	0.017 (0.012, 0.024, 0.014)	0.260 (0.274, 0.295, 0.210)	4	0.067 (0.071, 0.076, 0.055)	0.837 (0.843, 0.776, 0.893)
5	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	5	< 0.01 (< 0.01, < 0.01, < 0.01)	0.078 (0.071, 0.077, 0.086)	5	0.014 (< 0.01, 0.014, 0.014)	0.263 (0.281, 0.268, 0.240)	5	0.056 (0.062, 0.064, 0.044)	0.895 (0.950, 0.791, 0.945)
6	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	6	< 0.01 (< 0.01, < 0.01, < 0.01)	0.073 (0.070, 0.058, 0.092)	6	0.012 (0.011, 0.014, 0.011)	0.250 (0.254, 0.286, 0.211)	6	0.046 (0.050, 0.047, 0.043)	1.007 (1.052, 0.968, 1.001)
7	< 0.01 (< 0.01, < 0.01, < 0.01)	0.010 (0.011, < 0.01, < 0.01)	7	< 0.01 (< 0.01, < 0.01, < 0.01)	0.079 (0.074, 0.065, 0.099)	7	0.019 (0.018, 0.019, 0.019)	0.271 (0296, 0.309, 0.208)	7	0.058 (0.064, 0.057, 0.054)	0.874 (0.773, 0.982, 0.867)
8	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	8	< 0.01 (< 0.01, < 0.01, < 0.01)	0.079 (0.070, 0.062, 0.106)	8	0.016 (0.015, 0.015, 0.016)	0.244 (0.252, 0.258, 0.223)	8	0.068 (0.072, 0.058, 0.074)	0.820 (0.907, 0.718, 0.836)
10	< 0.01 (< 0.01, < 0.01, < 0.01)	0.010 (0.011, < 0.01, < 0.01)	9	< 0.01 (< 0.01, < 0.01, < 0.01)	0.079 (0.080, 0.063, 0.093)	10	0.014 (0.014, 0.014, 0.013)	0.254 (0.306, 0.264, 0.193)	9	0.062 (0.061, 0.052, 0.073)	0.963 (1.110, 0.866, 0.912)
14	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	13	< 0.01 (< 0.01, < 0.01, < 0.01)	0.079 (0.073, 0.059, 0.102)	14	0.012 (0.012, 0.015, 0.011)	0.246 (0.266, 0.296, 0.176)	13	0.048 (0.054, 0.054, 0.037)	0.985 (1.010, 0.831, 1.114)
17	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	16	< 0.01 (< 0.01, < 0.01, < 0.01)	0.084 (0.073, 0.059, 0.102)	17	0.017 (0.012, 0.015, 0.011)	0.311 (0.340, 0.357, 0.236)	16	0.075 (0.054, 0.054, 0.037)	0.865 (1.010, 0.831, 1.114)
21	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	20	< 0.01 (< 0.01, < 0.01, < 0.01)	0.091 (0.067, 0.087, 0.118)	21	0.013 (0.012, 0.015, 0.012)	0.295 (0.312, 0.370, 0.202)	20	0.050 (0.048, 0.051, 0.051)	1.023 (0.956, 0.935, 1.177)
24	< 0.01 (< 0.01, < 0.01, < 0.01)	0.0115 (0.014, 0.010, 0.010)	23	< 0.01 (< 0.01, < 0.01, < 0.01)	0.087 (0.096, 0.076, 0.088)	24	0.014 (< 0.01, 0.015, 0.012)	0.226 (0.196, 0.276, 0.206)	23	0.053 (0.053, 0.062, 0.045)	1.041 (1.071, 0.961, 1.090)
27	< 0.01 (< 0.01, < 0.01,	< 0.01 (< 0.01, < 0.01,	26	< 0.01 (< 0.01, < 0.01,	0.098 (0.124, 0.082,	27	0.013 (0.011, 0.016,	0.310 (0.330, 0.349,	26	0.052 (0.058, 0.054,	1.119 (1.076, 1.068,

Day	Very Low Dose (0.26 mg/kg feed)		Day	Low Dose (2.51 mg/kg feed)		Day	Mid Dose (7.47 mg/kg feed)		Day	High Dose (25.83 mg/kg feed)	
	Flonicamid (mg/kg)	TFNA-AM (mg/kg)		Flonicamid (mg/kg)	TFNA-AM (mg/kg)		Flonicamid (mg/kg)	TFNA-AM (mg/kg)		Flonicamid (mg/kg)	TFNA-AM (mg/kg)
	< 0.01)	< 0.01)		< 0.01)	0.089)		0.012)	0.252)		0.044)	1.214)
28	< 0.01 (< 0.01, < 0.01, < 0.01)	0.0083 (< 0.01, < 0.01, < 0.01)	28	< 0.01 (< 0.01, < 0.01, < 0.01)	0.080 (0.078, 0.064, 0.099)	28	0.017 (0.018, 0.018, 0.016)	0.321 (0.333, 0.365, 0.265)	28	0.074 (0.093, 0.081, 0.048)	0.993 (1.067, 0.891, 1.020)

No quantifiable residues (< LOQ) of flonicamid, TFNA and TFNG were found in muscle in any treatment group. No quantifiable residues (< LOQ) of TFNA-AM was measurable in muscle at the very low dose group, but there appeared to be a dose response relationship at all other dose levels; 0.049 mg/kg in the low dose group, 0.168 mg/kg in the mid dose group and 0.654 mg/kg in the high dose group. OH-TFNA-AM was measurable only at the high dose level (0.014 mg/kg).

In liver and fat, no quantifiable residues (< LOQ) of flonicamid, TFNA, OH-TFNA-AM and TFNG were found at any dosing level. For liver, TFNA-AM residues increased from < 0.01 mg/kg (very low) to 0.054 mg/kg (low) to 0.166 mg/kg (mid) and 0.706 mg/kg (high) while for fat, TFNA-AM residues increased from 0.01 mg/kg (very low) to 0.022 mg/kg (low) to 0.062 mg/kg (mid) and 0.286 mg/kg (high).

Table 98 Residues in muscle, liver and fat following 28 days oral administration to laying hens

Dose Level (mg/kg feed)	Muscle		Liver	Fat		
	OH-TFNA-AM	TFNA-AM	TFNA-AM	OH-TFNA-AM	TFNA-AM	TFNA
0.259	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01, < 0.01)
2.51	< 0.01 (< 0.01, < 0.01, < 0.01)	0.049 (0.050, 0.035, 0.062)	0.054 (0.057, 0.040, 0.065)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.022 (0.018, 0.016, 0.031)	< 0.01 (< 0.01, < 0.01, < 0.01)
7.47	< 0.01 (< 0.01, < 0.01, < 0.01)	0.168 (0.169, 0.187, 0.149)	0.166 (0.178, 0.187, 0.134)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.062 (0.061, 0.080, 0.046)	< 0.01 (< 0.01, < 0.01, < 0.01)
29.83	0.014 (0.014, 0.013, 0.016)	0.654 (0.654, 0.590, 0.718)	0.706 (0.786, 0.606, 0.671)	< 0.01 (< 0.01, < 0.01, < 0.01)	0.286 (0.353, 0.265, 0.242)	< 0.01 (< 0.01, < 0.01, < 0.01)

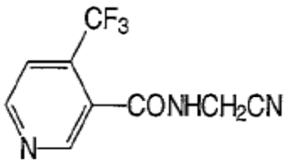
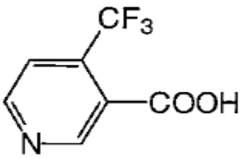
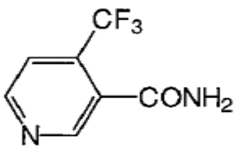
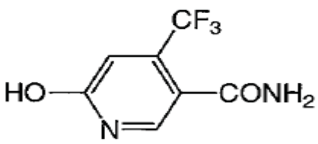
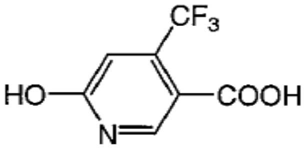
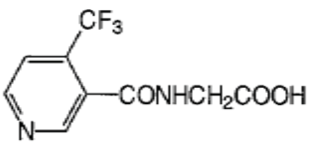
APPRAISAL

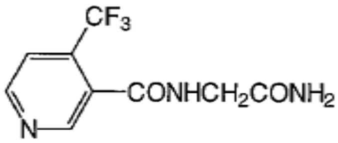
Flonicamid is a new insecticide for control of aphids and other sucking insects. It belongs to a new class of chemistry known as pyridinecarboxamide. At the Forty-sixth Session of the CCPR, flonicamid was scheduled for evaluation, for both toxicology and residues, as a new compound by the 2015 JMPR.

The Meeting received information on the metabolism of flonicamid in peaches, bell peppers, potatoes, wheat, lactating goats, laying hens and rotational crops, environmental fate, methods of residue analysis, freezer storage stability, GAP, supervised residue trials on various fruits, vegetables, tree nuts, oil seeds, dried hops, mint and tea, processing studies as well as livestock feeding studies.

In this document, the code names and chemical structures of the metabolites were as follows:

Flonicamid is *N*-cyanomethyl-4-(trifluoromethyl)nicotinamide (IUPAC).

Code Name	Structure	Chemical Name
Flonicamid IKI-220		N-cyanomethyl-4-(trifluoromethyl)nicotinamide
TFNA		4-trifluoromethylnicotinic acid
TFNA-AM		4-trifluoromethylnicotinamide
OH-TFNA-AM		6-hydroxy-4-trifluoromethylnicotinamide
TFNA-OH		6-hydroxy-4-trifluoromethylnicotinic acid
TFNG		N-(4-trifluoromethylnicotinoyl)glycine

TFNG-AM		N-(4-trifluoromethylnicotinoyl)glycinamide

Environmental fate in soil

The FAO Manual (FAO, 2009) explains the data requirements for studies of environmental fate. The focus should be on those aspects that are most relevant to MRL setting. For flonicamid, supervised residue trials were received for foliar spray on permanent crops and on annual crops. Therefore, according to the FAO manual, only studies on aerobic degradation, photolysis and rotational crops (confined, field) were evaluated.

Degradation

The route of degradation of [^{14}C]flonicamid in soil under aerobic conditions was investigated in a biologically active loamy sand soil collected from Madison, Ohio, USA and stored in a greenhouse. Flonicamid rapidly declined from 99.3% of the applied radioactivity (AR) at Day 0 to 2.3% by Day 30, resulting in a DT_{50} of 1 day and a DT_{90} of 3.4 days. TFNA and TFNA-OH were major components of the residue with TFNG, TFNG-AM and TFNA-AM all identified as minor metabolites.

The rate of aerobic degradation of [^{14}C]flonicamid, radiolabelled at the 3 position of the pyridine was investigated in three biologically active soils (sandy loam and sand at 10 °C and/or 20 °C)

For the soils incubated at 20 °C the DT_{50} and DT_{90} values for flonicamid ranged from 0.7 to 1.8 days and 2.3 to 6.0 days, respectively. For the soil incubated at 10 °C, the DT_{50} and DT_{90} values for flonicamid were 2.4 days and 7.9 days, respectively. TFNA, TFNA-OH and TFNG-AM were the major degradates in all soils over the course of the study. Minor degradates TFNG and TFNA-AM were detected at all sampling points over the course of the study. All of the degradates were metabolised and mineralised to carbon dioxide and immobilised as soil-bound residue.

Photolysis

The photochemical degradation of [pyridyl-^{14}C]flonicamid was investigated in a loamy sand under laboratory conditions.

[^{14}C]Flonicamid decreased to 60% of the applied radioactivity (AR) after 15 days of continuous illumination, resulting in a DT_{50} of 22 days. Concurrently, the major metabolite TFNG-AM was detected in Day 1 sample extracts and increased by Day 15. TFNA-AM and TFNG were also detected as minor metabolites in the illuminated soils, reaching maximum concentrations of 5% AR (Day 11 and Day 15) and 2% AR (Day 15), respectively.

In summary, based on the results of the environmental fate studies, flonicamid as well as its metabolites are likely to readily degrade and not persist in aerobic soil environments.

Plant metabolism

The metabolism of flonicamid was studied in peaches, bell peppers, potatoes and wheat.

Peach

Flonicamid, radiolabelled at the 3 position of the pyridine ring and formulated as a wettable granule, was applied twice to peach trees grown outdoor, with a 14-day re-treatment interval, at rates of 100 g ai/ha (low rate) or 500 g ai/ha (high rate) per application. Mature fruits and leaves were harvested 21 days after the last treatment (DALA). Overall total radioactive residues (TRRs) in fruits at the low rate and the high rate were 0.10 mg eq/kg and 0.32 mg eq/kg, respectively, while in the leaves, TRRs followed the same trend, where residues were lower at the lower application rate (6.2 mg eq/kg) compared to those at the higher treatment rate (24 mg eq/kg).

The peaches were subjected to a surface wash using deionised water which removed very little radioactivity (≤ 15% TRRs), evidence of limited penetration. The majority of the TRRs were partitioned into the juice fraction (64–73% of the TRR) and to a lesser extent into the pulp (21% TRRs). While juice was not further extracted with organic solvents, extraction of the pulp with acetonitrile:water:phosphoric acid recovered 92% TRR. At both treatment rates, flonicamid (30–60% TRRs) and TFNA (17–49% TRRs) were the predominant residues in juice and pulp. All other metabolites, TFNG, TFNG-AM and TFNA- AM were ≤ 6% TRRs.

Bell pepper

A single application of flonicamid, radiolabelled at the 3 position of the pyridine ring, formulated as a 50% wettable granule formulation, was made to greenhouse grown bell pepper plants at 100 g ai/ha. Fruits and leaves were harvested 7 days and 14 days after treatment (DAT).

The TRRs in fruits decreased insignificantly from 0.17 mg eq/kg (7 DAT) to 0.11 mg eq/kg (14 DAT) while TRRs in leaves decreased from 2.2 mg eq/kg, when harvested 7 days after treatment to 1.4 mg eq/kg at 14 DAT.

The %TRR in the methanol:water surface wash for both leaves and fruits decreased as the corresponding extracted TRRs (61–81% TRRs) and those in the post-extraction solids (PES) increased with increasing DAT. This trend demonstrated the penetration of the radioactivity from the surface into the leaves and fruits.

Flonicamid and TFNG were the predominant residues in leaves (47–74% TRRs and 12–28% TRRs, respectively) while only flonicamid was the predominant residue in fruits (77–91% TRRs) at both harvest intervals. All identified metabolites (TFNA, TFNA-AM and TFNG-AM) were either not detected or were ≤ 12% TRRs.

Potato

Potato plants maintained outdoor were treated, either at the lower rate of 100 g ai/ha or the higher rate of 500 g ai/ha, with flonicamid radiolabelled at the 3 position of the pyridine ring and formulated as a 50% wettable powder. Both treatments were repeated at a two-week interval and potato tubers and foliage were harvested 14 days after the last application.

Overall TRRs in tubers at the low rate and the high rate were 0.11 mg eq/kg and 0.20 mg eq/kg, respectively, while those in mature foliage were higher than those in tubers; 1.5 mg eq/kg at the low rate and 7.7 mg eq/kg at the high rate. Considering the applications were made to the foliage of the potato plants, the presence of measurable TRRs in the tubers is evidence of translocation of the radioactivity from the foliage to the tubers. Furthermore, while the TRRs in tubers and foliage increased with increased application, the distribution of TRRs was relatively the same irrespective of the treatment rate.

Extraction of the potato tubers with ACN and ACN:water recovered up to 93% TRRs while extraction of potato foliage with ACN:water:acetic acid recovered up to 90% TRRs. Analysis of each of the extracted fractions of potato tubers and foliage from the low and high rate demonstrated that the major components of the residue, TFNA and TFNG, accounted for a significant portion of the TRRs. Moreover, TFNA accounted for 34% TRRs in tubers and 12–17% TRRs in foliage at both rates while TFNG accounted for 25–39% TRRs in tubers and 28–36% TRRs in leaves at both rates. The parent, flonicamid, accounted for 6–19% TRRs in potato tubers and 10–25% TRRs in foliage. Each of the other identified metabolites (TFNA conjugate, TFNG-AM, TFNA-AM, PM-1a, PM-1b and PM-3a) accounted for < 10% TRRs in tubers and in foliage. Overall, the general metabolic profile in foliage was similar to that in tubers.

Spring wheat

Spring wheat plants grown outdoor were treated with flonicamid, radiolabelled at the 3 position of the pyridine ring and formulated as a wettable powder, at a single application rate of 100 g ai/ha or 2 applications at 100 g ai/ha with a re-treatment interval of 7 days. Forage and hay were harvested 14 days and 42 days, respectively, after the single application. Approximately 95 days after the second treatment (200 g ai/ha/season), mature plants were harvested and separated into straw, chaff and grain.

Overall residues were lower in the wheat grain sample (2.6 mg eq/kg) than the straw (5.6 mg eq/kg) or chaff (6.6 mg eq/kg). The TRRs in the chaff were higher compared to straw potentially because of tissue size differences (higher surface area to weight ratio) assuming a uniform application. Further to this, considering the timing of application of the test material and the measurable TRRs in grain, chaff and straw at maturity, there appears to have been translocation of the radioactivity from the site of application to the mature plant parts.

Only forage and hay were analysed to elucidate the nature of the flonicamid residues. Extraction of these matrices with ACN:water:acetic acid recovered 96% TRRs. The analysis of forage and hay samples demonstrated that the nature and distribution of metabolites were similar in both matrices. The parent compound, flonicamid, and the TFNG metabolite represented the majority of the TRRs in both the forage (flonicamid: 43% TRRs; TFNG: 33% TRRs) and hay (flonicamid: 22%TRRs; TFNG: 53% TRRs). Metabolites TFNG-AM, TFNA and TFNA-AM were present at ≤ 13% TRRs.

In a second spring wheat metabolism study, plants grown outdoor were treated with flonicamid, radiolabelled at the 3 position of the pyridine ring and formulated as a wettable granule, at rates equivalent to 100 g ai/ha or 500 g ai/ha. The wheat plants were harvested 21 DAT and separated into straw (leaves and stem), chaff and grain (with hulls attached).

The TRRs in wheat straw, chaff and grain increased with increased application rate with the highest TRRs observed in chaff, followed by straw and grain, irrespective of the application rate. The distribution of TRRs was relatively the same irrespective of the treatment rate.

Similar to the first wheat metabolism study, extraction with ACN:water:acetic acid recovered 80–94%TRRs with flonicamid (24–50%TRRs) and TFNG (17–44% TRRs) representing the predominant residues at both treatment rates. All identified metabolites (TFNA, TFNG-AM, TFNA-AM and N-oxide TFNA AM) were either not detected or were each ≤ 10% TRR.

In summary, the Meeting determined that the degree of metabolism in all crops tested, following foliar application, was qualitatively similar, with the parent compound as the predominant residue. The major metabolic pathway of flonicamid in plants involved hydrolysis of the cyano group and the amide groups.

Rotational crops

In the confined rotational crop study, flonicamid, radiolabelled at the 3 position of the pyridine ring and formulated as a wettable granule was applied twice to loamy sand soil at a rate equivalent to 100 g ai/ha at an interval of two weeks. After the appropriate plant-back intervals (PBIs) of 30, 120 or 360 days, the rotational crops, representative of the root vegetable (carrot), small grain (wheat), and leafy vegetable (lettuce) crop groups, were planted.

TRRs in all raw agricultural commodities (RACs) declined with prolonged PBIs such that, at the 120-day PBI, no further characterization/identification of the TRRs was performed for immature and mature lettuce and mature carrot roots due to the low total radioactivity. Further to this, at the 360-day PBI, none of the TRRs from any of the crop parts were further subjected to characterization/identification as these were also too low.

Analysis of the harvested crop samples demonstrated very little uptake of ^{14}C-residues. Of the radioactivity taken up by plants, only limited amounts of flonicamid were detected (≤ 13% TRRs). TFNG and TFNG-AM were identified at > 10% TRRs in most RACs. In addition to

TFNG, other identified metabolites accounting for > 10% TRRs in wheat matrices and mature carrot root included TFNA-AM and TFNA-OH.

Conversely, in the field accumulation study, no quantifiable residues of flonicamid or its metabolites TFNG, TFNA, and TFNA-AM were detected in wheat (forage, straw and grain) and turnip (tops and roots) planted at either 30 or 60 days after the last application of flonicamid to the primary crop, cotton.

Based on the findings of the field crop rotation studies, the Meeting concluded that the uptake of quantifiable residues of flonicamid or its associated metabolites in secondary crops is unlikely.

Animal metabolism

Metabolism studies in rats reviewed by the 2015 JMPR and conducted using [^{14}C]flonicamid labelled at the 3-nicotinamide position, indicated that flonicamid was rapidly absorbed and quickly excreted. The majority of administered radioactivity was excreted in the urine and within the first 24 hours. There was no evidence of bioaccumulation following repeat doses. Distribution into the tissues was extensive with levels similar to blood concentrations; however, slightly higher concentrations were seen in the liver, kidneys, adrenals, thyroid and ovaries following single or repeat dosing and in the lungs following repeat dosing in males.

The main urinary residue was unchanged parent, followed by TFNA-AM, which was also the predominant metabolite in the faeces and bile. Other metabolites were TFNA in the faeces of low-dose animals, TFNA-AM N-oxide conjugate in the high-dose animals, TFNG-AM in the bile of high-dose animals and TFNG and TFNA-AM in the liver.

Metabolism studies were conducted in lactating goats where they were dosed orally once daily for 5 consecutive days with 3-pyridine-^{14}C-labelled flonicamid at a dose level equivalent to 10 ppm in feed. The major route of elimination of the radioactivity was via the urine which accounted for 49% of the administered dose (AD), while faeces accounted for up to 21% of the AD and milk accounted for 1% of the AD. Overall, the tissue burden was low, accounting for < 10% of the AD. The TRRs were highest in liver (1.2 mg eq/kg) followed by kidney (0.70 mg eq/kg), muscle (0.30–0.40 mg eq/kg) and fat (0.05–0.14 mg eq/kg).

Extraction of milk, using ethanol and ethanol:water recovered 97% TRRs and extraction of tissues and organs using ACN and ACN:water containing 1% acetic acid recovered greater than 42% TRRs. Flonicamid was rapidly metabolised in lactating goats, representing less than 6% TRRs in tissues and organs. TFNA-AM was the major component of the residue in organs (29% TRRs in liver, 31–41% TRRs in kidney), tissues (74% TRRs in fat, 42–50% TRRs in muscle) and milk (97% TRRs). The minor metabolites TFNA and 6-OH-TFNA-AM each accounted for ≤ 7% TRRs in liver, kidney, muscle and milk.

Leghorn laying hens were dosed orally once daily for 5 consecutive days with 3-pyridine-^{14}C-labelled flonicamid at a dose level equivalent to 10 ppm in feed. Approximately 91% of AD including 6% of AD from the gastrointestinal tract and its contents was recovered. Most of the AD (72%) was excreta-related. TRRs in egg white and egg yolk accounted for about 2.4% of AD (1.8% AD in egg white plus 0.6% AD in yolk). The tissue burden was low (< 6% of the AD) with highest concentrations of ^{14}C-residues found in kidney (1.4 mg eq/kg) followed by liver (1.2 mg eq/kg), muscle (evenly distributed between breast and thigh muscle; 1.0 mg eq/kg each), skin (0.70 mg eq/kg) and fat (0.15 mg eq/kg).

Extraction of eggs, tissues and organs with ACN and ACN:water containing 1% acetic acid recovered more than 81% TRR. Flonicamid accounted for only a very small percentage of the TRRs in eggs (2–4% TRRs), tissues (< 1% TRRs) and organs (< 0.5% TRRs). TFNA-AM was the predominant component of the residue in egg whites and egg yolks (≤ 96%TRRs), liver (93%TRRs), kidney (76%TRRs) and tissues (97%TRRs in both breast muscle and thigh muscle, 96%TRRs in skin and 95%TRRs in fat). Other metabolites identified in organs and tissues were

OH-TFNA-AM and TFNG-AM, however, neither of these accounted for greater than 5% of TRR.

The Meeting concluded that, in all species investigated, the total administered radioactivity was quickly and almost completely eliminated in excreta. The metabolic profiles differed quantitatively between the species, but qualitatively there were no major differences. The routes and products of metabolism in animals were consistent across the studies resulting from the hydrolysis of the cyano function to the amide function as well as ring hydroxylation. Moreover, TFNA-AM was the major component of the residue in all tissues, organs, milk and eggs of livestock animals.

While the overall metabolism in plants, livestock and rats is similar, the metabolism of flonicamid in animals is more extensive with hydrolysis of flonicamid to the major amide metabolite TFNA-AM.

Methods of analysis

The Meeting received descriptions and validation data for analytical methods for residues of flonicamid and its relevant metabolites TFNA-AM, TFNA and TFNG in plant commodities and for flonicamid, TFNA-AM, TFNA, TFNG and OH-TFNA-AM in animal commodities. Residue analytical methods rely on LC/MS-MS. Typical limits of quantitation (LOQs) achieved for plant commodities fell in the range of 0.01–0.02 mg/kg for each analyte. The LOQs for milk and animal products (liver, kidney, muscle and eggs) were 0.01 mg/kg for each analyte. Methods were successfully subjected to independent laboratory validation.

The acid version (addition of 1% formic acid to the acetonitrile extraction solvent) of the QuEChERS multi residue LC-MS/MS method was used for flonicamid, TFNA, TFNG and TFNA-AM in plant matrices with LOQs of 0.01 mg/kg for each analyte.

The Meeting determined that suitable methods are available for the analysis of flonicamid and its relevant metabolites in plant and animal commodities.

Stability of residues in stored analytical samples

The Meeting received storage stability studies under freezer conditions at –17 °C for flonicamid and its relevant metabolites TFNA-AM, TFNA and TFNG for the duration of the storage of 18 to 23 months in a wide range of raw and processed crop matrices, including high-water, high-starch and high-oil crops. The Meeting concluded that residues of flonicamid, TFNA-AM, TFNA and TFNG are stable for at least 18 months. Freezer storage stability studies were also conducted concurrently with several of the crop field trials, demonstrating similar results.

All milk samples from the feeding studies were frozen at –20 ºC and analysed within 30 days after sampling. Therefore, storage stability data are not necessary. In contrast, all tissue samples were analysed within 12 months of collection. Freezer storage stability studies, conducted concurrently with the feeding studies, demonstrated that flonicamid, TFNA, TFNA-AM, OH-TFNA-AM and TFNG were stable for 374 days in all tissues except fat. For fat, flonicamid and its metabolites were demonstrated to be stable for 315 days.

Definition of the residue

In primary crops, the parent compound represented the majority of the residue accounting for up to 61% TRRs in peach fruits, 91%TRRs in bell pepper fruits, 19% TRRs in potato and up to 50% TRRs in wheat forage, hay, straw, chaff and grain. Metabolites TFNA and TFNG were identified as predominant metabolites (> 10%TRRs) in all crop commodities. In the crop field trials, residues of TFNA and TFNG were measurable in all crops, the magnitude of which was crop-dependent. However, both the TFNA and TFNG were seen in the rat metabolism study and considered to be up to 10-fold less toxic than the parent flonicamid based on toxicity studies reviewed by the 2015 WHO.

In the field accumulation study no measurable residues of parent or any of its associated metabolites were observed in secondary crops.

In light of the above, the Meeting decided to define the residue for enforcement/monitoring and for risk assessment for plant commodities as parent only.

In the farm animal metabolism studies, the parent, flonicamid, was rapidly degraded in ruminants and poultry, accounting for ≤ 6% TRRs in all tissues, milk and eggs. Conversely, the metabolite TFNA-AM accounted for the majority of the radioactivity in goat tissues (29–74% TRRs) and milk (92–97%TRRs) and laying hen tissues (76–97% TRRs) and eggs (ca. 95%TRRs).

Similar findings were observed in the livestock feeding studies whereby flonicamid was present at very low levels in all animal commodities with the metabolite TFNA-AM representing the majority of the residues in tissues, milk and eggs. Therefore, TFNA-AM will be included in the residue definition for enforcement as a marker compound. Since the method of analysis is capable of analysing both flonicamid and TFNA-AM, the Meeting agreed to define the residue for enforcement/monitoring as flonicamid and TFNA-AM.

The log K_{ow} for flonicamid is 0.3. In the metabolism studies there was no evidence of the parent compound and TFNA-AM partitioning into fatty matrices (fat, milk and egg yolks) as the total residues were present at comparable concentrations in all livestock matrices. In the dairy cattle and poultry feeding studies, there was no evidence of the total residues of flonicamid and TFNA-AM sequestering into milk, eggs or fat. Therefore, the Meeting did not consider the residue fat soluble.

As TFNA-AM was the major component of the residue in all animal matrices in both the metabolism and feeding studies, the Meeting decided to define the residue for dietary risk assessment for animal commodities as parent and TFNA-AM.

Based on the above, the Meeting recommended that the residue definition for compliance with MRLs and estimation of dietary intake should be as follows:

Definition of the residue for compliance with MRL and estimation of dietary intake for plant commodities: *Flonicamid*

Definition of the residue for compliance with MRL and estimation of dietary intake for animal commodities: Flonicamid and the metabolite TFNA-AM, expressed as parent.

The residue is not fat soluble.

Results of supervised residue trials on crops

Pome fruits

Results from supervised field trials on apples and pears conducted in the US were provided to the Meeting, including apple and pear data from Australia.

A total of 16 independent supervised trials were conducted in the US on apples (12) and pears (4). The GAP in the US for pome fruits allows three applications at a maximum rate of 0.1 kg ai/ha with a PHI of 21 days.

Flonicamid residues from 12 apple trials matching the US GAP were: 0.02, 0.04 (3), 0.05 (4), 0.06, 0.07, 0.10 and 0.11 mg/kg.

Flonicamid residues from four pear trials matching the US GAP were: 0.01 (3) and 0.02 mg/kg.

A total of seven independent supervised trials were also conducted on apples in Australia according to the Australian GAP which allows three applications at a maximum rate of 0.01 kg ai/hL with a PHI of 21 days. Nine supervised trials were conducted on pears in Australia, however, in the absence of an Australian GAP, these trials were not considered.

Flonicamid residues from seven apple trials matching the Australia GAP were 0.09, 0.12 (2), 0.13, 0.15, 0.22 and 0.24 mg/kg.

The Meeting noted that in the US a group GAP for pome fruit exists and decided to explore the possibility of setting a group maximum residue level. As the supervised trials on apples conducted in Australia in accordance with the Australian GAP lead to the higher residues, the Meeting recommended that the group maximum residue level be based on the dataset from Australia.

Based on the Australian residue data for apples, the Meeting estimated a maximum residue level for pome fruits of 0.8 mg/kg and an STMR of 0.13 mg/kg.

Stone fruits

Results from supervised field trials on peaches, cherries and plums conducted in the US were provided to the Meeting.

A total of 19 independent supervised trials were conducted in the US on peaches (8), cherries (6) and plums (5) according to the US GAP on stone fruits which allows three applications at a maximum rate of 0.1 kg ai/ha with a 14-day PHI.

Residues of flonicamid from eight peach trials matching the US GAP for stone fruits were: 0.09 (2), 0.10, 0.13, 0.15, 0.22 (2) and 0.42 mg/kg.

Residues of flonicamid from six cherry trials matching the US GAP for stone fruits were: 0.26, 0.27 (2), 0.28 (2) and 0.36 mg/kg.

Residues of flonicamid from five plum trials matching the GAP for stone fruits were: 0.01, 0.02, 0.03 and 0.04 (2) mg/kg.

The Meeting noted that in the US a group GAP for stone fruits exists and decided to explore the possibility of setting a group maximum residue level. Since median residues among the representative commodities were not within a 5-fold range (0.14 mg/kg vs. 0.28 mg/kg vs. 0.03 mg/kg), the Meeting decided to estimate maximum residue levels for each subgroup based on the individual dataset for each representative commodity.

The Meeting estimated a maximum residue level of 0.9 mg/kg and an STMR of 0.28 mg/kg for cherries subgroup.

The Meeting estimated a maximum residue level of 0.7 mg/kg and an STMR of 0.14 mg/kg for peaches subgroup.

The Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.03 mg/kg for plums subgroup.

Strawberries

Results from supervised field trials on strawberries conducted in the US were provided to the Meeting.

A total of eight independent supervised trials were conducted in the US on strawberries according to the US GAP for low growing berries, which allows three applications at a maximum rate of 0.1 kg ai/ha with a 0-day PHI.

Residues of flonicamid matching the US GAP were: 0.13, 0.19, 0.27, 0.33, 0.41, 0.47, 0.54 and 0.59 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg and an STMR of 0.37 mg/kg for low growing berries.

Brassica (Cole or cabbage) vegetables, Head cabbages, Flowerhead brassicas

Results from supervised field trials on cabbage and broccoli conducted in the US were provided to the Meeting.

A total of 12 independent supervised trials were conducted in the US on broccoli (6) and cabbage (6) according to the US GAP on Brassica (Cole) Leafy Vegetables which allows three applications at a maximum rate of 0.1 kg ai/ha with a 0-day PHI.

Residues of flonicamid from six broccoli trials matching the US GAP for Brassica (Cole) leafy vegetables were: 0.250, 0.428, 0.432, 0.462, 0.499 and 0.553 mg/kg.

Residues of flonicamid from six trials on cabbage (with wrapper leaves) matching the US GAP for Brassica (Cole) Leafy Vegetables were: < 0.025, 0.025, 0.062, 0.205, 0.288 and 1.262 mg/kg.

Residues of flonicamid from six trials on cabbage (without wrapper leaves) matching the US GAP for Brassica (Cole) Leafy Vegetables were: < 0.025 (6) mg/kg.

The Meeting noted that in the US a group GAP for Brassica (Cole) leafy vegetables exists and decided to explore the possibility of setting a group maximum residue level. Since median residues among the representative crops were within a 5-fold range (0.45 mg/kg vs. 0.134 mg/kg) and the Mann-Whitney test indicated that the residues were not statistically different, the Meeting decided to estimate a group maximum residue level based on the following combined residues: < 0.025(7), 0.025, 0.062, 0.205, 0.288 and 1.262 mg/kg.

The Meeting estimated a maximum residue level of 2.0 mg/kg and an STMR of 0.358 mg/kg for Brassica (Cole or cabbage) vegetables, head cabbages and flowerhead Brassicas.

The Meeting estimated an STMR of 0.02 mg/kg for cabbage (without wrapper leaves).

Fruiting vegetables, Cucurbits

Supervised field trials on field- and greenhouse-grown melons conducted in Southern EU and on field-grown pumpkins conducted in Hungary were provided to the Meeting. However, only four trials on melons and four trials on pumpkins matched the critical GAP of Slovenia which allows three foliar spray applications of a WG formulation at 0.05 kg ai/ha with a re-treatment interval of 7 days and a PHI of 1 day. Therefore, in the absence of a sufficient number of trials matching the Slovenia critical GAP, these trials were not considered further.

A total of 17 independent supervised trials, conducted in the US on cucumber (6), melon (6) and summer squash (5) according to the US GAP on cucurbit vegetables, which allows three applications at a maximum rate of 0.1 kg ai/ha with a 0-day PHI, were provided to the Meeting. In addition, the Meeting received four greenhouse cucumber trials conducted in Canada and the US according to the US critical GAP which allows two foliar spray or soil applications at a maximum rate 0.15 kg ai/ha with a re-treatment interval of 6–7 days and a 0-day PHI.

Residues of flonicamid from six field cucumber trials matching the US GAP for cucurbit vegetables were: 0.04, 0.06 (3), 0.07 and 0.12 mg/kg.

Residues of flonicamid from six melon trials matching the US GAP for cucurbit vegetables were: 0.020, 0.03, 0.04, 0.05, 0.06 and 0.09 mg/kg.

Residues of flonicamid from five summer squash trials matching the US GAP for cucurbit vegetables were: 0.01, 0.03 (3) and 0.04 mg/kg.

Residues of flonicamid from the greenhouse cucumber trials matching the US GAP for the foliar spray application were: 0.05, 0.06, 0.14 and 0.54 mg/kg.

Residues of flonicamid from the greenhouse cucumber trials where the growth media was treated were: 0.09, 0.13, 0.16 and 0.20 mg/kg.

For greenhouse cucumbers, as there is an insufficient number of supervised trials conducted in accordance with the US critical GAP, the Meeting did not consider these trials further.

In addition to the US trials, the Meeting received 10 independent supervised field trials conducted in Australia on cucumbers (2), melons (5) and summer squash (3) according to the

Australian GAP on cucurbit vegetables which allows three applications at a maximum rate of 0.1 kg ai/ha with a 1-day PHI.

Residues of flonicamid from two field cucumber trials matching the Australian GAP for Cucurbit Vegetables were: 0.03 (2) mg/kg.

Residues of flonicamid from five melon trials matching the Australian GAP for Cucurbit Vegetables were: 0.03, 0.05 (2), 0.09 and 0.17 mg/kg.

Residues of flonicamid from three summer squash trials matching the Australian GAP for Cucurbit Vegetables were: 0.01, 0.04 and 0.08 mg/kg.

Since the use of flonicamid on the cucurbits crop group is registered in Australia, the residue decline trials demonstrated limited dissipation of flonicamid residues with increasing PHI and that there are an insufficient number of Australian trials at the critical GAP, the Meeting compared the US field trials against the Australian GAP and combined them as follows:

Residues of flonicamid in field cucumbers from eight trials were: 0.03 (2), 0.04, 0.06 (3), 0.07 and 0.12 mg/kg.

Residues of flonicamid in melons from 11 trials were: 0.02, 0.03(2), 0.04 (2), 0.05 (2), 0.06, 0.09 (2) and 0.17 mg/kg.

Residues of flonicamid in summer squash from eight trials were: 0.01 (2), 0.03 (3), 0.04 (2) and 0.08 mg/kg.

The median residues among the representative crops were within a 5-fold range (0.06 mg/kg vs. 0.05 vs 0.03 mg/kg) and the Kruskall-Wallis test indicated that the residues were not statistically different, therefore, the Meeting decided to combine the dataset as follows: 0.01 (2), 0.02, 0.03 (7), 0.04 (5), 0.05 (2) 0.06 (4), 0.07, 0.08, 0.09 (2), 0.12 and 0.17 mg/kg.

The Meeting estimated a maximum residue level for fruiting vegetables, cucurbits, of 0.2 mg/kg and an STMR of 0.04 mg/kg.

Fruiting vegetables, other than cucurbits

Results from supervised field trials on tomatoes, bell peppers and non-bell peppers were conducted in the US as well as supervised trials on greenhouse tomatoes conducted in Canada and the US were provided to the Meeting.

A total of 34 independent supervised trials were conducted in the US on field tomatoes (26), bell peppers (6) and non-bell peppers (2) according to the US GAP on fruiting vegetables, which allows three foliar spray applications of a WG formulation at a maximum rate of 0.1 kg ai/ha or two applications of a SG formulation at a maximum rate of 0.15 kg ai/ha. For both formulations, the crops may be harvested at a 0-day PHI.

Three additional trials were conducted in Canada and the US on greenhouse tomatoes where treatments were conducted according to the US GAP which allows two foliar spray applications at a maximum rate of 0.15 kg ai/ha with a 0-day PHI.

Only field tomato trials were conducted with both the WG and SG formulations, however, it was not clear which formulation resulted in the critical GAP:

Residues of flonicamid from 12 field tomato trials where the WG formulation was applied according to the US critical GAP for fruiting vegetables were: 0.03, 0.05, 0.06, 0.07, 0.08, 0.09 (3), 0.14, 0.15, 0.22 and 0.23 mg/kg.

Residues of flonicamid from 14 field tomato trials where the SG formulation was applied according to the US critical GAP for fruiting vegetables were: < 0.01, 0.05(2), 0.06, 0.07, 0.08, 0.10 (2), 0.11, 0.12 (2), 0.13, 0.15 and 0.19, mg/kg.

As highest residues in tomatoes were observed following treatment with the WG formulation, only these were considered when estimating the maximum residue level.

Residues of flonicamid from six bell pepper trials matching the US critical GAP for fruiting vegetables were: 0.06 (3), 0.10 and 0.11 (2) mg/kg.

Residues of flonicamid from two non-bell pepper trials matching the US critical GAP for fruiting vegetables, other than cucurbits were: 0.21 and 0.22 mg/kg.

As the GAP in the US is for the fruiting vegetables crop group, the median values from the trials conducted in the US on tomatoes, bell peppers and non-bell peppers were within 5-fold (0.09 mg/kg vs 0.08 mg/kg vs 0.21 mg/kg) and the Kruskall-Wallis test indicated that the residues from field trials were not statistically different, the Meeting decided to estimate a group maximum residue level. The residues in tomatoes, bell peppers and non-bell peppers were combined as follows: 0.03, 0.05, 0.06 (4), 0.07, 0.08, 0.09 (3), 0.10, 0.11 (2), 0.14, 0.15, 0.21, 0.22 (2) and 0.23 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg and an STMR of 0.09 mg/kg for fruiting vegetables, other than cucurbits, excluding mushrooms and sweet corn.

Leafy vegetables

Leafy vegetables (excluding Brassica leafy vegetables)

Results from supervised field trials on head lettuce, leaf lettuce, spinach and radish leaves conducted in the US were provided to the Meeting.

A total of 18 independent supervised trials were conducted in the US on head lettuce (6), leaf lettuce (6) and spinach (6) according to the US GAP on leafy vegetables which allows three applications at a maximum rate of 0.1 kg ai/ha with a 0-day PHI.

A total of five independent supervised trials were conducted in the US on radish leaves according to the US GAP on root and tuber vegetables which allows three applications at a maximum rate of 0.1 kg ai/ha with a 3-day PHI.

Residues of flonicamid from six head lettuce (with wrapper leaves) trials matching the US GAP for leafy vegetables were: 0.39, 0.43, 0.49, 0.52, 0.58 and 0.62 mg/kg.

Residues of flonicamid from six trials on leaf lettuce matching the US GAP for leafy vegetables (except Brassica) were: 1.94, 2.18, 2.52, 2.67, 2.71, 3.06 and 3.11 mg/kg.

Side-by-side trials were conducted on cos lettuce comparing the WG formulation with the SG formulation with and without surfactant. These trials were not considered further in the estimation of the maximum residue level.

Residues of flonicamid from six trials on spinach matching the US GAP for leafy vegetables were: 4.82, 4.86, 5.71, 5.73, 6.59 and 6.97 mg/kg.

Residues of flonicamid from five trials on radish leaves matching the US GAP for root and tuber vegetables were: 0.21, 3.1, 5.4, 5.7 and 8.5 mg/kg.

As the GAP in the US is established for the leafy vegetables crop group, the Meeting decided to explore the possibility of setting a group MRL. The median residues in head lettuce, leaf lettuce and spinach, which are the representative commodities for this subgroup, differed by more than 5-fold (0.51 mg/kg vs 2.67 mg/kg vs 5.72 mg/kg). In addition, as the GAP for radish leaves differs from that of the other leafy vegetables, the Meeting decided to estimate maximum residue levels for each commodity based on the individual datasets without extrapolation to the entire subgroup.

The Meeting estimated a maximum residue level of 1.5 mg/kg and an STMR of 0.51 mg/kg for head lettuce with wrapper leaves.

For leaf lettuce, the Meeting estimated a maximum residue level of 8 mg/kg and an STMR of 2.67 mg/kg

The Meeting estimated a maximum residue level of 20 mg/kg and an STMR of 5.72 mg/kg for spinach.

The Meeting estimated a maximum residue level of 20 mg/kg and an STMR of 8.50 mg/kg for radish leaves.

Brassica leafy vegetables

Results from supervised field trials on mustard greens conducted in the US were provided to the Meeting.

A total of eight independent supervised trials were conducted in the US on mustard greens according to the US GAP on Brassica (Cole) leafy vegetables which allows three applications at a maximum rate of 0.1 kg ai/ha with a 0-day PHI.

Residues of flonicamid from eight trials on mustard greens matching the US GAP for Brassica (Cole) leafy vegetables were: 2.04, 2.21, 3.96, 4.40, 4.78, 4.92, 6.87 and 8.31 mg/kg.

The Meeting estimated a maximum residue level of 15 mg/kg and an STMR of 8.31 mg/kg for the Brassica leafy vegetables subgroup.

Root and tuber vegetables

Results from supervised field trials on potatoes, carrots and radish roots conducted in the US and Australia (potatoes only) were provided to the Meeting.

A total of 23 independent supervised trials were conducted in the US on potatoes (16), carrots (2) and radishes (5) according to the critical GAP in the US which allows three applications at a maximum rate of 0.1 kg ai/ha with a 7-day PHI for potatoes and a 3-day PHI for carrots and radishes.

Residues of flonicamid from 16 potato trials matching the US GAP were: < 0.01 (15) and 0.015 mg/kg.

Residues of flonicamid from two carrot trials matching the US GAP were: 0.02 (2) mg/kg.

Residues of flonicamid from five radish trials matching the US GAP were: 0.02, 0.07, 0.10, 0.13 and 0.21 mg/kg.

The Meeting noted that in the US, group GAPs for root and tuber vegetables and tuberous and corm vegetables exist; however, as these GAPs are different for each crop group and there is an insufficient number of supervised residue trials provided for carrots, the Meeting decided to estimate individual maximum residue levels for potato and radish roots only.

For potatoes, the Meeting estimated a maximum residue level of 0.015 mg/kg and an STMR of 0.01 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg and an STMR of 0.10 mg/kg for radish roots.

Celery

Results from supervised field trials on celery conducted in the US were provided to the Meeting.

A total of six independent supervised trials were conducted in the US on celery according to the US GAP for leafy vegetables, except Brassica vegetables, which includes the leaf petioles subgroup, and allows three applications at a maximum rate of 0.1 kg ai/ha with a 0 PHI.

Residues of flonicamid matching the US GAP were: 0.35, 0.43, 0.44, 0.45, 0.46, 0.93 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg and an STMR of 0.45 mg/kg for celery.

Cereal grains

Results from supervised trials on wheat and barley conducted in Northern and Southern EU were provided to the meeting.

A total of 23 independent supervised trials were conducted in EU on wheat (15) and barley (8). The wheat trials were conducted according to the Slovenia GAP which allows two applications at a maximum rate of 0.07 kg ai/ha with a 28-day PHI.

As there is no GAP for barley, these trials were not considered further.

Residues of flonicamid in wheat grain matching the Slovenia GAP were: < 0.01 (11), 0.01, 0.02, 0.04 and 0.06 mg/kg.

The Meeting estimated a maximum residue level of 0.08 mg/kg and an STMR of 0.01 mg/kg for wheat.

Tree nuts

Results from supervised field trials on almonds, pecans and pistachios conducted in the US were provided to the Meeting.

A total of 12 independent supervised trials were conducted in the US on almonds (5), pecans (5) and pistachios (2) according to the US GAP which allows three applications at a maximum rate of 0.1 kg ai/ha with a 40-day PHI.

Residues of flonicamid in almond nutmeats matching the US GAP were: < 0.01 (5) mg/kg.

Residues of flonicamid in pecan nutmeats matching the US GAP were: < 0.01 (5) mg/kg.

Residues of flonicamid in pistachios matching the US GAP were 0.02 and 0.04 mg/kg.

As the Meeting could not conclude that there are no measurable residues in all tree nuts in the tree nut crop group and considering the insufficient number of supervised residue trials for pistachios, the Meeting agreed to estimate individual maximum residue levels for almonds and pecans at 0.01* mg/kg with an STMR of 0.01 mg/kg.

Oilseeds

Rape seed

Results from supervised field trials on rape seed conducted in the US were provided to the Meeting.

A total of nine independent supervised trials were conducted in the US on rape seed according to the US GAP which allows three applications at a maximum rate of 0.1 kg ai/ha with a 7-day PHI.

Residues of flonicamid matching the US GAP were: < 0.02, 0.02 (3), 0.04, 0.08, 0.09, 0.17 and 0.33 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.04 mg/kg for rape seed.

Cotton seed

Results from supervised field trials on cotton conducted in the US and Australia were provided to the Meeting.

The GAP in the US is three applications at a maximum rate of 0.1 kg ai/ha with a 30-day PHI while the GAP in Australia is three applications at a maximum rate of 0.07 kg ai/ha with a 7-day PHI.

As the critical GAP is in Australia, only the Australian trials were considered.

Residues of flonicamid in cottonseed from eight independent supervised residue trials matching the Australian critical GAP were: 0.01 (2), 0.02, 0.04, 0.09, 0.13, 0.16 and 0.34 mg/kg.

The Meeting estimated a maximum residue level of 0.6 mg/kg and an STMR of 0.06 mg/kg for cottonseed.

Mint

Results from supervised field trials on fresh mint leaves conducted in the US were provided to the Meeting.

A total of three independent supervised trials were conducted in the US on mint according to the US GAP which allows three applications at a maximum rate of 0.1 kg ai/ha with a 7-day PHI.

Residues of flonicamid matching the US GAP were: 1.70, 1.92 and 2.36 mg/kg.

The Meeting estimated a maximum residue level of 6 mg/kg and an STMR of 1.92 mg/kg for mint.

Dried hops

Results from supervised field trials on dried hops conducted in the US were provided to the Meeting.

A total of four independent supervised trials were conducted in the US on dried hops according to the US GAP which allows three applications at a maximum rate of 0.1 kg ai/ha with a 10-day PHI.

Residues of flonicamid matching the US GAP were: 0.56, 1.15, 2.82 and 9.33 mg/kg.

The Meeting estimated a maximum residue level of 20 mg/kg and an STMR of 1.98 mg/kg for dried hops.

Teas

Results from supervised field trials on tea conducted in Japan were provided to the Meeting.

A total of two independent supervised trials were conducted in Japan on tea according to the Japanese GAP which allows one application at a maximum rate of 0.1 kg ai/ha with a 7-day PHI.

Residues of flonicamid in green tea leaves matching the Japanese GAP were: 15.7 and 20.1 mg/kg.

There is insufficient data for the Meeting to estimate a maximum residue level.

Animal feeds

Straw, fodder and forage of cereal grains and grasses including buckwheat fodder forage

Wheat

Results from supervised trials on wheat and barley conducted in Northern and Southern EU were provided to the meeting.

A total of 23 independent supervised trials were conducted in EU on wheat (15) and barley (8). The wheat trials were conducted according to the Slovenia GAP which allows two applications at a maximum rate of 0.07 kg ai/ha with a 28-day PHI.

As there is no GAP for barley, these trials were not considered further.

Residues of flonicamid in wheat forage matching the Slovenia Gap were: 0.64, 0.69, 0.83, 0.88 and 0.99 (2).

The Meeting estimated a maximum residue level of 3.0 mg/kg and a median of 0.86 mg/kg for wheat forage.

Residues of flonicamid in wheat straw matching the Slovenia GAP were: < 0.02 (5), 0.02, 0.04 (2), 0.05 (3), 0.08, 0.09, 0.11 and 0.23 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and a median of 0.04 mg/kg for wheat straw and fodder, dry.

Alfalfa

Results from six independent supervised field trials on alfalfa (4) and clover (2) conducted in the US were provided to the Meeting.

The US GAP for alfalfa grown west of the Rockies allows two applications at a maximum rate of 0.10 kg ai/ha with PHIs of 14 days for seed and forage and 60 days for hay.

Two supervised trials were conducted on clover in the US, however, in the absence of a US GAP, these trials were not considered.

Four trials were conducted on alfalfa in the US, of which only two were conducted according to the US GAP. In the absence of a sufficient number of trials, the Meeting could not estimate a maximum residue level or a median residue for alfalfa seed, forage and hay.

Miscellaneous fodder and forage crops (fodder)

Almond hulls

Results from supervised field trials on almonds conducted in the US were provided to the Meeting.

Five independent trials were conducted on almonds in the US. The GAP in the US allows three applications at a maximum rate of 0.10 kg ai/ha with a PHI of 40 days.

Residues in almond hulls (dry weight) from five trials matching US GAP were: 0.92, 1.09, 1.81, 2.75 and 4.73 mg/kg. The meeting estimated a maximum residue level of 9 mg/kg and a median residue of 1.8 mg/kg.

Cotton gin trash

Results from supervised field trials on cotton conducted in the US and Australia were provided to the Meeting.

The GAP in the US is three applications at a maximum rate of 0.1 kg ai/ha with a 30-day PHI while the GAP in Australia is three applications at a maximum rate of 0.07 kg ai/ha with a 7-day PHI.

As the critical GAP is in Australia, only the Australian trials were considered.

The residues of flonicamid in cotton gin trash from eight independent supervised trials matching the Australian critical GAP were: 0.66, 1.20, 1.33, 1.60, 1.70, 2.30, 2.75, 3.00 and 3.72 mg/kg.

The Meeting estimated a median residue of 1.7 mg/kg.

Fate of residues during processing

High temperature hydrolysis

To simulate the degradation of flonicamid during pasteurization, baking, brewing, boiling and sterilisation, the hydrolysis of radio-labelled flonicamid was investigated in sterile buffered aqueous solutions.

After incubation at 90 °C (pH 4) for 20 minutes, 100 °C (pH 5) for 60 minutes or 120 °C (pH 6) for 20 minutes, no loss of radioactivity occurred. More specifically, flonicamid accounted for at least 96% of the applied radioactivity. Therefore, very limited degradation of flonicamid was observed in aqueous buffer solutions under all the conditions tested with no significant degradation product being formed.

Processing

The Meeting received information on the fate of flonicamid residues and its metabolites TFNA-AM, TFNA and TFNG during the processing of raw agricultural commodities (RAC) like apples, peaches, plums, tomatoes, potatoes, rape seed, cotton and mint.

Processing factors calculated for the processed commodities of the above raw agricultural commodities are shown in the table below. STMR-Ps were calculated for processed commodities for which maximum residue levels were estimated.

RAC	Processed Commodity	Calculated processing factors	Best estimate	STMR-P
		Flonicamid		
Peaches	Canned peaches	0.3, 0.5, 0.3, 3.3	0.3 (median)	0.08
	Juice	1.0, 1.0, 0.3, 0.5	0.8 (median)	1.8
	Jam	0.3, 1.0, 1.0, 0.2	0.7 (median)	0.16
	Puree	0.7, 1.0, 1.0, 0.8	0.9 (median)	0.21
Plums	Dried prunes	1.0	1.0	0.04
Tomato	Paste	16.1	16.1	1.45
Potato	Chips	0.95	0.95	0.01
	Flakes	2.7	2.7	0.03
Canola	Refined oil	< 0.1	0.1	0.004
Cotton	Refined oil	< 0.24 (US), 0.6 and 0.04 (AUS)	0.32 (mean; AUS)	0.02
Mint	Oil	< 0.03	0.03	0.06

As the residue concentration in both apple juice and apple pomace were higher than in fresh apple which is not physically possible, the Meeting determined that the apple processing study was not reliable and did not calculate a processing factor for juice.

As the residue concentration is higher in tomato paste than in fresh tomato, the Meeting estimated a maximum residue level of 7.0 mg/kg by multiplying the maximum residue level for fruiting vegetables, other than cucurbits, (0.4 mg/kg) by 16.1.

Residues in animal commodities

Farm animal feeding studies

The Meeting received information on the residue levels arising in tissues and milk when three groups of dairy cows were fed with a diet containing 2.50, 6.89 and 23.7 ppm of a 1:1 mixture of flonicamid:TFNG for 28 consecutive days. As demonstrated in the metabolism studies, residues of TFNG present in feed items may be converted to TFNA-AM. Therefore, the Meeting concluded that the test material used in the feeding studies was appropriate.

In milk, no quantifiable (< LOQ) residues of flonicamid were detected in any test group. For TFNA-AM, the average residues increased from < LOQ in the low dose group to 0.02 mg/kg in the mid dose group and to 0.08 mg/kg in the high dose group.

In liver, no quantifiable residues of flonicamid were detected. For TFNA-AM, residues were detected in the mid and high dose groups above the LOQ using two different analytical methods (FMC-P-3580/RCC 844743) with different LOQ (0.025/0.01 mg/kg). TFNA-AM levels increased from less than LOQ in the low dose group to 0.039/0.02 mg/kg in the mid dose group and 0.113/0.05 mg/kg in the high dose group.

In kidney, TFNA-AM was detected in the medium and high dose groups above the LOQ using the same analytical methods as those used for kidney. TFNA-AM levels increased from levels below LOQ in the low dose group to 0.031/0.02 mg/kg in the mid dose group and 0.105/0.09 mg/kg in the high dose group.

In muscle, only TFNA-AM was found. The level increased from below LOQ (0.025 mg/kg) in the low dose group to 0.027 mg/kg in the mid dose group and 0.088 mg/kg in the high dose group. Similarly, only TFNA-AM was measurable in fat and only at the high dose level (0.015 mg/kg).

The Meeting also received information on the residue levels arising in tissues and eggs when groups of laying hens were fed with a diet containing 0.26, 2.51, 7.47 and 25.83 ppm of a 1:1 mixture of flonicamid:TFNG for 28 consecutive days.

The average flonicamid residues in eggs increased from < LOQ in the very low and low dose groups to 0.02 mg/kg in the mid dose group and to 0.08 mg/kg in the high dose group. Average residues of TFNA-AM increased from < LOQ in the very low and low dose groups to 0.27 mg/kg in the mid dose group and 0.95 mg/kg in the high dose group.

No quantifiable residues (< LOQ) of flonicamid were found in muscle in any treatment group. No quantifiable residues (< LOQ) of TFNA-AM was measurable in muscle at the very low dose group, but there appeared to be a dose response relationship at all other dose levels; 0.049 mg/kg in the low dose group, 0.168 mg/kg in the mid dose group and 0.654 mg/kg in the high dose group.

In liver and fat, no quantifiable residues (< LOQ) of flonicamid were found at any dosing level. For liver, TFNA-AM residues increased from < 0.01 mg/kg (very low) to 0.05 mg/kg (low) to 0.17 mg/kg (mid) and 0.71 mg/kg (high) while for fat, TFNA-AM residues increased from 0.01 mg/kg (very low) to 0.02 mg/kg (low) to 0.06 mg/kg (mid) and 0.29 mg/kg (high).

Estimated dietary burdens of farm animals

Maximum and mean dietary burden calculations for flonicamid are based on the feed items evaluated for cattle and poultry as presented in Annex 6. The calculations were made according to the livestock diets from Australia, the EU, Japan and US-Canada in the OECD feeding table.

The foliar application of flonicamid to apples, cabbage, potato, almonds, rape seed, cotton and wheat resulted in residues of flonicamid in the following feed items: wet apple pomace, head cabbage with wrapper leaves, potato culls, almond hulls, rape seed meal, undelinted cottonseed, cotton seed hulls, cottonseed meal, gin trash, wheat forage, grain and straw. Based on the named feed items, the calculated maximum animal dietary burden for dairy or beef cattle was in Australia (3.96 ppm), followed by EU (1.39 ppm) and US-Canada (0.29 ppm).

	Livestock dietary burden, flonicamid, ppm of dry matter							
	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.27	0.13	1.39	1.02	3.96 a	3.44 c	0.003	0.003
Dairy cattle	0.81	0.70	0.82	0.71	2.38 b	2.07 d	0.002	0.002
Poultry—broiler	0.03	0.03	0.01	0.01	0.02	0.02	0	0
Poultry—layer	0	0	0.40 e	0.34 f	0	0	0	0

[a] Suitable for MRL estimates for mammalian meat, fat and edible offal

[b]. Suitable for MRL estimates for milk

[c] Suitable for STMR estimates for mammalian meat, edible offal

[d] Suitable for STMR estimate for milk

[e] Suitable for MRL estimates for eggs, meat, fat and edible offal of poultry

[f] Suitable for STMR estimates for eggs, meat, fat and edible offal of poultry

Animal commodities maximum residue level estimation

As all dietary burdens were lower than the lowest feeding levels from the dairy cow and laying hen feeding studies and since all residues of flonicamid and TFNA-AM were below the limit of quantitation at the lowest feeding levels, there is no expectation of any measurable transfer of residues from the feed items into the livestock commodities (see tables below).

	Feed level (ppm) for milk residues	Total flonicamid and TFNA-AM residues in milk (mg/kg)	Feed level for tissue residues (ppm)	Flonicamid and TFNA-AM Residues			
				Muscle	Liver	Kidney	Fat
Maximum residue level—beef or dairy cattle							
Feeding study	2.50	0.043	2.50 6.89	< 0.045 0.050	< 0.045 0.062	< 0.045 0.054	< 0.02 < 0.02
Dietary burden and residue estimate	2.38	0.04	3.96	0.047	0.051	0.048	< 0.02
STMR—beef or dairy cattle							
Feeding study	2.50	0.041	2.50 6.89	< 0.045 0.047	< 0.045 0.059	< 0.045 0.051	< 0.02 < 0.02
Dietary burden and residue estimate	2.07	0.04	3.44	0.045	0.048	0.046	< 0.02

	Feed level (ppm) for egg residues	Total flonicamid and TFNA-AM residues in eggs (mg/kg)	Feed level for tissue residues (ppm)	Flonicamid and TFNA-AM Residues		
				Muscle	Liver	Fat
Maximum residue level—poultry broiler or layer						
Feeding study	0.26 2.51	0.02 0.11	0.26 2.51	< 0.02 0.07	< 0.02 0.08	< 0.02 0.04
Dietary burden and residue estimate	0.40	0.03	0.40	0.02	0.02	0.02
STMR—poultry broiler or layer						
Feeding study	0.26 2.51	0.02 0.09	0.26 2.51	< 0.02 0.06	< 0.02 0.06	< 0.02 0.03
Dietary burden and residue estimate	0.34	0.02	0.34	0.02	0.02	0.02

The Meeting estimated maximum residue levels of 0.02* mg/kg for mammalian fats, 0.04 mg/kg for milks and 0.05 mg/kg for meat from mammals other than marine mammals and 0.06 mg/kg for edible offal (mammalian). The STMRs for mammalian fats, milks, meat from mammals other than marine mammals and edible offal (mammalian) are 0.02 mg/kg, 0.04 mg/kg. 0.047 mg/kg and 0.051 mg/kg, respectively.

In addition, the Meeting estimated maximum residue levels of 0.02* mg/kg for poultry meat (including Pigeon meat), poultry fats and edible offal of poultry and 0.03 mg/kg for eggs. The STMRs were 0.02 mg/kg, 0.02 mg/kg, 0.02 mg/kg and 0.02 mg/kg for meat, edible offal, fat and eggs, respectively.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed in Annex 1 are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of the residue for compliance with the MRL and for estimation of dietary intake for plant commodities: *Flonicamid*

Definition of the residue for compliance with the MRL and for estimation of dietary intake for animal commodities: Sum of flonicamid, N-cyanomethyl-4-(trifluoromethyl)nicotinamide and the metabolite TFNA-AM, 4-(trifluoromethyl)nicotinamide.

CCN	**Commodity**	**Recommended Maximum residue level (mg/kg)**		**STMR or STMR-P mg/kg**	**HR or HR-P mg/kg**
		New	**Previous**		
TN 0660	Almonds	0.01*		0.01	
VB 0040	Brassica (cole or cabbage) vegetables, Head cabbages, Flowerhead brassicas	2		0.36	
VL 0054	Brassica leafy vegetables	15		8.31	
VS 0624	Celery	1.5		0.45	
FS 0013	Cherries	0.9		0.28	
SO 0691	Cotton seed	0.6		0.06	
MM 032	Edible offal (mammalian)	0.06		0.05	
PE 039	Eggs	0.03		0.02	
VC 0045	Fruiting vegetables, Cucurbits	0.2		0.04	
VO 0050	Fruiting vegetables, other than Cucurbits (except mushrooms and sweet corn)	0.4		0.09	
DH 1100	Hops, dry	20		1.98	
VL 0482	Lettuce, Head	1.5		0.51	
VL 0483	Lettuce, Leaf	8		2.67	
FB 2009	Low growing berries	1.5		0.37	
MM 031	Mammalian fats	0.02		0.02	
MM 030	Meat (from mammals other than marine mammals)	0.05		0.04	
MM 033	Milks	0.04		0.04	
HH 0738	Mints	6		1.92	
AM 0660	Miscellaneous fodder and forage crops (fodder)	9		1.81	
FS 2001	Peaches (including Nectarine and Apricot)	0.7		0.14	
TN 0672	Pecan	0.01*		0.01	
FS 0014	Plums (including Prunes)	0.1		0.03	
FP 0009	Pome fruits	0.8		0.13	
VR 0589	Potatoes	0.015		0.01	
PF 037	Poultry fats	0.02		0.02	
PM 036	Poultry meat (including Pigeon meat)	0.02		0.02	
PO 038	Poultry, Edible offal of	0.02		0.02	
VR 0494	Radish	0.4		0.1	
VL 0494	Radish leaves	20		8.5	
SO 0495	Rape seed	0.5		0.04	
VL 0502	Spinach	20		5.72	
AF 051	Straw, fodder and forage of cereal grains and grasses (including buckwheat fodder) (forage)	3		0.86	
AS 051	Straw, fodder and forage of cereal grains and grasses (including buckwheat fodder) (straws and fodders dry)	0.3		0.04	
VW 0448	Tomato paste	7		1.45	
GC 0654	Wheat	0.08		0.01	
	Canned peaches			0.1	
OC 0691	Cotton seed oil, crude			0.02	

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
DF 0014	Prunes			0.04	
	Head cabbage without wrapper leaves			0.025	
	Mint oil			0.06	
	Peach Jam			0.16	
	Peach Juice			1.8	
	Peach Puree			0.21	
	Potato chips			0.01	
	Potato flakes			0.03	
OR 0495	Rape seed oil, edible			0.004	
AB 0691	Cotton seed hulls			0.13	
AB 1203	Cotton seed meal			0.14	
	Rape seed meal			0.004	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Dietary Intakes (IEDIs) of flonicamid were calculated for the 17 GEMS/Food cluster diets using STMRs and STMR-Ps estimated by the current Meeting (Annex 3 to the 2015 Report) estimated by the current Meeting (Annex 3). The ADI is 0–0.07 mg/kg bw and the calculated IEDIs were 1–10% of the maximum ADI. The Meeting concluded that the long-term intake of residues of flonicamid resulting from the uses considered by the current JMPR is unlikely to present a public health concern.

Short-term intake

The Meeting decided that an ARfD is unnecessary and concluded that the short-term intake of residues resulting from the use of flonicamid, considered by the present Meeting, is unlikely to present a public health concern.

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