FLUMIOXAZIN (284)

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EXPLANATION

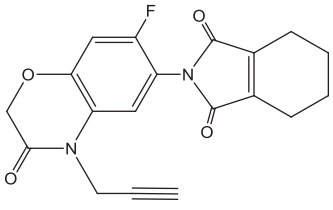
Flumioxazin (S-53482) is variously described as a dicarboxamide, diphenyl-ether or a phenyl-phthalimide herbicide, used for pre-emergent and post-emergent control of a range of broad-leaf weeds and suppression of some grass weed species in a range of fruit, vegetable and field crops. It is non-systemic but is readily absorbed by the foliage of susceptible plants. In the presence of oxygen and light flumioxazin inhibits protoporphyrinogen oxidase resulting in accumulation of porphyrins. The photosensitising action of the accumulated porphyrins enhances peroxidation of membrane lipids and this leads to irreversible damage to the membrane function and structure.

Authorisations exist for the use of flumioxazin as pre-emergence or early post-emergence broadcast treatments, as directed inter-row band soil treatments and as a pre-harvest desiccant (harvest aid) treatment in North America, Europe, Latin America, Australia and some Asian countries.

Flumioxazin was scheduled by the 46th Session of the CCPR as a new compound for consideration by the 2015 JMPR. Residue and analytical aspects of flumioxazin were considered for the first time by the present meeting. The manufacturer submitted studies on metabolism, analytical methods, supervised field trials, processing, freezer storage stability and environmental fate in soil.

In this evaluation, the values presented in the tables are as reported in the various studies, but in the accompanying text, they have generally been rounded to two significant digits.

IDENTITY

ISO common name:	Flumioxazin
Code number	S-53482, V-53482
IUPAC name:	*N*-(7-fluoro-3,4-dihydro-3-oxo-4-prop-2-ynyl-2*H*-1,4-benzoxazin-6-yl)cyclohex-1-ene-1,2-dicarboximide
Chemical Abstracts name:	2-[7-fluoro-3,4-dihydro-3-oxo-4-(2-propynyl)-2*H*-1,4-benzoxazin-6-yl]-4,5,6,7-tetrahydro-1*H*-isoindole-1,3(2*H*)-dione
CAS number	103361-09-7
CIPAC number	578
Molecular mass:	354.3
Molecular formula	$C_{19}H_{15}FN_2O_4$
Structural formula:	

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

A detailed chemical and physical characterisation of the active ingredient is given in the following table.

Test or Study & Annex point	Test material purity and specification	Findings and comments	Reference
Melting point	Pure ai (99.6%)	203.51–209.74 °C	SBP-0056
Boiling point	Pure ai (99.6%)	No boiling point measured decomposition at *ca.* 273 °C	SBP-0056
Relative density	Pure ai (99.6%)	1.4157 (20.1 °C)	SBP-0056
Vapour pressure	Pure ai (99.5%)	3.2×10^{-4} Pa at 22 °C	SBP-0010
pH	Technical (97.6%)	7.29 (25 °C) in saturated solution	SBP-0009
Henry's law constant	calculated	K_H = 0.145 Pa m³ mol^{-1} (20–22 °C)	SBH-059
Appearance	Pure ai (99.5%) Technical (97.6%)	White odourless powdery solid Yellowish brown odourless powder	SBP-0011
Solubility in water	Pure ai (99.6%)	0.786 ± 0.1081 mg/L (20 °C) in distilled water pH effect not investigated because of the neutral properties of flumioxazin	SBP-0057
Solubility in organic solvents (g/L, 25 °C)	Technical (97.6%)	Dichloromethane: 191 Tetrahydrofuran: 53.8 Acetonitrile: 32.3 Ethyl acetate: 17.8 Acetone: 17 Methanol: 1.56 n-Octanol 0.163 Hexane: 0.0247	SBP-0011
Octanol/water partition coefficient	Pure ai (99.9%)	Log P_{OW} 2.55 (20 °C, pH 5.92–5.98)	SBP-0001
Hydrolysis (sterile buffer in the dark, 25 °C)	^{14}C labelled pure ai (> 99%)	THP-label / Phenyl-label DT_{50} (pH 5): 3.4 days / 5 days DT_{50} (pH 7): 19–24 hours / 23–26 hours DT_{50} (pH 9): 14–15 minutes / 21–23 minutes	SBM-0006 SBM-0005
Photolysis characteristics	^{14}C labelled pure ai (> 99%)	DT_{50} (pH 5, 25 °C):20.94 hrs (phenyl-label) DT_{50} (pH 5, 25 °C):26.31 hrs (THP-label) under artificial sunlight conditions Degradates: THPA, APF and 482-PHO	JMPS 578

Formulations

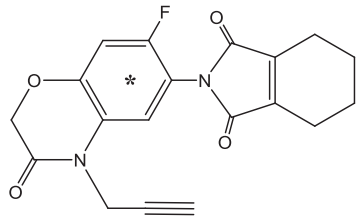
Formulations of flumioxazin are available for use as pre-emergent or post-emergent broadcast or banded soil applications, directed inter-row band sprays in established crops and pre-harvest desiccants, both as solo products or co-formulated or tank-mixed with other herbicides.

Specifications for flumioxazin technical material have been established by the JMPS (2015) and published as FAO Specification 578, available on the FAO Website.

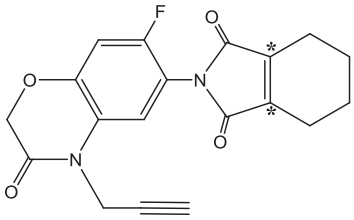
FORMULATION TYPE	FLUMIOXAZIN CONTENT	OTHER ACTIVE INGREDIENTS
WG (Water dispersible granule)	510 g/kg 500 g/kg 400 g/kg	chlorimuron ethyl
GR (Granule)	2.5 g/kg	

METABOLISM AND ENVIRONMENTAL FATE

The Meeting received flumioxazin metabolism studies on plants (soya beans, grapes, sugar cane, apples and peanuts), animals (rats, lactating goats and laying hens) and rotational crops (lettuce, carrots and wheat). Flumioxazin radio-labelled on the phenyl ring or the tetrahydrophthaloyl (THP) ring were used in these studies. The label positions (*) are shown below:

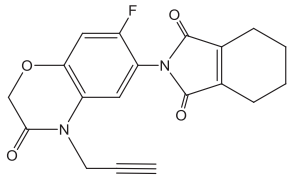
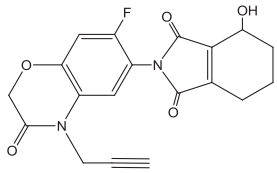
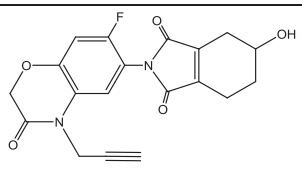


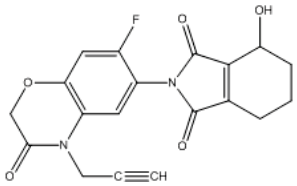
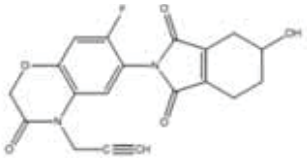
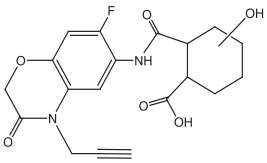
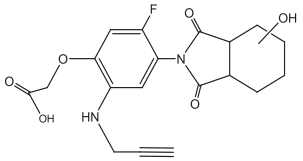
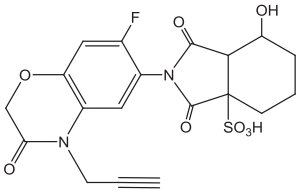
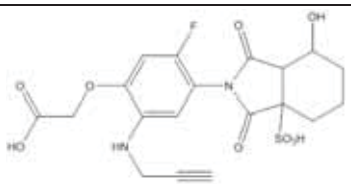
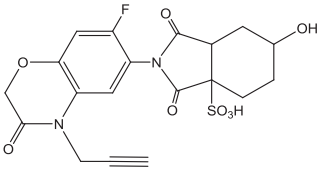
[phenyl-^{14}C]-flumioxazin

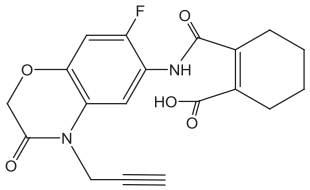
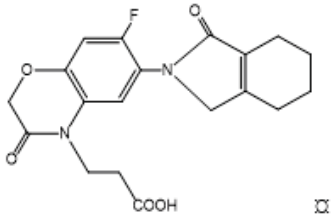
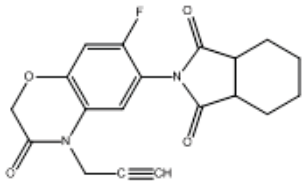
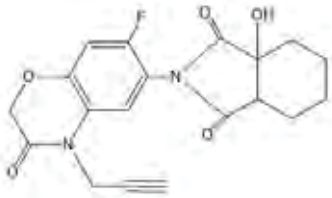
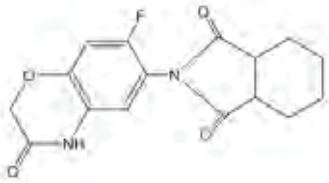
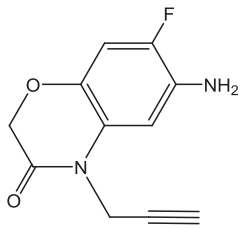
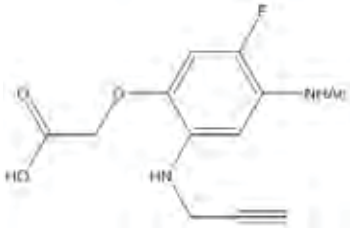


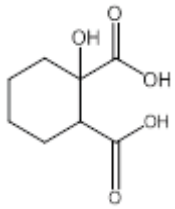
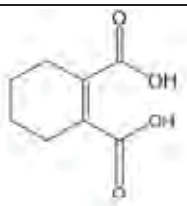
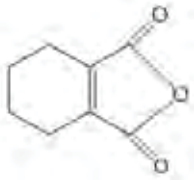
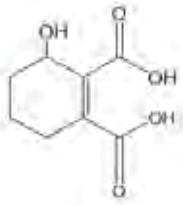
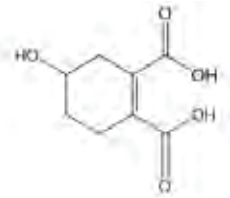
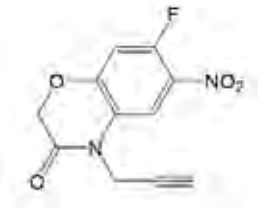
[THP-^{14}C]-flumioxazin

Major metabolites identified in these studies and discussed in this evaluation are listed below.

Compound Name/Code	Structure		Matrices
Flumioxazin (S-53482) (V-53482)		*N*-(7-fluoro-3,4-dihydro-3-oxo-4-prop-2-ynyl-2*H*-1,4-benzoxazin-6-yl)cyclohex-1-ene-1,2-dicarboxamide	Plants Goat Hen Rat Soil Photolysis
3-OH-Flumioxazin		7-fluoro-6-(3-hydroxy-3,4,5,6-tetrahydrophthalimido)-4-(2-propynyl)-2H-1,4-benzoxazin -3(4H)-one	Goat Hen Rat
4-OH-Flumioxazin		7-fluoro-6-(4-hydroxy-3,4,5,6-tetrahydrophthalimido)-4-(2-propynyl)-2H-1,4-benzoxazin-3(4H)-one	Goat Hen Rat

Compound Name/Code	Structure		Matrices
Metabolite B or metabolite F 3-OH-SAT-482		7-fluoro-6-(3-hydroxy-1,2-cyclohexanedicarboximido)-4-(2-propynyl)-2*H*-1,4-benzoxazin-3(4*H*)-one	Goat Rat
4-OH-SAT-482 Exponent asked for revised structures		7-fluoro-6-(4-hydroxy-1,2-cyclohexanedicarboximido)-4-(2-propynyl)-2*H*-1,4-benzoxazin-3(4*H*)-one	
Metabolite C	 OR 	not available	Goat
3-OH-Flumioxazin-SA		7-fluoro-6-(1-sulfo-3-hydoxy-1,2-cyclohexanedicarboximido)-4-(2-propynyl)-2H-1,4-benzoxazin-3(4H)-one	Goat Hen Rat
3-OH-Flumioxazin-ASA		5-fluoro-2-(2-propynylamino-4-(1-sulfo-3-hydroxy-1,2-cyclohexanedicarboximide)phenoxyacetic acid	Rat
4-OH-Flumioxazin-SA		7-fluoro-6-(1-sulfo-4-hydroxy-1,2-cyclohexanedicarboximido)-4-(2-propynyl)-2H-1,4 benzoxazin-3(4H)-one	Goat Hen Rat

Compound Name/Code	Structure		Matrices
482-HA		N-(7-fluoro-3,4-dihdyro-3-oxo-4-prop-2-ynyl-2H- 1,4-benzoxazin-6-yl)cyclohex-1-ene-1-carboxamide-2-carboxylic acid	Plants (rotational) Goat Rat Soil Photolysis
482-CA		2-[7-fluoro-3-oxo-6-(3,4,5,6-tetrahydrophthalimido)-2H-1,4-benzoxazin-4-yl] propionic acid	Plants (rotational) Soil
SAT-482		6-(cis-1,2-cyclohexanedicarboximido)-7-fluoro-4-(2-propynyl)2H-1,4-benzoxazin-3(4H)-one	Goat Rat
1-OH-SAT-482		not available	Plants (rotational)
IMOXA		2-[7-fluoro-3,4-dihydro-3-oxo-2H-1,4-benzoxazin-6-yl]-4,5,6,7-tetrahydro-1H-isoindole-1,3(2H)-dione	Plants (rotational) Soil Photolysis
APF		6-amino-7-fluoro-4-(2-propenyl)-2H-1,4-benzoxazin-3(4H)-one	Plants Goat Hen Rat Soil Photolysis
Ac-APFA		4-acetylamino-5-fluoro-2-(2-propynylamino)phenoxyacetic acid	Rat

Compound Name/Code	Structure		Matrices
1-OH-HPA		1-hydroxy-*trans*-1,2-cyclohexanedicarboxylic acid	Plants Rat Photolysis
THPA		3,4,5,6-tetrahydrophthalic acid	Plants Goat Hen Rat Soil Photolysis
Δ^1-TPA		3,4,5,6-tetrahydrophthalic anhydride	Plants (rotational) Hen Soil Photolysis
3-OH-THPA		3-hydroxy-1-cyclohexene-1,2-dicarboxylic acid	Hen
4-OH-THPA		4-hydroxy-1-cyclohexene-1,2-dicarboxylic acid	Goat Hen
PNF		7-fluoro-6-nitro-4-(2-propynyl)-2*H*-1,4-benzoxazin-3(4*H*)-one	Plants

Environmental fate

The Meeting received information the environmental fate and behaviour of flumioxazin, including hydrolytic stability, photochemical degradation in soils and aerobic metabolism studies.

Hydrolysis

The hydrolytic degradation of flumioxazin was investigated at pH 5, 7 and 9 using either [phenyl-^{14}C]-flumioxazin or [THP-^{14}C]-flumioxazin and reported by Katagi, 1990 [Ref: SBM-0005 and Ref: SBM-0006].

Radio-labelled flumioxazin (0.1 mg/L) was incubated in the dark in sterile aqueous buffered solutions at pH 5, 7, and 9 for up to 30 days at 25 °C. Samples were taken at regular intervals throughout the study and were analysed for total radioactivity by LSC. HPLC was used

to determine the hydrolysis rate and identify the degradation products. Further characterization of degradation products was carried out by two-dimensional TLC with reference standards. The hydrolytic half-lives at each pH were calculated from the analytical data.

In both studies, 94–105% of the applied radioactivity was recovered in all samples analysed. Flumioxazin was rapidly hydrolysed in all three buffered solutions and the half-lives were calculated to be about 3.4–5 days at pH 5, 19–26 hours at pH 7 and 14–23 minutes at pH 9.

The major degradation products after 30 days of incubation in the phenyl-label study were APF (87%) at pH 5; APF (80%) and 482-HA (8–10%) at pH 7; and 482-HA (99%) at pH 9. In the THP-label study, the major degradation products were THPA (96%) and Δ^1-TPA (2.5%) at pH 5; THPA (84%), Δ^1-TPA (6%) and 482-HA (8%) at pH 7; and 482-HA (96%) at pH 9.

Table 1 Major degradation products in aqueous solutions containing [^{14}C]flumioxazin after incubation in the dark at 25 °C for 30 days

DEGRADATION PRODUCTS	% APPLIED RADIOACTIVITY					
	PH 5		PH 7		PH 9	
	PHENYL-LABEL	THP-LABEL	PHENYL-LABEL	THP-LABEL	PHENYL-LABEL	THP-LABEL
Flumioxazin						
1 hr					15	5.5
2 hrs			92	94		
8 hrs	91	89	80	77		
1 days	81	75	41	32	< 0.1	< 0.1
3 days	57	51	25	20	< 0.1	< 0.1
7 days	31	23	20	16	< 0.1	< 0.1
30 days	< 0.1	< 0.1	5.8	3.5	< 0.1	< 0.1
482-HA						
1 hr					84	95
2 hrs			5.1	6.2		
8 hrs	5.3	5.9	19	24		
1 days	4.7	4.2	53	63	99	98
3 days	3.5	2.9	59	68	100	101
7 days	2.8	< 0.1	46	50	99	102
30 days	< 0.1	< 0.1	10	8.1	99	96
THPA						
1 hr						< 0.1
2 hrs				< 0.1		
8 hrs		5.4		< 0.1		
1 days		18		3.3		< 0.1
3 days		47		13		< 0.1
7 days		76		34		< 0.1
30 days		96		84		< 0.1
Δ^1-TPA						
1 hr						< 0.1
2 hrs				< 0.1		
8 hrs		< 0.1		< 0.1		
1 days		< 0.1		< 0.1		< 0.1
3 days		< 0.1		< 0.1		< 0.1
7 days		1.5		0.2		< 0.1
30 days		2.5		6.0		< 0.1

DEGRADATION PRODUCTS	% APPLIED RADIOACTIVITY					
	PH 5		PH 7		PH 9	
	PHENYL-LABEL	THP-LABEL	PHENYL-LABEL	THP-LABEL	PHENYL-LABEL	THP-LABEL
APF						
1 hr						
2 hrs			< 0.1		< 0.1	
8 hrs	4.7		< 0.1			
1 days	13		3.8			
3 days	39		15		< 0.1	
7 days	64		33		< 0.1	
30 days	87		80		< 0.1	

The proposed degradation pathway involves hydrolysis to the amide 482-HA, with further cleavage of the amide link occurring at pH 7 or below, forming THPA (and its anhydride Δ^1-TPA) and APF.

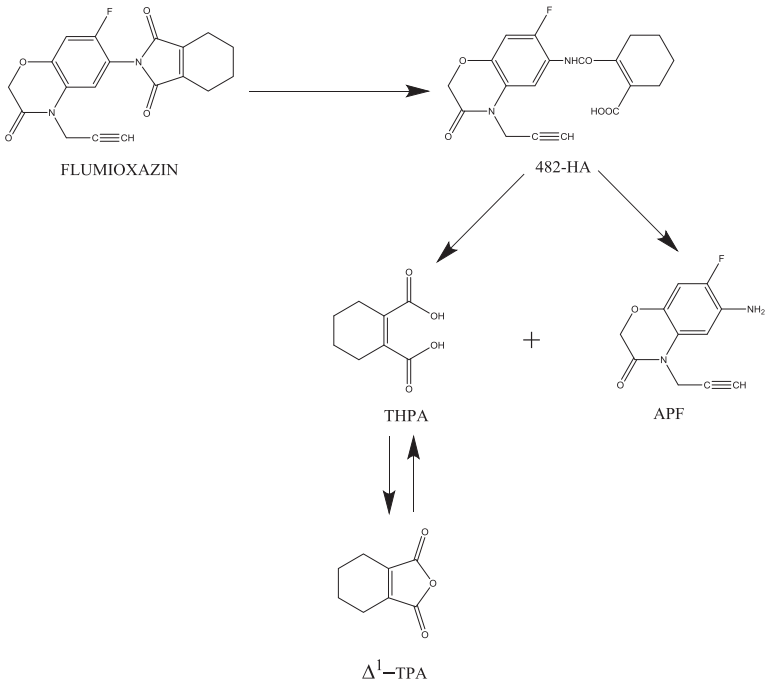


Fig 1 Proposed degradation pathway of flumioxazin in aqueous solutions

Photochemical degradation in soil

Artificial sunlight photo-degradation of [phenyl-^{14}C]-flumioxazin and [THP-^{14}C]-flumioxazin in sandy loam soils was investigated in two studies reported by Fathulla, 1993 [Ref: SBM-0029 and Ref: SBM-0035], respectively.

In these studies, the radio-labelled flumioxazin was applied in acetonitrile to thin layers (1–2 mm thick) of similar Californian sandy loam soils (61–63% sand, 29–30% silt, 8–9% clay, 0.9–1.4% O.M., pH 7.6–7.9) and the soil moisture was adjusted to 75% FC. Samples were irradiated (xenon lamp) for about 12 hours/day at 25–28 °C and duplicate samples were analysed immediately after fortification (Day 0) and intervals for the next 6–14 days.

Soil samples were extracted with acetone:water (5:1, v/v) and then with acidified (pH 1) acetone:water, and analysed using thin-layer chromatography. In the phenyl-label study, the post-

extracted samples containing more than 10% AR were more exhaustively extracted by acid then base refluxing in methanol or by refluxing in dimethylformamide/oxalic acid then basic methanol.

The mean recovery of the applied radioactivity in both studies ranged from 89% to 108%. Volatiles did not exceed 0.5% of the applied radiocarbon for the irradiated samples or 0.2% for the dark controls.

In the phenyl-label study, the acetone extracts from the Day-0 samples contained 102% AR and this decreased in the Day-6 irradiated samples to 48% AR (86% AR in the dark control samples). The more aggressive reflux treatments were able to extract most of the remaining residue, leaving less than 10% AR unextracted.

In the THP-label study, the radioactivity in the combined acetone:water extracts decreased from an initial 99% AR to 83% AR (irradiated) and 87% AR (dark controls) by the end of the 14-day study period, with an increase in the amount of ^{14}C bound to soil, up to 9.3% AR in the irradiated samples and 5% AR in the dark controls.

Flumioxazin accounted for 97–99% AR in the Day-0 samples, decreasing in the irradiated samples to 29% (Day 6—phenyl-label) and 82% AR (Day 7—THP-label) and to 37% AR in the THP-label samples on Day 14. No other TLC areas of radioactivity were more than 10% AR except for Δ^1-TPA and THPA.

Levels of Δ^1-TPA peaked at 22% AR on Day 9 in the irradiated samples, but were < 10% AR in all other sampling times (and in all dark control samples). THPA reached a maximum of about 13% AR (10% AR in the dark control samples). Other minor components were identified as IMOXA and 1-OH-HPA, both measured at < 4% AR in the irradiated samples.

Table 2 Photochemical degradation on soil of [^{14}C]flumioxazin in a Californian sandy loam soil at 25 °C

COMPONENT	% APPLIED RADIOACTIVITY						
	DAY 0	DAY 6–7 [A]		DAY 9		DAY 14	
		IRRADIATED	DARK	IRRADIATED	DARK	IRRADIATED	DARK
Phenyl-label							
Flumioxazin	96.9	29.1	68.4				
IMOXA	0.8	3.1	3.8				
APF + 482-HA	1.4	0.6	ND				
CO_2	< 0.1	0.5	0.2				
Unextracted	3.0	43.3	17.1				
Recovery	105.1	92.3	103.2				
THP-label							
Flumioxazin	99.2	82.2	89.4	36.9	81.5	37.0	51.7
Δ^1-TPA	ND	5.2	3.8	21.6	2.6	8.6	9.0
THPA	ND	1.7	0.2	7.4	10.2	12.9	7.7
1-OH-HPA	ND	ND	ND	3.0	1.5	4.4	8.3
CO_2	ND	ND	ND	ND	ND	ND	ND
Unextracted	1.7	3.1	1.4	4.7	2.3	9.3	5.0
Recovery	100.9	98.3	99.2	93.9	100.5	92.4	92.1

[a] Samples taken on Day 6 in the phenyl-label study and Day 7 in the THP-label study

Radio-labelled flumioxazin degraded more rapidly on irradiated soil than on dark soil, with the amount of ^{14}C bound to soil increasing over time. The calculated soil degradation half-lives were 3.2 days (phenyl-label study) and 8.4 days (THP-label study) and were 12–16 days in non-irradiated samples.

The proposed degradation pathways include hydrolysis of the parent to the amide 482-HA, with further cleavage of the amide link, forming THPA (and its anhydride Δ^1-TPA) and then 1-OH-HPA and also the dealkylation of flumioxazin to form IMOXA.

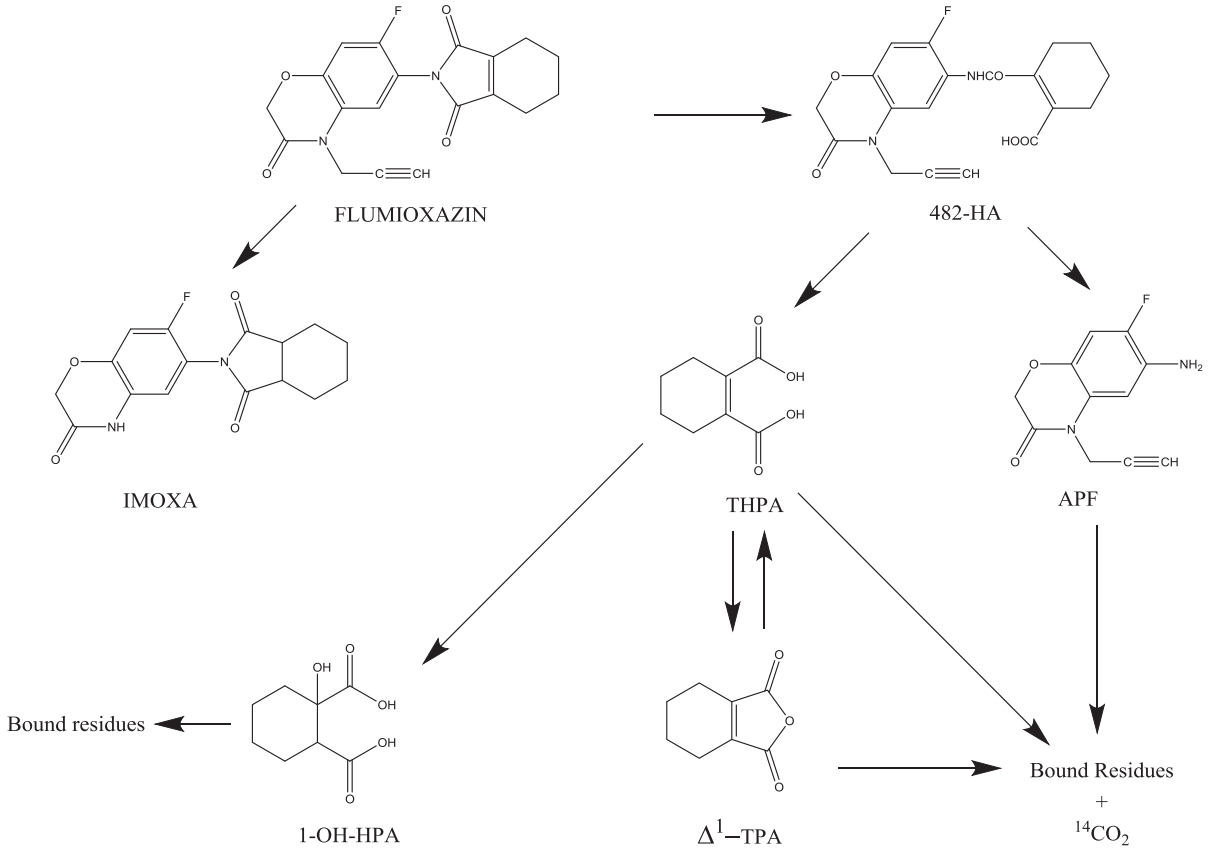


Fig 2 Metabolic pathway proposed for photochemical degradation of flumioxazin on soil

Aerobic soil metabolism

The degradation of flumioxazin in soil was investigated under aerobic conditions using phenyl-labelled flumioxazin (Fathulla, 1991 [Ref: SBM-0012]) and using THP-labelled flumioxazin ((Fathulla, 1993 [Ref: SBM-0030]). In these studies, radio-labelled flumioxazin was applied to sieved, sandy loam soils at a rate of 0.25–0.26 mg/kg, equivalent to about 0.3 kg ai/ha (7.6 cm depth). The characteristics of the soils are summarized below.

Table 3 Characteristics of the soils used in the flumioxazin aerobic soil metabolism studies

SOIL CHARACTERISTICS	PHENYL-LABEL STUDY	THP-LABEL STUDY
Soil type	Sandy Loam	Sandy Loam
Sand	67%	61.2%
Silt	29%	30%
Clay	4%	8.8%
Organic matter	1.2%	1.4%
Cation exchange capacity	18 meq/100 g	6.4 meq/100 g
pH (H_2O)	7.8	7.9
Field moisture capacity	8.9% (at 0.33 bar)	13.4

The soil samples were incubated at 25 °C in glass chambers maintained in a dark, temperature-controlled room for up to 181 days in the phenyl-label study and up to 91 days in the

THP-label study. The glass chambers were connected to traps containing charcoal, ethylene glycol and 2-ethoxyethanol:ethanolamine (1:1, v/v) for collection of volatile organic components and carbon dioxide. Samples were collected for analysis of radioactivity on Day 0 and at various intervals throughout the study periods and extracted with acetone:water (5:1, v/v) and acetone: 0.1 N HCl (9:1, v/v). The combined extracts were analysed by LSC and the distribution of radioactivity in the samples was determined by two-dimensional TLC, HPLC, and comparison with reference standards. The radioactivity remaining in the soil was determined by combustion and LSC. Residues were further extracted with acetonitrile: 0.25 N HCl (4:1, v/v) then 0.5 N sodium hydroxide in the phenyl-label study and acetonitrile: methanol:0.1 N HCl (25:15:10) followed by 0.5 N sodium hydroxide in the THP-label study, with analysis by TLC or LSC. Extraction efficiencies ranged from 94–102% in the two studies.

Radioactivity was distributed primarily among unchanged flumioxazin, CO_2 and soil-bound residues with minor identified components being 482-HA, 482-CA, APF, Δ^1-TPA, THPA and IMOXA. None of these individually exceeded 8% AR. Radioactivity recovered as CO_2 accounted for 12% of the applied radioactivity by Day 181 in the phenyl-label study and accounted for 55% AR at the end of the 91-day TPH-label study period.

Flumioxazin accounted for about 3.5% of the applied radioactivity in phenyl-label soils incubated for 89–181 days, and about 12% AR in the THP-label soils incubated for 90 days and the calculated half-lives in the respective studies were 12 days and 17.5 days. Calculated DT_{90} values (FOMC) were about 51 days (phenyl-label) and 95 days (THP-label).

Table 4 Aerobic degradation of [^{14}C]flumioxazin in a Californian sandy loam soil at 25 °C

COMPONENT	% APPLIED RADIOACTIVITY (0.25-0.26 MG/KG) [A]								
	DAY 0	DAY 3	DAY 7	DAY 14	DAY 28–30	DAY 59–63	DAY 89–91	DAY 120	DAY 181
Phenyl-label									
Flumioxazin	92.9	68.4	60	36.3	18.0	7.6	3.2	3.5	3.7
Origin		1.3	2.4	4.1	8.1	3.9	2.5	2.4	2.8
Region 1			0.4	0.3		2.3 [b]	0.5		
Region 2			0.3	0.3		2.2 [c]	0.1		
Region 3					4.6		5.1	5.5	1.4
Unresolved	6.5	10.8	8.8	17.5	9.2	5.8	4.6	1.1	1.9
Total extracted	99.4	80.5	71.9	58.5	39.9	21.8	16	12.5	9.8
Unextracted	0.7	16.9	25.8	43.0	52.7	71.3	70.0	73.9	73.6
CO_2	–	0.1	0.2	0.6	2.3	5.6	7.7	9.2	11.5
Recovery	100.1	97.4	97.8	102.1	94.9	98.7	94.2	95.8	95.4
THP-label									
Flumioxazin	97.3	78.4	63.6	51.4	28.9	12.3	11.8	–	–
THPA		6.6	5.7	1.0		0.7		–	–
Δ^1-TPA		4.6	5.1	4.8	2.1	0.3		–	–
IMOXA				1.6	2.7	3.0	2.0	–	–
Unresolved		4.2	7.1	2.4	7.0	8.4	1.7	–	–
Total extracted	97.3	93.8	81.5	61.2	40.7	24.7	15.5	–	–
CO_2	-	1.5	7.7	18.4	33.9	48.9	54.9	–	–
Unextracted	2.7	3.9	12.1	16.5	20.0	23.7	29.0	–	–
Recovery	100	99.6	101.3	97.9	96.4	97.5	100.1	–	–

[a] Mean of duplicate samples

[b] Identified as IMOXA plus an unknown component

[c] Identified as 482-HA and 482-CA

Flumioxazin degrades in aerobic soil with calculated half-lives of 12–18 days, with degradation products being CO_2 and a number of minor soil-bound degradates. The proposed metabolic pathways include hydrolysis of the parent compound to 482-HA, oxidation to 482-CA, and by dealkylation to IMOXA. Both IMOXA and 482-HA hydrolyse to THPA, which would be in equilibrium with Δ^1-TPA. THPA appears to be an end product that is incorporated into soil organic components or oxidized to CO_2.

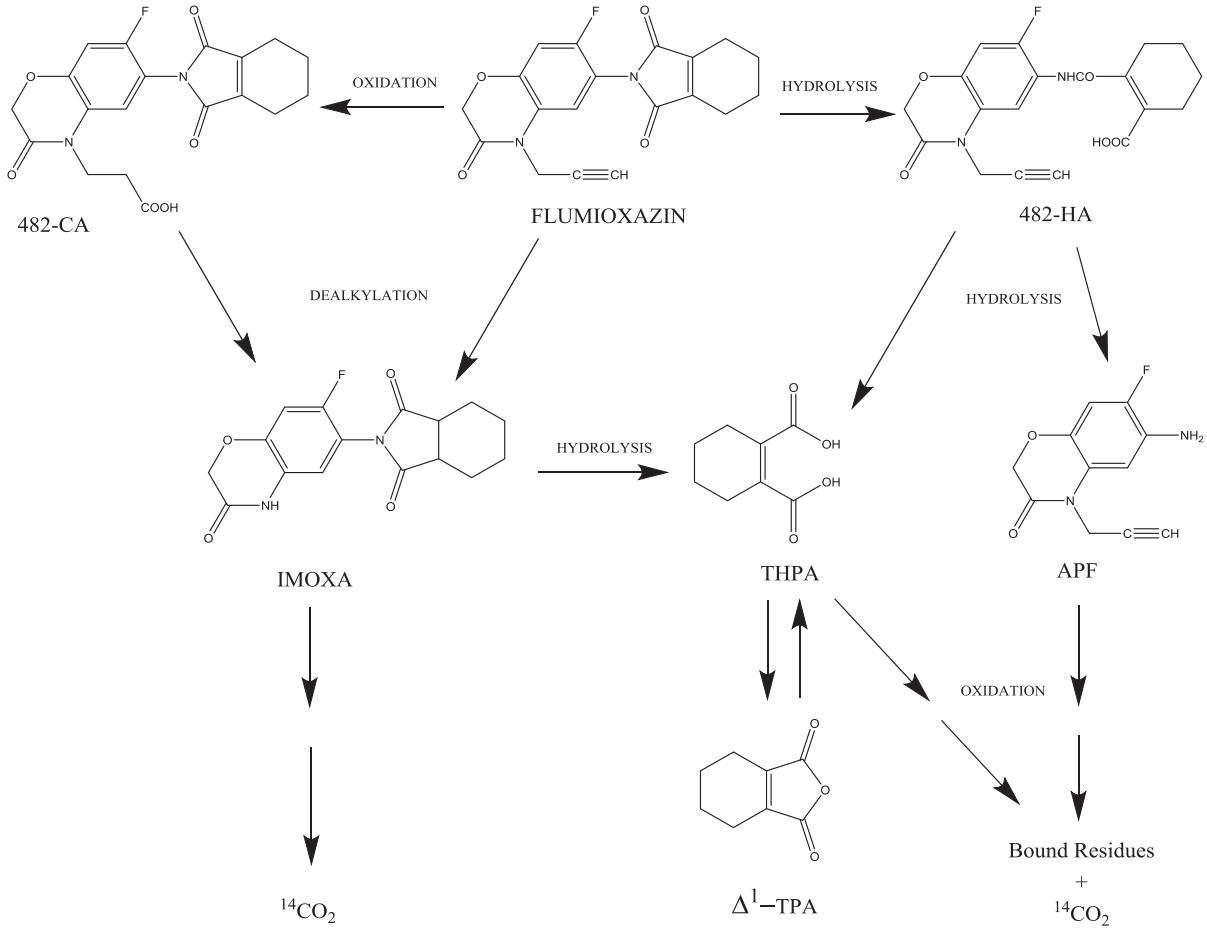


Fig 3 Metabolic Pathway for Aerobic Degradation of Flumioxazin in Soil

Flumioxazin is rapidly hydrolysed in aqueous solutions with average half-lives of 4–5 days (pH 5) decreasing to about 20 minutes at pH 9. Degradation products include 482-HA, THPA (and its anhydride Δ^1-TPA) and APF. The compound 482-HA was the predominant degradate (> 97%) in the pH 9 solution and the cleavage compounds APF and THPA were the major components in the pH 5 and 7 solutions.

Radio-labelled flumioxazin degraded more rapidly on irradiated soil than on dark soil, with the amount of ^{14}C bound to soil increasing over time. THPA and its anhydride Δ^1-TPA together accounted for up to 29% AR in irradiated samples (up to 17% in dark samples). The calculated soil degradation half-lives were 3.2 days (phenyl-label study) and 8.4 days (THP-label study) and were 12–16 days in non-irradiated samples.

In aerobic soil, calculated half-lives for flumioxazin are 12–18 days, with degradates 482-HA, 482-CA and IMOXA, each accounting for less than 7% of the applied radioactivity and generally present at < 0.01 mg/kg. The parent compound accounted for the majority of extractable radioactivity in almost all samples examined.

Plant metabolism

The Meeting received plant metabolism studies on soya beans, grapes, sugar cane, apples, peanuts and rotational crops following treatments with flumioxazin radio-labelled in the phenyl ring or the tetrahydrophthaloyl (THP) ring.

Grape

In a confined metabolism study on grape vines reported by Goodyear, 1998 [Ref: SBM-0064], flumioxazin, radio-labelled in the phenyl ring or the THP ring, was applied to soil surrounding grape vines at a rate equivalent to 0.6 kg ai/ha, the vines were grown to maturity in a glass house and at maturity (91 DAT), samples of grapes and shoots were extracted with acetone:water (1:1, v/v) and radioactivity in the extracts was measured by liquid scintillation counting (LSC) and by combustion analysis in the post-extraction solids.

Total radioactivity in the mature grapes and shoots were extremely low. The mean levels of radioactivity in grapes were 0.0021 mg/kg (-phenyl label) and 0.0054 mg/kg (THP-label) and in the shoots, radioactivity measured 0.014 mg/kg (-phenyl label) and 0.04 mg/kg (THP-label).

The majority of the residue (78–92%) was extracted into acetone or acetone:water with 9–21% of the residue remaining "bound" to the plant material. HPLC analysis of the aqueous extracts indicated the presence of a number of metabolites, the majority of which were polar in nature and were not retained on the column under the chromatographic conditions used. The polar fraction contained about 58% TRR, one other metabolite was present at about 11–14% TRR and eight other components were each present at < 6% TRR. Co-chromatography of the radioactivity with the known standards was not possible due to the high levels of UV-absorbing co-extracted samples.

Apple

In a metabolism study on apples reported by Jalal, 2003 [Ref: SBM-0073], flumioxazin, radio-labelled in the phenyl ring or the THP ring, was applied twice as broadcast sprays to bare soil (1.2 m × 1.2 m loamy sand plots) surrounding 4 year-old trees, with about 30 cm of tree trunk receiving direct spray. Treatments equivalent to 0.47 kg ai/ha were applied 47 days before fruit thinning and 60 days later (about 60 days before fruit maturity).

Apples were sampled and analysed at tree thinning (immature apples) and at harvest (mature apples. Combustion analysis was validated using spiked control apples, with a recovery rate of about 95%.

Total radioactive residues (TRR) were 0.002 mg/kg in immature apples from either the [phenyl-^{14}C]flumioxazin treated plot or from the [THP-^{14}C]flumioxazin treated plot. TRRs were 0.001 mg/kg in the mature apples from the [phenyl-^{14}C]flumioxazin treated plot and 0.003 mg/kg in apples from the [THP-^{14}C]flumioxazin treated plot. Since these residue levels were extremely low, further characterization or identification of the residues could not be conducted.

Table 5 Radioactive residues in apples following 1–2 soil/trunk applications of [^{14}C]flumioxazin at rates equivalent to 0.47 kg ai/ha

TREATMENT	TOTAL RADIOACTIVITY (MG/KG)	
	Immature apple (47 days after 1st application)	Mature apple (60 days after 2nd application)
Control	< 0.001	< 0.001
Phenyl-label	0.002	0.001
THP-label	0.002	0.003

Peanut

In a metabolism study on peanuts reported by Comezoglu, 1994 [Ref: SBM-0044], flumioxazin radio-labelled in the phenyl ring or the THP ring was applied once as a pre-emergent broadcast soil treatment at rates equivalent to 0.11 kg ai/ha (3 days after sowing) or 0.33 kg ai/ha as a pre-plant treatment, 32 day before sowing (treated plots were re-sown following poor initial crop emergence). Treatments were made by mixing the labelled flumioxazin into soil (sandy loam) taken from each plot and adding the treated soil back to the tops of the respective plots.

Samples of mature foliage and whole peanuts were harvested from the 0.11 kg ai/ha plots 194 days after treatment (DAT) and from the 0.33 kg ai/ha plots 245 days after resowing (277 DAT). Samples of foliage (vines) were frozen immediately after sampling. Whole peanuts were washed to remove adhering soil and separated into hulls, seed coats and nutmeats. Samples were frozen and shipped on dry ice by overnight courier to the analytical laboratory where samples were stored at < –10 °C prior to analysis.

Samples were homogenized with dry ice and total radioactive residues (TRR) were measured by combustion and LSC analysis.

Total radioactive residues (TRR) in all matrices from the 0.11 kg ai/ha pre-plant treatment were < 0.04 mg/kg, with ^{14}C-residues being lower in the phenyl-label samples. TRRs in samples from the 0.33 kg ai/ha pre-plant treatment were ca.3× higher than those from the 0.11 kg ai/ha treatment except for the phenyl-label hulls and the THP-label vines. Radioactive residues were generally lowest in vines (0.009–0.027 mg/kg) and highest in hulls (0.019–0.166 mg/kg).

Table 6 Radioactive residues in peanut matrices following single pre-plant or pre-emergence soil treatments of [^{14}C]flumioxazin

MATRIX	TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)			
	PRE-EMERGENT TREATMENT (0.11 KG AI/HA) 3 DAYS AFTER SOWING, SAMPLED 194 DAT		PRE-PLANT TREATMENT (0.33 KG AI/HA) 32 DAYS BEFORE SOWING, SAMPLED 277 DAT	
	PHENYL-LABEL	THP-LABEL	PHENYL-LABEL	THP-LABEL
Nutmeats	0.012	0.031	0.044	0.085
Hulls	0.019	0.02	0.166	0.097
Vines	0.009	0.021	0.027	0.023
Seed coats	0.013	0.036	0.045	0.093

Samples were also extracted with acetone:water (4:1) and partitioned with hexane, with total radioactivity in the extracts and the post-extraction solids being measured by combustion and LSC analysis. Radioactivity in the hexane fraction from vines and hulls was too low (≤ 0.002 mg/kg) to permit further characterisation or identification.

The hexane fractions from the nutmeat samples were further partitioned between hexane:acetonitrile (1:1), with essentially all the radioactivity remaining in the hexane phase. Attempts to separate this radioactivity from the oil fraction by freezing to precipitate fats or by chromatography using a silica gel, C_{18}, or gel permeation columns were not successful. However, data from extraction of control nutmeat samples fortified with flumioxazin indicated that parent is unlikely to be present in this fraction.

The aqueous fractions from all samples (except the vines from the pre-plant treatment) were acidified to pH 2–3 and partitioned with ethyl acetate (EtOAc), and selected fractions from various samples were then analysed by reverse-phase HPLC.

Following solvent extraction, the majority of ^{14}C-residues in nutmeats (67–83% TRR), hulls (62–69% TRR) and vines (51–59% TRR) remained in the post extraction solids. To further characterize these residues, the post-extraction solids (PES) fractions from the pre-emergence treatment samples were subjected to sequential enzymatic (cellulase), acid (2 N HCl) and base

(2 N NaOH) hydrolyses. Radioactive residues remaining in the final PES fractions accounted for 23–35% TRR (0.003–0.01 mg/kg) in nutmeats and hulls and 6.4–8.6% TRR (0.001–0.002 mg/kg) in vines.

Radioactive residues in selected aqueous, organic and hydrolysate fractions containing ≥ 10% of the TRR were analysed by reverse phase HPLC using a C_{18} column. Radioactive residues were detected and quantified by LSC and reference standards were detected using a UV absorbance detector (220 nm). Peak retention times for ^{14}C-residues were compared to retention times of reference standards. HPLC peaks containing significant amounts of radioactivity were also analysed by TLC using silica gel plates with a variety of solvent systems.

Flumioxazin residues were measured at levels of < 1% TRR (< 0.001 mg/kg) in the ethyl acetate fractions from hulls and vines. The majority of ^{14}C-residues in solvent and hydrolysate fractions was generally comprised of four regions (A, B, C, and D). Regions A and B were polar in nature and did not correspond to any of the reference standards used in the study. Region C was typically a broad peak, suggesting multiple components, such as 1-OH-HPA, THPA, APF, and 482-HA. Region D was a minor peak with peaks similar to the standards IMOXA, PNF, and 482-CA.

In the nutmeats and vine extracts, each of these general regions accounted for ≤ 0.01 mg/kg in each fraction analysed by HPLC. These regions also each accounted for ≤ 0.005 mg/kg in fractions from hulls, with the exception of Region C which accounted for 0.025–0.038 mg/kg in solvent extracts from the pre-plant (0.33 kg ai/ha) hulls. Subsequent TLC analyses suggested that this region contained minor levels of 1-OH-HPA (≤ 4% TRR, ≤ 0.006 mg/kg) and THPA (≤ 2% TRR, ≤ 0.004 mg/kg) in hulls and vines from the pre-plant samples, however, the majority of ^{14}C-residues in Region C were multiple unknown polar components.

Table 7 Distribution of radioactive residues in peanut nutmeat following one pre-plant or pre-emergent soil application of [^{14}C]flumioxazin

		TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)							
FRACTION	HPLC	PHENYL-LABEL				THP-LABEL			
		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)	
		%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
Acetone/water		24.2	0.002	32.8	0.014	16.9	0.005	29.3	0.027
1st hexane		12.2	0.001	23.0	0.01	6.4	0.002	16.5	0.015
2nd hexane		12.0	0.001	22.7	0.01			16.4	0.015
Acetonitrile		0.14	< 0.001	0.33	< 0.001			0.07	< 0.001
1st aqueous		12.0	0.001	9.8	0.004	10.6	0.003	12.9	0.012
	Region A			5.8	0.002				
	Region B			ND	ND				
	Region C			4.0	0.002				
	Others			ND	ND				
2nd aqueous		8.4	0.001			7.1	0.002	5.7	0.005
	Region A							3.5	0.003
	Region B							0.5	< 0.001
	Region C							1.7	0.002
	Others							ND	ND
Ethyl acetate		3.5	< 0.001			3.5	0.001	7.2	0.007
PES-1		75.9	0.009	67.2	0.03	83.1	0.026	70.7	0.066
Enzyme filtrate		13.9	0.002			15.6	0.005		
	Region A	5.4	< 0.001			7.8	0.002		
	Region B	ND	ND			ND	ND		
	Region C	7.7	0.001			7.7	0.002		

		TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)							
FRACTION	HPLC	PHENYL-LABEL				THP-LABEL			
		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)	
		%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
	Others	0.86 (4)	< 0.001			0.07 (1)	< 0.001		
PES-enzyme		61.9	0.007			67.4	0.021		
Acid-aqueous		22.9	0.003			23.2	0.007		
	Region A	14.4	0.002			16.3	0.005		
	Region B	0.5	< 0.001			ND	ND		
	Region C	1.2	< 0.001			1.5	< 0.001		
	Others	0.4 (5)	< 0.001			0.1 (1)	< 0.001		
	MeOH eluate	6.4	0.001			5.2	0.002		
Acid-EtOAc		5.8	0.001			7.5	0.002		
PES-acid		33.2	0.004			36.8	0.011		
Base-aqueous		8.9	0.001			3.8	0.001		
Base-EtOAc									
PES-base		24.3	0.003			32.9	0.01		

Fractions indicated in bold were analysed by HPLC.

Numbers of other peaks listed in brackets

Regions A and B were polar in nature, not corresponding to any reference standards

Region C, a broad peak possibly including 1-OH-HPA, THPA, APF, and 482-HA

Table 8 Distribution of radioactive residues in peanut hulls following one pre-plant or pre-emergent soil application of [^{14}C]flumioxazin

	TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)								
FRACTION		PHENYL-LABEL				THP-LABEL			
		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)	
		%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
Acetone/water		34.5	0.006	30.9	0.051	31.2	0.006	38.2	0.037
1st hexane		1.0	< 0.001	0.75	0.001	1.2	< 0.001	1.1	0.001
1st aqueous		33.5	0.006	30.2	0.05	30.0	0.006	37.1	0.036
2nd aqueous		18.5	0.004	10.8	0.018	17.0	0.003	13.1	0.013
	Region A	5.2	< 0.001	1.7	0.003	3.8	< 0.001	2.2	0.001
	Region B	ND	ND	0.18	< 0.001	ND	ND	1.1	< 0.001
	Region C	13.3	0.003	8.6	0.014	13.1	0.002	9.8	0.009
	Others	ND	ND	0.36 (2)	0.001	0.08 (1)	< 0.001	ND	ND
Ethyl acetate		15.0	0.003	19.4	0.032	13.1	0.003	24.0	0.023
	Region A	0.54	< 0.001	1.8	0.003	ND	ND	4.3	0.004
	Region B	ND	ND	ND	ND	1.4	< 0.001	2.0	0.002
	Region C	12.1	0.002	14.8	0.024	9.7	0.002	16.3	0.016
	Region D	0.99	< 0.001	0.54	< 0.001	0.71	< 0.001	0.57	< 0.001
	Flumioxazin	0.55	< 0.001	0.68	< 0.001	0.48	< 0.001	0.67	< 0.001
	Others	0.88 (3)	< 0.001	1.6 (3)	< 0.001	0.77 (3)	< 0.001	0.18 (1)	< 0.001
PES-1		65.5	0.012	69.1	0.115	68.8	0.014	61.8	0.06
Enzyme filtrate		10.3	0.002			7.7	0.002		
	Region A	4.5	< 0.001						

		TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)							
FRACTION		PHENYL-LABEL				THP-LABEL			
		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)	
		%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
	Region B	2.3	< 0.001						
	Region C	3.2	< 0.001						
	Region D	ND	ND						
	Others	0.31 (2)	< 0.001						
PES-enzyme		55.2	0.01			61.2	0.012		
Acid-aqueous		14.8	0.003			11.9	0.002		
	Region A	7.7	0.001			8.4	0.002		
	Region B	ND	ND			ND	ND		
	Region C	1.0	< 0.001			0.73	< 0.001		
	Region D	ND	ND			ND	ND		
	Others	2.7 (3)	< 0.001			ND	ND		
	MeOH eluate	3.47	0.001			2.8	< 0.001		
Acid-EtOAc		7.0	0.001			4.6	0.001		
PES-acid		33.4	0.006			44.7	0.009		
Base-aqueous		5.0	0.001			4.0	0.001		
Base-EtOAc		5.2	0.001			5.5	0.001		
PES-base		23.2	0.004			35.3	0.007		

Fractions indicated in bold were analysed by HPLC.

Numbers of other peaks listed in brackets

Regions A and B were polar in nature, not corresponding to any reference standards

Region C, a broad peak possibly including 1-OH-HPA, THPA, APF, and 482-HA

Region D, a minor peak possibly including IMOXA, PNF, and 482-CA.

Table 9 Distribution of radioactive residues in peanut vines following one pre-plant or pre-emergent soil application of [^{14}C]flumioxazin

		TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)							
FRACTION		PHENYL-LABEL				THP-LABEL			
		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)	
		%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
Acetone/water		47.0	0.004	49.5	0.013	41.1	0.009	47.2	0.011
1st hexane		0.25	< 0.001	4.5	0.001	3.5	0.001	8.8	0.002
1st aqueous		46.7	0.004	45.1	0.012	37.6	0.008	38.4	0.009
	Region A			9.5	0.002			8.2	0.002
	Region B			ND	ND			3.4	< 0.001
	Region C			35.0	0.009			26.1	0.006
	Others			0.52 (1)	< 0.001			0.79 (3)	< 0.001
2nd aqueous		25.4	0.002			23.8	0.005		
	Region A	9.9	< 0.001			9.5	0.002		
	Region B	0.8	< 0.001			0.36	< 0.001		
	Region C	14.6	0.001			13.9	0.003		
	Others	0.11 (1)	< 0.001			0.05 (1)	< 0.001		
Ethyl acetate		21.3	0.002			13.8	0.003		

		TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)							
FRACTION		PHENYL-LABEL				THP-LABEL			
		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)		PRE-EMERGENT (0.11 KG AI/HA)		PRE-PLANT (0.33 KG AI/HA)	
		%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
	Region A	0.27	< 0.001			ND	ND		
	Region B	2.0	< 0.001			1.3	< 0.001		
	Region C	14.9	0.001			9.8	0.002		
	Region D	0.63	< 0.001			0.33	< 0.001		
	Flumioxazin	0.16	< 0.001			ND	ND		
	Others	3.3 (3)	< 0.001			2.4 (5)	< 0.001		
PES-1		53.0	0.005	50.5	0.014	58.9	0.012	52.8	0.012
Enzyme filtrate		11.2	0.001			13.9	0.003		
	Region A	ND	ND			7.7	0.002		
	Region B	2.8	< 0.001			ND	ND		
	Region C	7.5	< 0.001			6.25	0.001		
	Region D	0.3	< 0.001			ND	ND		
	Others	0.56 (1)	< 0.001			ND	ND		
PES-enzyme		41.9	0.004			45.0	0.009		
Acid-aqueous		17.8	0.002			18.1	0.004		
	Region A	9.8	< 0.001			14.8	0.003		
	Region B	ND	ND			ND	ND		
	Region C	1.1	< 0.001			0.95	< 0.001		
	Region D	ND	ND			ND	ND		
	Others	0.07 (1)	< 0.001			0.04	< 0.001		
	MeOH eluate	6.9	0.001			2.4 (1)	0.001		
Acid-EtOAc		5.7	0.001			8.9	0.002		
PES-acid		18.3	0.002			18.0	0.004		
Base-aqueous		6.9	0.001			5.5	0.001		
Base-EtOAc		5.0	0.001			3.9	0.001		
PES-base		6.4	0.001			8.6	0.002		

Fractions indicated in bold were analysed by HPLC.

Numbers of other peaks listed in brackets

Regions A and B were polar in nature, not corresponding to any reference standards

Region C, a broad peak possibly including 1-OH-HPA, THPA, APF, and 482-HA

Region D, a minor peak possibly including IMOXA, PNF, and 482-CA.

Soya bean—Study 1

In a confined metabolism study on soya beans reported by Hubert, 1992 [Ref: SBM-0021], flumioxazin, radio-labelled in the phenyl ring or the THP ring, was applied to soil (sandy loam) three days after sowing at rates equivalent to 0.1 kg ai/ha or 0.2 kg ai/ha. Forage and root samples were taken 70 days after treatment and samples of plants (without pods), pods, seeds and roots were harvested at maturity, 100 days after treatment.

Soya bean forage and seed samples were extracted with acetone:water (4:1) followed by acetone:0.1 M HCl (4:1). The concentrated extracts were partitioned with ethyl acetate and the radioactivity quantified by LSC. The radioactivity in the post-extraction solids was determined by oxidation and LSC. In order to liberate further amounts of radioactivity, successive hydrolysis with 2 N HCl and 2 N sodium hydroxide under reflux (2 hours) was carried out. Following hydrolysis, the aqueous phases were acidified (pH 2–3), extracted with ethyl acetate and

radioactivity in the extracts and post-extraction solids was measured by liquid scintillation counting (LSC). Residues in the post-extraction solids were also analysed by HPLC.

Analysis of the radioactivity in the immature forage and mature plants, pods and seeds indicated preferential uptake from the ^{14}C-THP-radio-labelled material. Total radioactive residues in immature forage were 0.03 mg/kg and 0.06 mg/kg flumioxazin equivalents for the low and high rates of ^{14}C-phenyl-labelled material, respectively. The corresponding values for ^{14}C-THP-labelled treatments were 0.12 mg/kg and 0.14 mg/kg flumioxazin equivalents. Hay from immature forage (dried for 3–7 days to achieve a moisture content of 8.5–12%) contained 0.19 and 0.29 mg/kg flumioxazin equivalents for the high rate ^{14}C-phenyl and ^{14}C-THP treatments, respectively. In pods and seeds, radioactivity levels were 0.02–0.03 mg/kg (phenyl-label) and 0.23–0.36 mg/kg and 0.12–0.18 mg/kg respectively in the THP-label treatments.

Table 10 Radioactive residues in soya bean forage, pods and seeds following a pre-emergent soil application of [^{14}C]flumioxazin

MATRIX	DOSE (KG AI/HA)	RADIOACTIVE RESIDUES (MG/KG FLUMIOXAZIN EQUIVALENTS)			
		70 DAT		100 DAT	
		[PHENYL-^{14}C]-FLUMIOXAZIN	[THP-^{14}C]]-FLUMIOXAZIN	[PHENYL-^{14}C]]-FLUMIOXAZIN	[THP-^{14}C]]-FLUMIOXAZIN
Forage (immature)	0.1	0.03, 0.03	0.13, 0.11		
	0.2	0.07, 0.05	0.12, 0.16		
Hay (immature)	0.1	–	–		–
	0.2	0.19	0.29	–	–
Plants (without pods)	0.1			0.05, 0.04	0.22, 0.3
	0.2			0.06, 0.08	0.29, 0.4
Pods	0.1			0.03, 0.02	0.2, 0.26
	0.2			0.03, 0.02	0.29, 0.42
Seeds	0.1			0.03, 0.02	0.12, 0.13
	0.2			0.03, 0.03	0.17, 0.18

Sequential acetone:water and acetone:HCl extractions were able to extract close to 60% TRR in hay and when followed by acid and base hydrolysis in the case of the forage and seed, was able to extract more than 90% TRR in immature forage and more than 95% TRR in seed.

Table 11 Distribution of radioactive residues in soya bean forage, hay and seeds following one pre-emergent soil application equivalent to 0.2 kg ai [^{14}C]flumioxazin/ha

MATRIX	FORAGE (70 DAY)		SEED (100 DAY)		HAY (70 DAY)	
	% TRR	MG/KG	% TRR	MG/KG	% TRR	MG/KG
[phenyl-^{14}C]Flumioxazin						
Acetone:water	49	0.03	20.3	0.005	46.2	0.2
Acetone:HCl	16.7	0.01	4.7	0.001	12.6	0.02
Residue	29.3	0.02	68.9	0.02	34.5	0.07
Acid hydrolysis	17.7	0.01	49.1	0.009		
Base hydrolysis	3.5	0.002	8.6	0.002		
Unextracted residue	9.4	0.007	4.4	0.0008		
[THP-^{14}C]Flumioxazin						
Acetone:water	43.2	0.07	48.4	0.09		
Acetone:HCl	33.5	0.05	4.7	0.008		

MATRIX	FORAGE (70 DAY)		SEED (100 DAY)		HAY (70 DAY)	
	% TRR	MG/KG	% TRR	MG/KG	% TRR	MG/KG
Residue	27.4	0.04	49.1	0.09		
Acid hydrolysis	15.5	0.03	34.6	0.03		
Base hydrolysis	3.5	0.006	6.5	0.006		
Unextracted residue	6.6	0.01	3.3	0.003		

None of the radioactivity measured in forage, mature seeds or hay from immature forage could be identified as either the parent flumioxazin or any of the available reference standards. Analysis of some of the solubilized forage fractions indicated the presence of 10–16 unknown components that together accounted for 59–89% (0.017–0.086 mg/kg) TRR.

Soya bean—Study 2

In a further confined metabolism study on soya beans reported by Miyashita & Nambu, 1993 [Ref: SBM-0031], flumioxazin, radio-labelled in the phenyl ring or tetrahydrophthaloyl (THP) ring, was applied to sandy loam soil three days after sowing, at rates of about 0.1 kg ai/ha and 0.2 kg ai/ha. Samples of immature whole plants (forage) were taken 53 days after soil treatment and dried to prepare forage hay. Samples of seeds, pods and straw were harvested at maturity, 138 days after treatment.

Forage, hay and seed samples were extracted three times with acetone/water (4:1). The combined acetone/water extracts were concentrated and the aqueous remainder was partitioned three times with hexane. The aqueous remainder was adjusted to pH 2 with hydrochloric acid and partitioned three times into ethyl acetate. Finally the aqueous remainder was neutralised with sodium hydrogen carbonate. In each fraction, radioactivity was quantified by LSC and characterized by HPLC and TLC. The post-extraction solids were further extracted using cellulase digestion, acid and base hydrolysis with the liberated radioactivity being partitioned into ethyl acetate and quantified by LSC.

Total radioactive residues in immature forage (53 DAT) did not exceed 0.7% of the applied radioactivity, indicating that the radioactivity applied to the soil surface did not tend to translocate into the soya bean plants until a later stage. In the phenyl-label study, TRRs in forage from the 0.1 kg ai/ha and 0.2 kg ai/ha plots were about 0.06 mg eq/kg and 0.11 mg eq/kg respectively. Higher levels were present in the forage in the THP-label study (about 0.07 mg eq/kg and 0.2 mg eq/kg in the low and high rate plots). The TRR levels in hay were approximately three to four times higher compared to forage reflecting a concentration of residues due to the loss of water.

In mature soya bean seeds (138 DAT) in the phenyl-label study, TRRs were about 0.03 mg eq/kg (low rate) and about 0.06 mg eq/kg (high rate), and significantly higher in seeds from the equivalent plots in the THP-label study (about 0.25 mg eq/kg and 0.18 mg eq/kg respectively), indicating a preferential uptake of the THP-label.

Table 12 Total radioactive residues in soya bean forage, hay, seeds, pods and straw following a pre-emergent soil application of [^{14}C]flumioxazin

Matrix	Dose (kg ai/ha)	Radioactive residues (mg eq/kg flumioxazin)							
		53 DAT				138 DAT			
		[phenyl-^{14}C]-flumioxazin		[THP-^{14}C]-flumioxazin		[phenyl-^{14}C]-flumioxazin		[THP-^{14}C]-flumioxazin	
		mg eq/kg	%AR	mg eq/kg	%AR	mg eq/kg	%AR	mg eq/kg	%AR
Forage (immature)	0.1	0.055	0.6	0.069	0.7				
	0.2	0.108	0.7	0.196	0.5				
Hay (immature)	0.1	0.155		0.257					
	0.2	0.348		0.617					

Matrix	Dose (kg ai/ha)	Radioactive residues (mg eq/kg flumioxazin)							
		53 DAT				138 DAT			
Seeds	0.1					0.033	0.1	0.245	0.7
	0.2					0.055	0.1	0.177	0.9
Pods	0.1					0.06	0.1	0.326	1.7
	0.2					0.118	0.1	0.551	0.3
Straw	0.1					0.152	0.6	0.207	0.8
	0.2					0.176	0.3	0.254	0.6

Acetone:water extraction was able to retrieve 61–71% TRR from immature forage and hay and 36–66% TRR from seeds. Further partitioning and more aggressive cellulase digestion, acid and base hydrolysis was able to extract most of the remaining radioactivity, with about 1–4% TRR remaining in the post extraction solids.

Flumioxazin made up < 1.8–6.1% TRR in forage and hay, at levels of < 0.01 mg/kg in forage and up to 0.03 mg/kg in hay with trace levels (< 2.3% TRR, < 0.004 mg/kg) reported only in seed from the 0.2 kg ai/ha treatment in the THPA-label study.

The major component of the residue was metabolite 1-OH-HPA (free or partly cellulose conjugated), making up about 15–31% of the TRR in immature forage and hay and about 38–42% TRR (0.06–0.09 mg/kg) in seed. Minor metabolites included THPA (up to 8.6% TRR in forage and hay and < 3.2% TRR, < 0.007 mg/kg) in seeds) and 482-HA and APF found at trace amounts (< 1.8% TRR) in the immature commodities forage and hay.

Table 13 Characterization and identification of residues in soya bean forage 53 days after pre-emergence soil surface application with [^{14}C]flumioxazin

Metabolite	[Phenyl-^{14}C]Flumioxazin				[THP-^{14}C]Flumioxazin			
	100 g ai/ha		200 g ai/ha		100 g ai/ha		200 g ai/ha	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Acetone:water	0.039	69.2	0.081	70.5	0.044	61.2	0.124	70.8
1st Partition								
Hexane phase	0.007	12.7	0.012	10.7	0.006	7.9	0.017	9.5
Flumioxazin	0.004	6.1	0.006	5.5	< 0.001	< 1.8	0.008	4.4
APF	< 0.001	< 1.8	ND	ND				
Single unidentified	< 0.001	< 1.8	0.003	2.7	0.001	1.5	0003	1.4
Others (max & number)	< 0.001 (3)		0.002 (5)		< 0.001 (2)		0.002 (2)	
Others (total)	0.003	6.6	0.003	2.5	0.005	6.4	0.006	3.7
2nd Partition								
Ethyl acetate phase	0.020	35.3	0.043	37.4	0.032	43.7	0.087	49.7
Flumioxazin	< 0.001	< 1.0			< 0.001	< 1.6	< 0.002	< 1.4
482-HA	< 0.001	< 1.0	0.001	0.7	ND	ND	ND	ND
APF	ND	ND	< 0.001	< 0.5				
THPA					0.002	2.6	0.007	4.2
1-OH-HPA					0.011	15.3	0.028	15.8
Single unidentified	0.015	25.8	0.033	28.9	0.012	15.9	0.030	17.0
Others (max & number)	0.003 (22)		0.004 (32)		0.002 (12)		0.006 (11)	
Others (total)	0.005	9.5	0.009	7.8	0.007	9.9	0.022	12.7
Aqueous phase	0.012	21.2	0.026	22.4	0.006	9.6	0.020	11.6
Single unidentified	0.005	9.5	0.017	14.7	Not analysed		0.020	5.6
Others (max & number)	0.002 (7)		0.006 (12)				0.008 (3)	
Others (total)	0.007	11.7	0.09	7.7			0.010	6.0
Cellulase treatment								
Extract	0.006	9.9	0.008	7.2	0.011	15.6	0.029	16.8
1-OH-HPA	Not analysed				< 0.003	< 4.0	0.017	9.4
Single unidentified					0.006	7.9	0.005	2.8
Others (max & number)					< 0.003 (7)		0.004 (3)	
Others (total)					0.005	7.7	0.007	4.6
Acid hydrolysis								
Extract	0.006	9.9	0.013	11.7	0.010	13.9	0.015	8.8
Ethyl acetate phase	Not analysed		0.003	3.0	0.005	7.5	0.007	4.0

Metabolite	[Phenyl-^{14}C]Flumioxazin				[THP-^{14}C]Flumioxazin			
	100 g ai/ha		200 g ai/ha		100 g ai/ha		200 g ai/ha	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Aqueous phase			0.010	8.7	0.005	6.4	0.008	4.8
Alkaline hydrolysis								
Extract	0.004	6.8	0.008	7.4	0.005	7.3	0.005	2.7
PES	0.002	4.2	0.004	3.2	0.003	2.0	0.002	0.9
Total	0.057	100	0.114	100	0.073	100	0.175	100

ND = Non detectable

Table 14 Characterization and identification of residues in soya bean forage hay 53 days after pre-emergence soil surface application with [^{14}C]flumioxazin

Metabolite	[Phenyl-^{14}C]Flumioxazin				[THP-^{14}C]Flumioxazin			
	100 g ai/ha		200 g ai/ha		100 g ai/ha		200 g ai/ha	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Acetone:water	0.107	64.7	0.230	68.9	0.161	60.1	0.354	61.3
1st Partition								
Hexane phase	0.014	8.2	0.028	8.3	0.013	5.0	0.040	6.9
Flumioxazin	0.007	4.4	0.017	5.2	0.006	2.2	0.030	5.1
THPA					< 0.004	< 1.5	ND	ND
Single unidentified	< 0.002	< 1.0	0.005	1.5	ND	ND	0.010	1.8
Others (max & number)	< 0.002 (1)		0.003 (2)		ND		0.006 (2)	
Others (total)	0.007	3.8	0.006	1.6	0.007	2.8	< 0.004	< 0.6
2nd Partition								
Ethyl acetate phase	0.066	39.7	0.134	40.0	0.117	43.7	0.265	45.8
Flumioxazin	ND	ND	ND	ND	ND	ND	ND	ND
482-HA	ND	ND	< 0.003	< 1.0	ND	ND	ND	ND
APF	< 0.003	< 1.6	< 0.003	< 1.0				
THPA					0.010	3.6	0.027	4.7
1-OH-HPA					0.043	15.9	0.068	11.7
Single unidentified	0.048	28.7	0.102	30.6	0.040	14.8	0.110	19.0
Others (max & number).	0.014 (14)		0.027 (19)		0.012 (6)		0.027 (14)	
Others (total)	0.018	11.0	0.032	9.4	0.024	9.4	0.060	10.4
Aqueous phase	0.027	16.8	0.068	20.6	0.031	11.4	0.049	8.6
THPA					0.003	1.2	0.004	0.8
Single unidentified	0.021	12.9	0.061	18.2	0.012	4.6	0.029	5.0
Others (max & number)	0.003 (26)		0.006 (37)		0.003 (7)		0.004 (18)	
Others (total)	0.006	3.9	0.007	2.4	0.016	5.6	0.016	2.8
Cellulase treatment								
Extract	0.021	12.5	0.048	14.3	0.082	30.7	0.172	29.7
THPA					< 0.005	< 1.9	0.018	3.1
1-OH-HPA					0.042	15.6	0.082	14.1
Single unidentified	0.009	5.4	0.024	7.0	0.009	3.5	0.016	2.8
Others (max & number).	< 0.004 (6)		< 0.004 (10)		0.007 (2)		< 0.014 (2)	
Others (total)	0.012	7.1	0.024	7.3	0.031	11.6	0.056	9.7
Acid hydrolysis								
Extract	0.012	7.3	0.032	9.6	0.013	4.9	0.039	6.7
Ethyl acetate phase	0.003	1.9	0.008	2.5	0.013	4.9	0.018	3.1
Aqueous phase	0.009	5.4	0.024	7.1	not analysed		0.021	3.6
Alkaline hydrolysis								
Extract	0.020	12.2	0.012	3.5	0.007	2.7	0.007	1.2
Ethyl acetate phase	0.005	3.1	0.012	3.5	0.007	2.7	0.007	1.2
Aqueous phase	0.015	9.1	not analysed		not analysed		not analysed	
PES	0.006	3.3	0.012	3.7	0.004	1.6	0.006	1.1
Total	0.166	100	0.334	100	0.267	100	0.578	100

ND = non detectable

Table 15 Characterization and identification of residues in soya bean seeds 138 days after pre-emergence soil surface application with [^{14}C]flumioxazin

Metabolite	[Phenyl-^{14}C]Flumioxazin				[THP-^{14}C]Flumioxazin			
	100 g ai/ha		200 g ai/ha		100 g ai/ha		200 g ai/ha	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Acetone:water	0.012	35.9	0.022	45.0	0.145	66.5	0.108	66.3
1st Partition								
Hexane phase	not analysed		not analysed		not analysed		not analysed	
2nd Partition								
Ethyl acetate phase	0.008	24.0	0.014	27.4	0.126	57.7	0..093	56.9
Flumioxazin	ND	ND	ND	ND	< 0.004	< 1.8	< 0.003	< 2.3
THPA					0.013	6.0	0.006	4.0
1-OH-HPA					0.070	32.0	0.055	32.9
Single unidentified	0.003	10.0	0.007	14.1	0.028	13.0	0.017	9.7
Others (max & number)	< 0.002 (5)		0.005 (3)		0.013 (7)		0.015 (7)	
Others (total)	0.005	14.0	0.007	13.3	0.015	6.7	0.015	10.3
Aqueous phase	0.004	11.9	0.008	17.6	0.019	8.8	0.015	9.4
Single unidentified	not analysed				0.011	5.1	0.010	6.6
Others (max & number)					0.003 (12)		0.003 (8)	
Others (total)					0.008	3.7	0.005	2.8
Cellulase treatment								
Extract	0.010	30.4	0.013	26.2	0.061	28.5	0.026	15.9
THPA					< 0.007	< 3.2	< 0.003	< 2.1
1-OH-HPA					0.022	10.2	0.008	5.0
Single unidentified	0.007	20.8	0.006	12.6	0.018	8.4	0.004	2.8
Others (max & number)	< 0.002 (6)		0.004 (5)		0.014 (4)		0.003 (3)	
Others (total)	0.003	9.6	0.007	13.6	0.021	9.9	0.014	8.1
Acid hydrolysis								
Extract	0.010	28.9	0.012	22.3	0.011	4.9	0.018	12.4
Ethyl acetate phase	0.002	4.8	0.001	2.0	0.002	0.8	0.006	3.5
Aqueous phase	0.008	24.1	0.011	20.3	0.009	4.1	0.012	8.9
Alkaline hydrolysis								
Extract	0.001	3.7	0.003	5.9	not analysed		0.007	5.1
PES	0.002	1.1	0.001	0.6	< 0.001	0.1	< 0.001	0.3
Total	0.035	100	0.051	100	0.217	100	0.159	100

ND = non detectable

Table 16 Identified main metabolites in soya bean forage, hay and seeds after pre-emergence soil surface application with [^{14}C]flumioxazin

Metabolite	[Phenyl-^{14}C]Flumioxazin				[THP-^{14}C]Flumioxazin			
	100 g ai/ha		200 g ai/ha		100 g ai/ha		200 g ai/ha	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Forage								
Flumioxazin	0.004	6.1	0.006	5.5	< 0.001	< 1.8	0.008	4.4
482-HA	< 0.001	< 1.0	0.001	0.7	ND	ND	ND	ND
APF	< 0.001	< 1.8	< 0.001	< 0.5				
THPA					0.002	2.6	0.007	4.2
1-OH-HPA (free and conj.)					0.011	15.3	0.045	25.2
1-OH-HPA (conjugated)					< 0.003	< 4.0	0.017	9.4
Hay								
Flumioxazin	0.007	4.4	0.017	5.2	0.006	2.2	0.030	5.1
482-HA	ND	ND	< 0.003	< 1.0	ND	ND	ND	ND
APF	< 0.003	< 1.6	< 0.003	< 1.0				
THPA					0.013	4.8	0.049	8.6
1-OH-HPA (free and conj.)					0.085	31.5	0.150	25.8
1-OH-HPA (conjugated)					0.042	15.6	0.082	14.1
Seeds								
Flumioxazin	ND	ND	ND	ND	< 0.004	< 1.8	< 0.003	< 2.3
482-HA	ND	ND	ND	ND	ND	ND	ND	ND

Metabolite	[Phenyl-^{14}C]Flumioxazin				[THP-^{14}C]Flumioxazin			
	100 g ai/ha		200 g ai/ha		100 g ai/ha		200 g ai/ha	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
APF	ND	ND	ND	ND				
THPA					< 0.007	< 3.2	< 0.003	< 2.1
1-OH-HPA (free and conj.)					0.092	42.2	0.063	37.9
1-OH-HPA (conjugated)					0.022	10.2	0.008	5.0

ND = Non detectable

Sugar cane

In a metabolism study on sugar cane reported by Jalal, 2003 [Ref: SBM-0074], flumioxazin, radio-labelled in the phenyl ring or the THP ring, was applied at a rate equivalent to 0.48 kg ai/ha as a directed spray to 1.5–2 m high sugar cane prior to stem elongation, at the 6–10 leaf stage, with up to 1 m of the cane receiving direct spray. Immature sugarcane forage (leaves and cane) were sampled about a month after the application and mature canes and leaves (3–3.6 m high) were also sampled at maturity, 90 days after treatment, when the canes were 5 cm in diameter.

Samples were homogenized with dry ice and combusted to determine the total radioactive residue (TRR) and were also sequentially extracted with acetonitrile and water with total radioactivity in the extracts and the post-extraction solids being measured by combustion and LSC analysis.

The total radioactive residues determined by combustion analysis were 0.001–0.004 mg/kg in mature cane, 0.23–0.89 mg/kg in immature forage and 0.5–0.52 mg/kg in mature leaves. Acetonitrile and water extraction was able to retrieve more than 90% TRR in immature forage and mature canes. Higher levels (0.53–1.0 mg/kg) were reported in the extracted mature leaf samples (with larger aliquots of a more homogenous mixture of vascular and non-vascular tissues).

Table 17 Total radioactive residues in sugarcane immature forage, mature leaves and canes after a directed foliar application equivalent to 0.48 kg ai [^{14}C]flumioxazin/ha

DETERMINATION METHOD		TOTAL RADIOACTIVITY (MG/KG FLUMIOXAZIN EQUIVALENTS)		
		IMMATURE FORAGE (30 DAT)	MATURE LEAVES (90 DAT)	MATURE CANE (90 DAT)
Combustion:	Phenyl-label	0.227	0.517	0.001
Extraction:	Phenyl-label	0.209	1.046	0.002
Combustion:	THP-label	0.889	0.496	0.004
Extraction:	THP-label	0.888	0.526	0.004

The acetonitrile extracts of the forage, mature leaves and cane were analysed by HPLC and TLC. The post-extraction solids from the mature leaf samples were hydrolysed by refluxing in 2 M HCl for 2 hours, and after ethyl acetate partitioning, the remaining solid fractions were then refluxed in 2 M NaOH for approximately 2 hours and the base hydrolysate was adjusted to pH 1 to precipitate and centrifuge out the insoluble lignin fraction.

More than 90% TRR was able to be solvent-extracted, with the more aggressive extraction methods able to retrieve all but 2% of the remaining TRR.

Flumioxazin was the predominant residue in immature forage (leaves and canes) accounting for 90–93% TRR (0.19 mg/kg—phenyl-label, 0.83 mg/kg—THP-label). Among the minor components, one polar constituent made up 2.8–3.8% of TRR (0.008–0.025 mg/kg). The unextracted residue in the post-extraction solids accounted for 2.3–4.7% of the TRR (0.01–0.02 mg/kg).

Flumioxazin was also the predominant residue in mature leaves, making up 81–88% of TRR (0.92 mg/kg—phenyl-label, 0.427 mg/kg—THP-label). Among the minor components, a polar constituent was found in various extract fractions, making up a total of 5.1–8.7% of TRR (0.046–0.053 mg/kg). In the post-extraction solids, radioactivity was distributed into all plant constituents including the starch, cellulose, lignin, lipids and proteins, but did not exceed 0.03 mg/kg in any individual PES sub-fraction, with none of the individual TLC bands containing significant residue and none corresponded to any of the reference standards.

Flumioxazin also accounted for most of the mature cane residue (68–75% of TRR, 0.001–0.003 mg/kg), with the aqueous extract and PES contained only a trace level (≤ 0.001 mg/kg) of the radioactivity

Table 18 Characterisation and identification of residues in sugar cane matrices following one directed foliar application equivalent to 0.48 kg ai [^{14}C]flumioxazin/ha

Matrix	Immature forage (30 DAT)				Mature leaves (90 DAT)				Mature cane (90 DAT)			
[^{14}C]-label	Phenyl-label		THP-label		Phenyl-label		THP-label		Phenyl-label		THP-label	
TRR mg/kg	0.209		0.888		1.046		0.526		0.002		0.004	
%TRR extracted	95.3		97.7		93.7		90.5		90.0		92.0	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Flumioxazin	0.189	90.3	0.825	92.9	0.922	88.2	0.427	81.1	0.001	74.7	0.003	68.3
Polar	0.008	3.8	0.025	2.8	0.053	5.1	0.046	8.7	< 0.001	6.7	< 0.001	8.9
Others	0.002	1.1	0.017	2.0	0.005	0.5	0.004	0.7	< 0.001	18.6	< 0.001	22.8
Extracted-Acetonitrile	0.189	90.2	0.843	94.9	0.921	88.1	0.427	81.1	0.002	81.4	0.003	77.1
Extracted-Aqueous	0.011	5.1	0.025	2.8	0.059	5.6	0.049	9.3	< 0.001	8.6	0.001	14.8
Unextracted (PES)	0.01	4.7	0.02	2.3	0.065	6.3	0.05	9.5	< 0.001	10.0	< 0.001	8.0
PES lipid/phenol fraction					0.011	1.1	0.012	2.2				
PES starch fraction					0.026	2.5	0.019	3.6				
PES protein fraction					0.007	0.7	0.007	1.3				
PES lignin fraction					0.018	1.8	0.011	2.1				
PES cellulose fraction					0.002	0.2	0.002	0.3				
PES acid hydrolysis - EtOAc fraction					0.011	1.1	0.012	2.2				
PES acid hydrolysis - aqueous fraction					0.026	2.5	0.019	3.6				
Base hydrolysis - acid soluble					0.007	0.7	0.007	1.3				
base hydrolysis - acid insolubles					0.018	1.8	0.011	2.1				
Total	0.209	100	0.888	100	1.046	100	0.526	100	0.002	100	0.004	100

When applied to soil prior to crop emergence or as directed treatments to soil surrounding established plants, flumioxazin does not translocate or accumulate in significant concentrations in plant matrices. In general, no parent or identifiable metabolites were found in the plant matrices analysed although very low levels of metabolites (most also identified as rat metabolites) were identified in peanut plant samples.

Following directed foliar applications, flumioxazin is not translocated, with the majority of the residue remaining as the parent, with some incorporation into natural plant constituents.

Rotational crop metabolism

The Meeting received information on the metabolism of flumioxazin in lettuce, carrot and wheat grown as rotational crops in flumioxazin-treated soil.

Two confined rotational crop studies using lettuce, carrots and wheat were conducted with flumioxazin labelled in the phenyl ring (Patrick, 1993 [Ref: SBM-0034]) or in the THP ring (Patrick, 1993 [Ref: SBM-0048]). In both studies, the radio-label was applied to bare sandy loam soil plots at rates equivalent to 0.11 kg ai/ha or 0.21 kg ai/ha and the rotational crops were planted 30 days after treatment in all plots and 120, 180 and 365 days after treatment in the higher treatment plots. Fallowed plots were maintained outdoors, and except during periods of heavy rainfall when they were covered to prevent flooding, they were exposed to the environment. Planted crops were maintained in screen houses. Phytotoxicity was observed in most of the rotational crops, particularly in lettuce and carrots, with some plots being replanted because of crop failure (See footnotes to the following Tables).

Radioactive residues in soil and plant materials were determined by combustion followed by Liquid Scintillation Counting and extracted residues were characterized and identified by HPLC or TLC using known reference standards. The stability of stored analytical samples was established by analysis of samples at the beginning and at the end of the study, with no significant degradation being observed.

Soils core samples were taken at each planting and sampling date and segmented into 0–10 cm and 10–20 cm samples prior to combustion analysis and in the phenyl-label study, extracted with acetone:water and acetone:aqueous 0.1 N HCl (5:1) prior to HPLC analysis.

In both studies, results showed that crops assimilated only very small amounts of radioactivity when grown in soil treated with radiolabelled flumioxazin. In the phenyl-label study (0.21 kg ai/ha treatment), TRRs above 0.01 mg/kg were found in wheat straw and chaff at all PBIs, in carrot tops (120 day PBI) and in wheat grain from the 30 day PBI plot. Highest residues were 0.02–0.03 mg/kg eq. in wheat straw.

In the THP-label study, TRRs above 0.01 mg/kg eq were found in carrot tops, wheat straw, chaff and grain from the 0.11 kg ai/ha 30 day PBI plots. TRRs increased in some commodities at the 120-day and 180-day plant-back intervals, suggesting that THP-derived cleavage products in soil are either more readily assimilated by the plants or less tightly bound to soil than those from the phenyl label. In the 0.21 kg ai/ha treated plots, highest residues were 0.015 mg/kg eq in wheat forage (180 day PBI), 0.012 mg/kg eq. in lettuce (180 day PBI), 0.045 mg/kg eq. in carrot tops (30 day PBI), 0.022 mg/kg eq. in carrot roots (30 day PBI), 0.13 mg/kg eq. in wheat straw (120 day PBI), 0.043 mg/kg eq. in wheat chaff (120 day PBI) and 0.023 mg/kg eq. in wheat grain (120 day PBI).

Table 19 Total radioactive residues (mg/kg eq) in rotational crops planted 30 days after soil application of [^{14}C]flumioxazin

Crop	Matrix	Total radioactive residues			
		30-day PBI			
		0.11 kg ai/ha		0.21 kg ai/ha	
phenyl-label (mg eq/kg)					
		DAT	mg eq/kg	DAT	mg eq/kg
Wheat forage	Foliage	84	0.002	84	0.006

Crop	Matrix	Total radioactive residues			
		30-day PBI			
		0.11 kg ai/ha		0.21 kg ai/ha	
Lettuce	Foliage	122	0.002 [a]	180	0.005 [b]
Carrot	Foliage	132	0.002	132	0.01
	Root	132	0.001	132	0.005
Wheat	Straw	176	0.013	176	0.029
	Chaff	176	0.005	176	0.011
	Grain	176	0.006	176	0.011
THP-label (mg eq/kg)					
		DAT	mg eq/kg	DAT	mg eq/kg
Wheat forage	Foliage	95	0.006	95	0.008
Lettuce	Foliage	172	0.004 [a]	172	0.003 [b]
Carrot	Foliage	175	0.028	175	0.045 [a]
	Root	175	0.01	175	0.022
Wheat	Straw	159	0.057	159	0.072
	Chaff	159	0.026	159	0.033
	Grain	159	0.013	159	0.017

PBI = Plant-back interval

[a] 60–61 day Plant-back intervals (crop failure)

[b] 90 day Plant-back interval (crop failure)

Table 20 Total radioactive residues (mg/kg) in rotational crops planted 120–365 days after soil application of [^{14}C]flumioxazin (0.21 kg ai/ha)

Crop	Matrix			Total radioactive residues			
		120-day PBI		180-day PBI		365-day PBI	
phenyl-label							
		DAT	mg eq/kg	DAT	mg eq/kg	DAT	mg eq/kg
Wheat forage	Foliage	180	0.003	261	0.003	412	0.001
Lettuce	Foliage	226	0.007 [a]	254	0.002	440	0.002
Carrot	Foliage	281	0.011	330	0.004	462	0.004
	Root	281	0.005	330	0.005	462	0.001
Wheat	Straw	295	0.02	364	0.028	492	0.009
	Chaff	295	0.016	364	0.013	492	0.003
	Grain	295	0.013	364	0.006	492	0.002
THP-label							
		DAT	mg eq/kg	DAT	mg eq/kg	DAT	mg eq/kg
Wheat forage	Foliage	195	0.011	238	0.015	431	0.004
Lettuce	Foliage	195	0.006	253	0.012	431	0.004
Carrot	Foliage	253	0.026	294	0.013	494	0.013
	Root	253	0.01	294	0.004	494	0.005
Wheat	Straw	253	0.131	308	0.062	494	0.049
	Chaff	253	0.043	308	0.027	494	0.016
	Grain	253	0.023	308	0.008	494	0.005

[a] 149 day Plant-back interval (crop failure)

In soil, the extractable radiocarbon showed a slow decrease over time, decreasing to about 50% during the second half of the study period and remained mostly in the top 0–10 cm layer. The major component was flumioxazin, with minor components (each < 0.01 mg/kg) being tentatively identified as 482-HA, 482-CA, IMOXA and APF based on their retention times.

Table 21 Total radioactive residues and flumioxazin residues in soil (0–10 cm layer) following soil applications of [^{14}C]-flumioxazin

Residues in soil (TRR and extracted flumioxazin)						
0.11 kg ai/ha			0.21 kg ai/ha			Sampling point
DAT		Flumioxazin	DAT	TRR	Flumioxazin	

Residues in soil (TRR and extracted flumioxazin)						
0.11 kg ai/ha			0.21 kg ai/ha			Sampling point
DAT		Flumioxazin	DAT	TRR	Flumioxazin	
phenyl-label						
DAT	TRR	Flumioxazin	DAT	TRR	Flumioxazin	
0	0.129		0	0.259	0.305	Application
30	0.113	0.078	30	0.212	0.154	30 d PBI planting
61	0.152	0.082	61	0.239	0.143	61 d PBI replanting (lettuce, carrot)
84	0.149	0.057	84	0.208	0.09	30 d PBI wheat forage sampling
			90	0.155	0.074	90 d PBI replanting (lettuce)
			120	0.208	0.091	120 d PBI planting
122	0.068	0.017				61 d PBI lettuce sampling
132	0.083	0.029				30 d PBI carrot sampling
			132	0.184	0.063	61 d PBI carrot sampling
			149	0.11	0.055	149 d PBI lettuce planting
176	0.086	0.028	176	0.188	0.058	30 d PBI wheat sampling
			180	0.16	0.07	180 d PBI planting
			180	0.138	0.029	149 d PBI lettuce sampling
			180	0.099	0.02	120 d PBI wheat forage sampling
			226	0.207	0.059	120 d PBI lettuce sampling
			261	0.165	0.056	180 d PBI wheat forage sampling
			254	0.173	0.056	180 d PBI lettuce sampling
			281	0.256	0.078	120 d PBI carrot sampling
			295	0.05	0.007	120 d PBI wheat sampling
			330	0.122	0.028	180 d PBI carrot sampling
			364	0.158	0.031	180 d PBI wheat sampling
			365	0.129	0.037	365 d PBI planting
			412	0.061	0.003	365 d PBI wheat forage
			440	0.121	0.009	365 d PBI lettuce sampling
			462	0.148	0.015	365 d PBI carrot sampling

Residues in soil (TRR and extracted flumioxazin)						
0.11 kg ai/ha			0.21 kg ai/ha			Sampling point
DAT		Flumioxazin	DAT	TRR	Flumioxazin	
			492	0.059	0.006	365 d PBI wheat sampling
THP-label						
DAT	TRR	Flumioxazin	DAT	TRR	Flumioxazin	
0	0.1		0	0.194		Application
30	0.111		30	0.144		30 d PBI planting
60	0.096		60	0.131		30 d PBI replanting (lettuce)
			60	0.209		30 d PBI replanting (carrot)
			90	0.138		
95	0.089		95	0.177		30 d PBI wheat forage sampling
			120	0.138		120 d PBI planting
159	0.067		159	0.081		30 d PBI wheat sampling
172	0.062		172	0.095		60 d PBI lettuce sampling
175	0.064					30 d PBI carrot sampling
			175	0.17		60 d PBI carrot sampling
			180	0.118		180 d PBI planting
			195	0.115		120 d PBI wheat forage sampling
			195	0.125		120 d PBI lettuce sampling
			238	0.108		180 d PBI wheat forage sampling
			253	0.132		180 d PBI lettuce sampling
			253	0.118		120 d PBI carrot sampling
			253	0.113		120 d PBI wheat sampling
			294	0.093		180 d PBI carrot sampling
			308	0.106		180 d PBI wheat sampling
			365	0.108		365 d PBI planting
			431	0.122		365 d PBI wheat forage sampling
			431	0.097		365 d PBI lettuce sampling
			494	0.074		365 d PBI carrot sampling

Residues in soil (TRR and extracted flumioxazin)						
0.11 kg ai/ha			0.21 kg ai/ha			Sampling point
DAT		Flumioxazin	DAT	TRR	Flumioxazin	
			494	0.1		365 d PBI wheat sampling

Sequential solvent extractions of samples containing more than 0.01 mg/kg using acetone:water (4:1), acetone:0.1 N HCl (4:1) and refluxing with acetonitrile:0.25 N HCl was able to extract 62–85.5% TRR in wheat straw and chaff from the 30 day PBI plots and 36–61% TRR in the 120 day and 180 day PBI plots in the phenyl-label study and in the THP-label study, extraction efficiencies were 61–84% in wheat straw, chaff and carrot roots, 75–69% in carrot tops at PBIs of 30 days and 120 days, decreasing to 59% (180 day PBI) and 47% in the 365 day PBI samples. Some plant samples containing < 0.01 mg/kg were also analysed and the similar metabolic profile was confirmed.

In wheat grain, 5–13% TRR was able to be extracted in the 30 day and 120 day PBI plots with a further 22–26% TRR being extracted after cellulase incubation for 24 hours at 37 °C in the phenyl-label study and in the THP-label study, more aggressive digestion and fractionation was able to show that 12–21% TRR was present in cellulose, hemicellulose and starch fractions and 3–9% TRR was found in the protein, lignin and pectin fractions.

HPLC analysis of the acetone:water extracts containing more than 0.01 mg/kg TRR from the phenyl-label study identified the presence of flumioxazin and the metabolites 482-HA, IMOXA, and 482-CA, with wheat straw also containing low levels of 1-OH-SAT-482, 1-OH-HPA, THPA, and TPA, all at < 0.01 mg/kg eq. Flumioxazin residues above 0.01 mg/kg were only found in wheat straw (0.03 mg/kg) from the 120-day plant-back treatment.

Table 22 Characterisation and identification of radioactive residues in rotational crops planted after soil application of 0.21 kg ai/ha [^{14}C-THP]flumioxazin

Matrix	PBI	Treatment (kg ai/ha)	Hvst DAT	TRR (mg/kg eq)	Component					
					mg/kg (%TRR)					
					Polar	Flumioxazin	482-HA	IMOXA	482-CA	Others
Wheat straw	30	0.11	159	0.03	0.015 (50%)	0.002 (5.9%)	< 0.001 (2.5%)	< 0.001 (0.87%)	–	–
Wheat chaff	30	0.11	159	0.012	0.007 (59.6%)	< 0.001 (2.3%)	< 0.001 (1.4%)	–	–	–
Carrot foliage	30	0.11	175	0.017	0.008 (48.6%)	0.003 (17.5%)	< 0.001 (5.5%)	–	< 0.001 (2.1%)	–
Wheat straw	30	0.21	159	0.034	0.012 (34%)	0.003 (8.6%)	0.001 (3.3%)	< 0.001 (1.6%)	< 0.001 (1.6%)	–
	120	0.21	253	0.08	0.012 (15.2%)	0.033 (40.7%)	< 0.001 (2.2%)	–	–	[a]
	180	0.21	308	0.027	0.002 (6.4%)	0.009 (35.1%)	–	–	–	[b]

Matrix	PBI	Treatment (kg ai/ha)	Hvst DAT	TRR (mg/kg eq)	Component					
					mg/kg (%TRR)					
					Polar	Flumioxazin	482-HA	IMOXA	482-CA	Others
	365	0.21	494	0.04	0.005 (13.2%)	0.007 (16.3%)	–	–	–	
Wheat chaff	30	0.21	159	0.012	0.007 (54.9%)	< 0.001 (0.99%)	< 0.001 (3.7%)	–	–	–
	120	0.21	253	0.026	0.015 (56.6%)	0.002 (8.8%)	0.002 (6.1%)	< 0.001 (0.56%)	< 0.001 (2.1%)	–
	180	0.21	308	0.011	0.005 (49.2%)	0.002 (10.7%)	< 0.001 (5.3%)	< 0.001 (1.9%)	–	–
	365	0.21	494	0.011	0.005 (41.1%)	0.001 (20.3%)	–	–	–	–
Carrot foliage	60	0.21	175	0.022	0.013 (58.5%)	0.002 (10.4%)	0.001 (6.6%)	< 0.001 (0.48%)	< 0.001 (1.7%)	
	120	0.21	253	0.016	0.006 (39.2%)	0.007 (42.8%)	< 0.001 (1.7%)	< 0.001 (1.6%)	< 0.001 (0.84%)	–
Carrot roots	60	0.21	175	0.013	0.007 (57.3%)	0.005 (38.8%)	< 0.001 (1.2%)	< 0.001 (0.66%)	–	–

[a] 1-OH-SAT (0.008 mg/kg eq, 9.7% TRR)

[b] 1-OH-HPA (0.004 mg/kg eq, 13.4% TRR)

THPA (0.004 mg/kg eq, 15.3% TRR)

TPA (0.004 mg/kg eq, 15.2% TRR)

Radioactive residues in rotational crops planted 30–365 days after bare soil treatments with [^{14}C]flumioxazin were low, generally less than 0.01 mg/kg and less than 0.05 mg/kg in all matrices except wheat straw, where up to 0.13 mg/kg were found in the THP-label study. The only significant residue identified in rotated crop matrices above 0.01 mg/kg was the parent, flumioxazin, in wheat straw (0.013 mg/kg). Low levels of 482-HA, IMOXA and 482-CA were found in most crop matrices, up to 6.6% TRR (< 0.002 mg/kg) with wheat straw also containing low levels of 1-OH-SAT-482, 1-OH-HPA, THPA, and TPA, all < 0.01 mg/kg.

Animal metabolism

The Meeting received animal metabolism studies on rats, lactating goats and laying hens where animals were dosed with flumioxazin radio-labelled in the phenyl ring or the tetrahydrophthaloyl (THP) ring.

Rats

The metabolism of flumioxazin in rats was evaluated by the WHO Core Assessment Group of the 2015 JMPR. Studies were carried out to investigate the metabolism of phenyl-label and THP-label flumioxazin in rats. Excretion of radioactivity was rapid, with 69–87% being eliminated in urine and faeces within 24 hours with the reminder found mainly in excretory organs. Flumioxazin was extensively metabolized (29–35 metabolites detected and quantified), with 7–10 of these being identified. Flumioxazin accounted for 47–66% of the administered dose in the 100 mg/kg bw and 0.3–2% in the 1 mg/kg bw dose group. Metabolites found at more than 5% of the administered dose were

3-OH-flumioxazin, 3-OH-flumioxazin-SA, 4-OH-flumioxazin and 4-OH-flumioxazin-SA. The proposed metabolic pathways included hydroxylation of the cyclohexene ring, cleavage of the imide linkage, cleavage of the amide linkage in the benzoxadine ring, reduction of the double bond in the THP ring, acetylation of the amino group of the aniline derivative and the addition of a sulphonic acid group to the THP ring.

Lactating goats

Two studies were carried out to investigate the absorption and deposition of phenyl-label and THP-label flumioxazin in lactating goats. In the first study, reported by Sharp, 1993 [Ref: SBM-0026], two lactating goats (average body-weight of 48 kg) were dosed orally for 5 days with capsules containing [^{14}C-phenyl] flumioxazin at the rate equivalent to 11.8 ppm in the diet (based on an average feed consumption of 2.1 kg/goat/day and a total dose of 2.63 mg/kg bw). Milk, urine and faeces were collected twice daily and liver, fat, muscle, blood, gastrointestinal tract and contents were collected at sacrifice, about 6 hours after the last dose.

The second study, reported by Panthani, 1994 [Ref: SBM-0040] used a similar protocol involving two goats (average body-weight of 45 kg) but with [^{14}C-THP] flumioxazin at a dose equivalent to 7.2 ppm in the diet (based on an average feed consumption of 2 kg/goat/day and a total dose of 1.44 mg/kg bw).

Radioactivity was quantified by LSC. Samples of liver, kidney, muscle, and fat were initially homogenized in dry ice, and then subjected to combustion/LSC.

The average total recoveries of radioactivity were 81% and 94% of the administered radioactivity (AR) in the phenyl-label and the THP-label studies respectively, mostly found in faeces, urine and the GI tract contents (80–93% AR). Tissues and milk contained relatively small amounts of radioactivity (< 1% and 0.22% AR respectively).

Average concentrations of radioactivity were low in muscle and fat, up to 0.014 mg/kg (phenyl-label) and 0.028 mg/kg (THP-label), but were higher in liver, up to 0.21 mg/kg (phenyl-label) and 0.33 mg/kg (THP-label). In kidney the radioactive residues were up to 0.18 mg/kg (phenyl-label) and 0.24 mg/kg (THP-label). The average total radioactivity concentration in milk plateaued around Day 3 at about 0.03 mg/kg (phenyl-label) and about 0.06 mg/kg in the THP-label study.

Table 23 Distribution of radioactive residues in tissues, excreta and milk of lactating goats following 5 daily doses of [^{14}C]flumioxazin

MATRIX	RADIOACTIVE RESIDUES (MG FLUMIOXIN EQUIVALENTS/KG)							
	[^{14}C-PHENYL] FLUMIOXAZIN (11.8 PPM IN THE DIET)				[^{14}C-THP] FLUMIOXAZIN (7.2 PPM IN THE DIET)			
	GOAT 2		GOAT 3		GOAT 500090		GOAT 500092	
	%AD	MG/KG	%AD	MG/KG	%AD	MG/KG	%AD	MG/KG
Fat (omental)	< 0.01	0.006	< 0.01	0.005	0.01	0.006	0.01	0.01
Fat (perirenal)	< 0.01	0.006	< 0.01	0.004	0.01	0.008	0.01	0.008
Kidneys	0.02	0.182	0.01	0.11	0.05	0.189	0.04	0.238
Liver	0.19	0.209	0.12	0.165	0.44	0.286	0.40	0.33
Muscle (rear leg)	0.01	0.014	0.01	0.013	0.02	0.023	0.02	0.028
Muscle (loin)	0.01	0.014	0.01	0.012	0.02	0.022	0.03	0.025
Total tissues	0.25	0.43	0.17	0.31	0.55	0.53	0.51	0.64
Milk day 1 (pm)		0.019		0.023		0.033		0.049
Milk day 2 (am)		0.005		0.005		0.005		0.010
Milk day 2 (pm)		0.023		0.026		0.041		0.053
Milk day 3 (am)		0.007		0.007		0.007		0.009
Milk day 3 (pm)		0.026		0.032		0.042		0.046
Milk day 4 (am)		0.007		0.007		0.007		0.012

MATRIX	RADIOACTIVE RESIDUES (MG FLUMIOXIN EQUIVALENTS/KG)							
	[^{14}C-PHENYL] FLUMIOXAZIN (11.8 PPM IN THE DIET)				[^{14}C-THP] FLUMIOXAZIN (7.2 PPM IN THE DIET)			
	GOAT 2		GOAT 3		GOAT 500090		GOAT 500092	
	%AD	MG/KG	%AD	MG/KG	%AD	MG/KG	%AD	MG/KG
Milk day 4 (pm)		0.025		0.03		0.046		0.055
Milk day 5 (am)		0.007		0.006		0.006		0.011
Milk day 5 (pm)		0.028		0.031		0.043		0.05
Total milk	0.05		0.17		0.22		0.2	
Blood	< 0.01	0.019	< 0.01	0.025	–	0.061	–	0.068
Urine	14.5		15.4		33.8		27.1	
Faeces	50.3		50.2		44.6		45.5	
GI tract	15.0 [a]	2.01	15.0 [a]	2.26	14.9 [a]		18.8 [a]	
Pan rinse	0.08		0.29		0.45		0.61	
Total	80.2		81.2		94. 5		92.7	

%AD = % administered dose

[a] Includes GI tract contents

In the first study, milk and tissue samples (except fat) from goats dosed with the phenyl-label were solvent-extracted and subjected to protease digestion (liver and kidney) to characterize and identify residues. Milk samples were extracted with hexane and the extracted residues were triple-extracted into methanol, filtered, evaporated and redissolved in methanol for LSC and HPLC analysis. Muscle samples were extracted with acetonitrile and after evaporation and reconstitution in methylene chloride (to solubilise the lipids), the supernatants were partitioned between acetonitrile and hexane for analysis. The remaining solid phases from the acetonitrile extractions were also subjected to an additional sodium bicarbonate extraction step and aliquots were subjected to overnight enzyme digestion (protease) prior to sequential extraction with water, methanol and acetonitrile and analysis.

In the second study, milk and tissue samples (except fat) from goats dosed with the THP-label were solvent-extracted to characterize and identify residues. Milk samples were mixed with ethanol, filtered, concentrated and partitioned with hexane. The aqueous phases were concentrated, additional ethanol was added, and after centrifuging, the supernatants were concentrated for TLC and HPLC analysis. Tissue samples were extracted with acetonitrile and acetonitrile:water with 1%HOAc (1:1), filtered and the supernatants were concentrated for TLC and HPLC analysis. The solids fractions were dried and combusted to quantitate the unextracted radioactivity.

About 80–94% TRR in milk was able to be extracted with methanol or ethanol, and acetonitrile was able to extract 58–74% TRR from muscle (45–53% in the THP-label samples, with a further 30% extracted in acetonitrile:water). In the phenyl-label liver and kidney samples, sequential extractions with acetonitrile and bicarbonate were able to extract more than 90% TRR and further enzyme extraction released an additional 10% TRR. In the THP-label liver and kidney samples, sequential acetonitrile and acetonitrile:water extractions were able to extract 80–87% TRR.

Table 24 Total radioactive residues recovered in tissues and milk of lactating goats following five daily doses of [^{14}C]flumioxazin

EXTRACT	LIVER		KIDNEY		LOIN MUSCLE		REAR LEG MUSCLE		MILK	
	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
Phenyl-label (11.8 ppm in the diet)										
Acetonitrile	42.3	0.88	55.8	0.1	73.7	0.009	58.3	0.008		
Methanol									80.4	0.02
Bicarbonate	47.7	0.1	44.3	0.081						

EXTRACT	LIVER		KIDNEY		LOIN MUSCLE		REAR LEG MUSCLE		MILK	
	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
Protease	9.2	0.019	11.2	0.02						
Post-extraction solids	5.1	0.011	4.5	0.008	37.8	0.005	49.6	0.006	10.2	0.003
%TRR	104		116		112		108		90.6	
THP-label (7.2 ppm in the diet)										
Acetonitrile	55.7	0.159	49.9	0.094	52.9	0.012	45.3	0.011		
Ethanol									94.1	0.024
Acetonitrile:water	31.1	0.089	29.7	0.056	20.4	0.005	26.9	0.006		
Post-extraction solids	7.6	0.022	11.7	0.022	19.5	0.004	21.4	0.005	11.4	0.003
%TRR	94		91		93		94		105	

Metabolites were characterized and identified following sample extraction and co-chromatography with known reference materials using thin-layer chromatography and HPLC with uv detection high-performance liquid chromatography within 4–6 months of sampling. Concurrent analysis of stored analytical samples indicated that residues were stable over the storage intervals in the studies.

Flumioxazin was extensively metabolized, with residues above 0.001 mg/kg found only in liver (up to 0.01 mg/kg and < 5% TRR).

The only identified metabolite present at more than 10% TRR was the 4-OH-flumioxazin, accounting for up to 14% TRR in kidney (up to 0.025 mg/kg) and muscle (up to 0.003 mg/kg). In liver, 4-OH-flumioxazin residues did not exceed 0.025 mg/kg (9.4% TRR) and were up to 0.002 mg/kg (8.6% TRR) in milk.

Other identified metabolites found at more than 5% TRR were 482-HA, found in liver and kidney (close to 10% TRR, 0.02 mg/kg), 3-OH-flumioxazin in liver (up to 8.6% TRR, 0.023 mg/kg) and kidney (up to 6% TRR, 0.011 mg/kg), APF in kidney (5.8% TRR, 0.011 mg/kg) and SAT-482 in liver and kidney (5–6% TRR, up to 0.013 mg/kg).

In kidney, metabolite B, tentatively identified as 3- or 4-OH-SAT-482, made up about 14% TRR (0.024 mg/kg) and residues of metabolite C was measured at 0.015 mg/kg (8.5% TRR).

In liver, metabolite F, tentatively identified as an isomer of 3- or 4-OH-SAT-482, made up about 11% TRR (0.03 mg/kg) and residues of metabolite D were measured at 0.013 mg/kg (4.9% TRR).

In muscle, metabolite C accounted for 20-23% TRR and in milk, metabolites B and C were found at 12–18% TRR. However, absolute levels were all 0.005 mg/kg or less.

Table 25 Characterisation and identification of radioactive residues in goat tissues and milk following five daily doses of [^{14}C-phenyl]-flumioxazin (11.8 ppm in the diet)

METABOLITE	LIVER		KIDNEY		LOIN MUSCLE		REAR LEG MUSCLE		MILK	
	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
Flumioxazin	4.7	0.01	0.2	< 0.001	1.2	< 0.001	0.7	< 0.001	ND	< 0.001
3-OH-flumioxazin	4.2	0.009	6.2	0.011	1.2	< 0.001	ND	< 0.001	1.8	< 0.001
4-OH-flumioxazin	6.5	0.014	13.7	0.025	1.6	< 0.001	2.7	< 0.001	1.5	< 0.001
3-OH-flumioxazin-SA+ 4-OH-flumioxazin-SA	1.8	0.004	ND	< 0.001	ND	< 0.001	ND	< 0.001	6.5	0.002
482-HA	9.8	0.02	8.7	0.016	4.2	< 0.001	5.1	< 0.001	14.4	0.004
APF	3.8	0.008	5.8	0.011	3.5	< 0.001	ND	< 0.001	0.2	< 0.001
Maximum single other metabolite	7.6	0.016	18.1	0.033	7.4	0.001	13.9	0.002	11.5	0.003
Total identified	30.8		34.6		11.7		8.5		24.4	

ND = non detectable

Table 26 Characterisation and identification of radioactive residues in goat tissues and milk following five daily doses of [^{14}C-THP]-flumioxazin (7.2 ppm in the diet)

METABOLITES	LIVER		KIDNEY		LOIN MUSCLE		REAR LEG MUSCLE		MILK	
	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG	%TRR	MG/KG
Flumioxazin	1.4	0.004	–	–	1.4	0.0003	1.8	0.0004	–	–
3-OH-flumioxazin	8.6	0.023	4.5	0.008	–	–	–	–	–	–
4-OH-flumioxazin	9.4	0.025	7.9	0.014	11.7	0.002	13.2	0.003	8.6	0.002
4-OH-THPA	0.9	0.003	3.8	0.007	6.9	0.001	6.8	0.002	6.0	0.002
SAT-482	4.7	0.013	5.5	0.01	–	–	–	–	–	–
THPA	3.2	0.009	1.2	0.002	–	–	–	–	–	–
Metabolite B	1.6	0.004	14.0	0.024	–	–	–	–	17.9	0.005
Metabolite C	–	–	8.5	0.015	23.3	0.005	19.6	0.004	12.3	0.003
Metabolite D	4.9	0.013	3.7	0.006	–	–	–	–	1.5	0.0004
Metabolite E	1.5	0.004	0.9	0.002	–	–	–	–	–	–
Metabolite F	11.4	0.031	–	–	–	–	–	–	–	–
Unknowns	38.2	0.103	26.3	0.057	28.5	0.006	30.5	0.007	39.5	0.011
Maximum single other metabolite	8.5	0.023	8.5	0.015	19.4	0.004	19.6	0.004	11.2	0.003
Nonextractable	8.0	0.022	12.8	0.022	20.8	0.004	22.8	0.005	10.8	0.003

The major metabolic pathways proposed for flumioxazin in goats include: the hydroxylation of the parent to 3-OH-flumioxazin and the subsequent incorporation of a sulfonic group to form 3-OH-flumioxazin-SA; the reduction of the parent molecule and subsequent hydroxylation to SAT-482; and the cleavage of the imide and amide linkages of the parent molecule to THPA and APF.

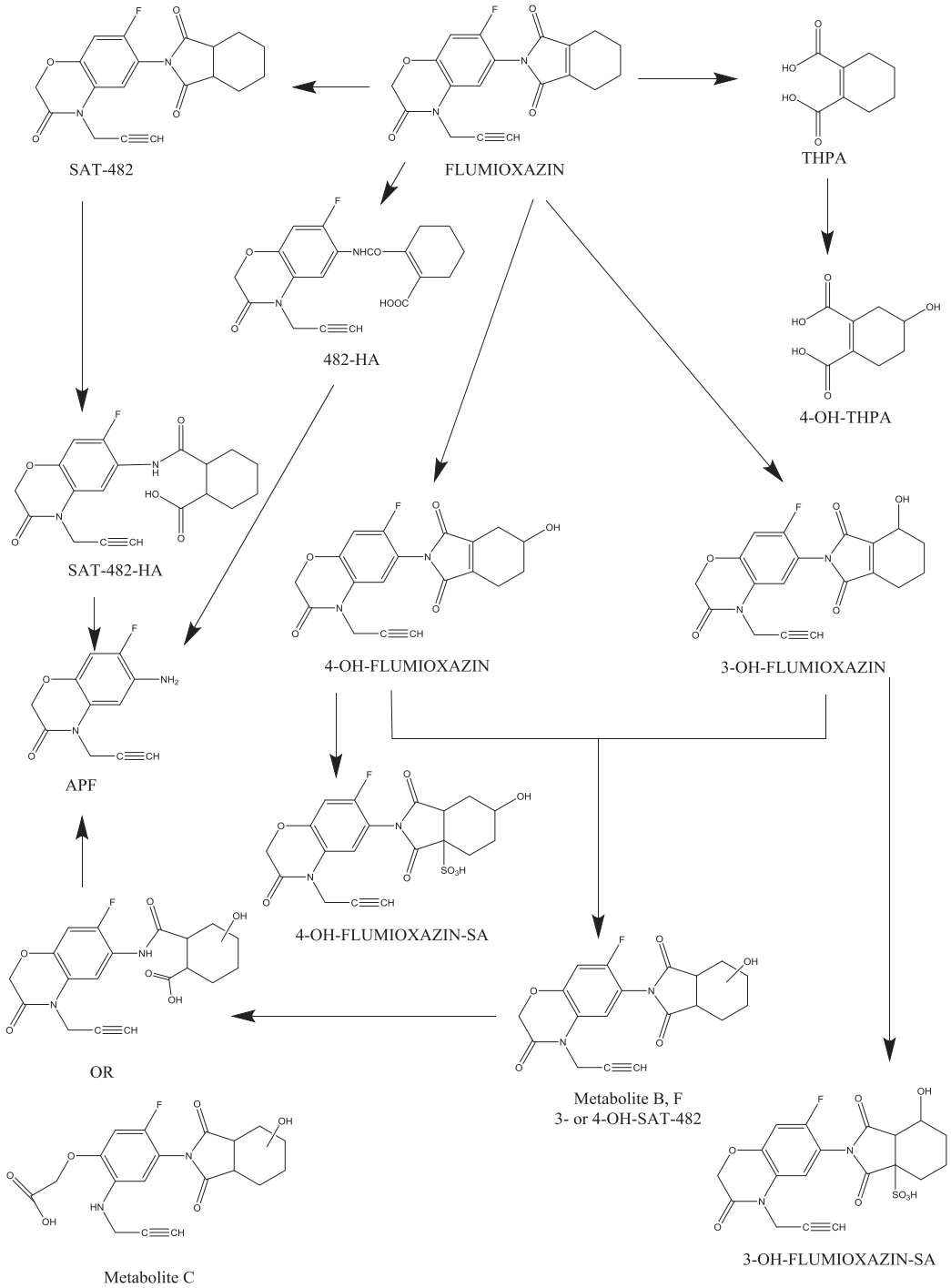


Fig 4 Metabolic Pathway of Flumioxazin in Lactating Goats

Laying Hens

Two studies were carried out to investigate the absorption and deposition of phenyl-label and THP-label flumioxazin in laying hens. In the first study, reported by Sharp, 1993 [Ref: SBM-0027], ten laying hens (average body-weight of 1.65 kg) were dosed orally for 14 days with capsules containing [^{14}C-phenyl] flumioxazin at the rate equivalent to 10 ppm in the diet (based on an average feed consumption of 0.122 kg/hen/day and an average daily dose of 0.683 mg/kg bw/day).

The second study, reported by Panthani, 1994 [Ref: SBM-0039] used a similar protocol involving 10 hens (1.3–1.9 kg bodyweight) and [^{14}C-THP] flumioxazin at a dose equivalent to 10 ppm in the diet (based on an average feed consumption of 0.127 kg/hen/day).

Eggs were collected twice daily. Eggs collected on the same day were pooled and then separated into yolks and whites. Samples of excreta were collected daily. The hens were sacrificed 4 hours after the last dose and the following samples were collected: kidney, heart, liver, muscle (breast and thigh), abdominal fat, skin with fat, gizzard, reproductive organs, and GI tract and contents.

Samples of kidney, liver, muscle, fat, and skin with fat were homogenized in dry ice, and then subjected to combustion/LSC. Egg yolk and white samples were blended and then subjected to combustion/LSC. Samples of excreta and cage washings were also collected and were analysed for TRR.

The average total recoveries of radioactivity in the samples collected for analysis from the treated animals were 95% of the administered dose (phenyl-label study) and 87% in the THP-label study. Most of the radioactivity was found in the excreta, GI tract contents and cage wash, which, together accounted for 94% and 83% of the doses in the respective studies. Liver, kidney, muscle, fat, skin and eggs contained relatively small amounts of radioactivity (totalling < 0.6% and < 0.9% of the administered dose, respectively).

Radioactivity in egg yolks accounted for 0.35–0.36% AR, with < 0.01% AR in the corresponding egg whites and liver contained 0.08% AR in the phenyl-label study and 0.27% AR in the THP-label study (0.24 mg/kg eq and 1.14 mg/kg eq respectively. In egg yolks, residues reached a plateau of 0.4–0.6 mg/kg eq by Day 10 or 11 in the two studies.

Table 27 Distribution of radioactive residues in tissues, excreta and eggs of laying hens following 14 daily doses of [^{14}C]flumioxazin

MATRIX	RADIOACTIVE RESIDUES (MG FLUMIOXIN EQUIVALENTS/KG)					
	[^{14}C-PHENYL] FLUMIOXAZIN (10 PPM IN THE DIET)			[^{14}C-THP] FLUMIOXAZIN (10 PPM IN THE DIET)		
	% ADMINISTERED DOSE	MG/KG		% ADMINISTERED DOSE	MG/KG	
Liver	0.08	0.237		0.27	1.137	
Kidney	0.02	0.272		0.06	0.887	
Breast muscle	0.04	0.040		0.05	0.138	
Thigh muscle	0.03	0.050		0.06	0.175	
Fat	0.02	0.074		0.01	0.226	
Skin with fat	0.04	0.143		0.02	0.667	
Total tissues	0.23			0.47		
Eggs		Yolk	White		Yolk	White
Day 1		ND	ND		0.009	0.029
Day 2		0.01	0.017		0.034	0.033
Day 3		0.036	0.012		0.119	0.025
Day 4		0.099	0.015		0.154	0.041
Day 5		0.178	0.018		0.240	0.037
Day 6		0.237	0.017		0.338	0.03
Day 7		0.323	0.018		0.414	0.036
Day 8		0.349	0.015		0.467	0.034
Day 9		0.407	0.01		0.531	0.03
Day 10		0.425	0.008		0.57	0.036
Day 11		0.437	0.008		0.638	0.027
Day 12		0.422	0.01		0.64	0.025
Day 13		0.409	0.005		0.63	0.024

MATRIX	RADIOACTIVE RESIDUES (MG FLUMIOXIN EQUIVALENTS/KG)					
	[^{14}C-PHENYL] FLUMIOXAZIN (10 PPM IN THE DIET)			[^{14}C-THP] FLUMIOXAZIN (10 PPM IN THE DIET)		
	% ADMINISTERED DOSE	MG/KG		% ADMINISTERED DOSE	MG/KG	
Day 14		0.382	0.007		0.76	0.032
Total eggs	0.35			0.43		
Heart	< 0.01	0.161		0.04	0.761	
Gizzard	0.02	0.104		1.14	5.253	
GI tract & contents	1.43 [a]	0.62		4.67 [a]	6.018 [a]	
Blood	0.03	0.603		1.53	1.326	
Cage wash	0.5	–		2.89	–	
Reproductive organs	0.23	0.25		0.35	0.483	
Excreta	92.1	–		75.36	–	
Total	94.9			86.87		

[a] Includes GI tract contents

Egg yolk, egg white, and tissues samples were extracted with various solvents and the solvent-extracted radio-labelled residues were analysed by HPLC and TLC to characterize and identify the major metabolites. Identification was made by co-chromatography of extracts with known standards. The unextracted radioactive residues were characterized by acid or base hydrolysis, or by enzyme digestion. Additional metabolites were isolated from excreta extracts for mass spectral analysis to confirm the identity of the structures of the metabolites.

In the first study, egg (Day 7 and 13) and tissue samples from hens dosed with the phenyl-label were solvent-extracted and subjected to enzyme digestion to characterize and identify residues. Egg yolk samples were extracted with acetonitrile, the supernatant partitioned with hexane and the remaining residue subjected to enzyme (lipase) digestion and sodium bicarbonate extraction of the insoluble fraction with sequential extractions/elutions in hexane, methanol and acetonitrile. Egg white samples were extracted in acetonitrile. Liver samples were extracted with acetonitrile, the supernatant partitioned between acetonitrile and hexane and the remaining residue further extracted with sodium bicarbonate and subjected to enzyme (protease) digestion with the various fractions being sequentially extracted or eluted with water, methanol and acetonitrile. Kidney and muscle samples were extracted with acetonitrile, the supernatants partitioned between acetonitrile and hexane and the remaining residue further extracted with water (except breast muscle) and subjected to enzyme (protease) digestion and acid hydrolysis (6 N HCl), with the various fractions being sequentially extracted or eluted with water, hexane, methanol, dichloromethane and acetonitrile. Skin + fat and fat samples were extracted with chloroform:methanol (2:1) with the chloroform phase being partitioned between acetonitrile and hexane. The remaining residue from the skin + fat samples were subjected to enzyme (protease) digestion with the various fractions being sequentially extracted or eluted with water, methanol and acetonitrile.

In the second study, egg (Day 13) and tissue samples from hens dosed with the THP-label were extracted with acetonitrile and acetonitrile:water with 1% HOAc (1:1), filtered and the supernatants were concentrated for TLC and HPLC analysis. The post-extraction solids were also subjected to pronase hydrolysis. The remaining solids fractions were dried and combusted to quantitate the unextracted radioactivity.

In the THP-label and the phenyl-label studies respectively, highest concentrations of radioactivity were in liver (1.1 mg/kg and 0.24 mg/kg) and kidney (0.89 mg/kg and 0.27 mg/kg) with lower levels in egg yolks (0.63 mg/kg and 0.41 mg/kg). Radioactivity in fat and skin + fat were 0.23–0.67 mg/kg (THP-label) and 0.07–0.14 mg/kg (phenyl-label) respectively. In muscle, radioactive residues were 0.14–0.18 mg/kg (THP-label) and 0.04–0.05 mg/kg (phenyl-label) and egg white contained about 0.02 mg/kg in both studies.

More than 87% TRR in eggs was able to be extracted, and acetonitrile was able to extract 37–67% TRR from muscle. In the phenyl-label liver and kidney samples, sequential extractions with acetonitrile and bicarbonate were able to extract more than 60% TRR and further enzyme extraction released an additional 30% TRR. In the THP-label liver and kidney samples, sequential acetonitrile and acetonitrile:water extractions were able to extract 75–78% TRR. In fat and fat + skin, extraction efficiencies were 76–91% TRR and 54–95% respectively.

Table 28 Total radioactive residues recovered in tissues and eggs of laying hens following 14 daily doses of [^{14}C]flumioxazin (10 ppm in the diet)

EXTRACT	EGG WHITE	EGG YOLK	LIVER	KIDNEY	THIGH	BREAST	FAT	SKIN + FAT
	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)
Phenyl-label								
TRR (mg/kg)	0.018	0.409	0.237	0.272	0.05	0.04	0.074	0.143
Acetonitrile	100.0 (0.018)	42.9 (0.175)	45.9 (0.109)	53.0 (0.144)	36.7 (0.018)	42.3 (0.017)		
Water				13.6 (0.037)				
Bicarbonate		10.6 (0.043)	14.9 (0.035)					
Enzyme		40.3 (0.165)	31.3 (0.074)	20.1 (0.055)	39.4 (0.02)	27.2 (0.011)		13.7 (0.02)
Acid hydrolysate				9.8 (0.027)	14.8 (0.007)	29.8 (0.012)		
MeOH/$CHCl_3$ (organic)							54.0 (0.040)	48.7 (0.07)
MeOH/$CHCl_3$ (aqueous)							21.5 (0.016)	32.9 (0.047)
%Total extracted	100	93.8	92.1	96.5	90.9	99.3	75.5	95.3
Post-extraction solids	10.0 (0.002)	4.8 (0.02)	11.1 (0.026)	2.7 (0.007)	5.2 (0.003)	6.6 (0.003)	9.9 (0.007)	0.81 (0.001)
%TRR	110	98.6	103	99.2	96.1	106	85.4	96.1
THP-label								
TRR (mg/kg)	0.024	0.63	1.137	0.887	0.175	0.138	0.226	0.667
Acetonitrile	79.4 (0.017)	20.0 (0.184)	48.9 (0.45)	52.6 (0.101)	55.7 (0.101)	62.9 (0.086)	69.0 (0.179)	36.6 (0.229)
Acetonitrile:water	13.2 0.003	64.5 (0.515)	28.9 (0.325)	22.2 (0.19)	9.0 (0.016)	4.2 (0.006)	22.0 (0.057)	17.8 (0.111)
%Total extracted	92.6	87.5	77.8	74.8	64.7	67.0	91.0	54.4
Post-extraction solids	7.4 (0.002	12.5 (0.1)	22.2 (0.25)	25.2 (0.217)	35.3 (0.064)	33.0 (0.045)	9.0 (0.023)	45.5 (0.284)
%TRR	87.5	126.8	99.1	96.7	103.6	98.8	114.9	93.5

Values reported for eggs are from Day 13 Samples

Metabolites were characterized and identified following solvent extraction and co-chromatography with known reference materials using thin-layer chromatography and HPLC with uv detection high-performance liquid chromatography within 4.5 months of sampling. Concurrent analysis of stored analytical samples indicated that residues were stable (more than 93% recovery from spiked samples) over the storage intervals in the studies.

In the phenyl-label study, flumioxazin was the predominant residue in fat (49% TRR), skin + fat (25% TRR), muscle (10–14% TRR), liver (9.1% TRR) and kidney (6.9% TRR), made up about 3.8% TRR in egg yolk and was not detected in egg white. Absolute levels of

flumioxazin were < 0.05 mg/kg in skin + fat and fat, < 0.02 mg/kg in liver, kidney and egg yolk and about 0.005 mg/kg in muscle.

The major identified metabolites, present at more than 10% TRR were APF and 482-HA. The APF metabolite accounted for 20% TRR in egg white and 10% TRR in muscle (but both at absolute levels of < 0.005 mg/kg) and 482-HA made up about 20% of the TRR in egg white. All other identified metabolites were found at < 8% TRR and the highest level of any single unidentified metabolite was measured in liver, at 12% TRR.

Table 29 Characterisation and identification of radioactive residues in hen tissues and eggs following 14 daily doses of [^{14}C-phenyl]-flumioxazin (10 ppm in the diet)

METABOLITE	EGG WHITE	EGG YOLK	LIVER	KIDNEY	THIGH	BREAST	FAT	SKIN + FAT
	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)
Flumioxazin	ND	3.8 (0.016)	9.1 (0.022)	6.9 (0.019)	9.9 (0.005)	13.9 (0.006)	48.8 (0.046)	24.7 (0.035)
3-OH- flumioxazin SA	ND	0.2 (ND)	0.7 (0.002)	1.3 (0.004)	0.7 (ND)	0.5 (ND)	1.2 (ND)	0.3 (ND)
4-OH-flumioxazin SA	ND	0.1 (ND)	ND	1.4 (0.004)	3.3 (0.002)	0.6 (ND)	ND	ND
482-HA	20.0 (0.004)	0.6 (0.002)	1.2 (0.003)	0.1 (ND)	5.5 (0.003)	1.2 (ND)	ND	6.9 (0.01)
APF	23.2 (0.004)	3.5 (0.015)	3.1 (0.007)	4.8 (0.013)	7.7 (0.004)	10.4 (0.004)	ND	1.1 (0.001)
4-OH flumioxazin	ND	1.1 (0.004)	3.9 (0.009)	7.2 (0.02)	6.8 (0.003)	7.7 (0.003)	3.7 (0.003)	1.6 (0.002)
3-OH flumioxazin	ND	0.5 (0.002)	2.6 (0.006)	3.1 (0.008)	5.6 (0.003)	6.7 (0.003)	1.0 (ND)	2.6 (0.004)
Maximum other single metabolite	8.4	4.3	11.9	5.0	8.2	5.1	2.3	10.3
Total of identified metabolites	43.2 (0.008)	9.8 (0.039)	20.6 (0.049)	24.8 0.068)	39.5 (0.021)	41.0 (0.016)	54.7 (0.049)	37.2 (0.052)

ND = non detectable

Values reported for eggs are from Day 13 Samples

In the THP-label study, flumioxazin was the predominant residue in fat (49% TRR), skin + fat (12% TRR) and muscle (11% TRR) and was found at 7% TRR in liver, kidney and 9% TRR in egg yolk. Absolute levels of flumioxazin were 0.07–0.13 mg/kg in skin + fat and fat, < 0.08 mg/kg in liver and kidney, < 0.04 mg/kg in egg yolk and < 0.02 mg/kg in muscle.

The major identified metabolites, present at more than 10% TRR in tissues were 4-OH-flumioxazin, 3-OH-flumioxazin and 4-OH-THPA. The 4-OH-flumioxazin accounted for 9–12% TRR in all tissues (< 0.03 mg/kg in muscle and fat, < 0.08 mg/kg in kidney and skin + fat, 0.12 mg/kg in liver) while the 3-OH-flumioxazin accounted for 8–12% TRR (0.015 mg/kg) in muscle. The 4-OH-THPA metabolite made up 10% TRR (0.09 mg/kg) in kidney.

In eggs, metabolites present at more than 10% TRR were 4-OH-flumioxazin-SA in egg yolk (32% TRR, 0.14 mg/kg) and in egg white, THPA and 4-OH-THPA each accounted for 23–26% TRR and (but < 0.01 mg/kg), with TPA and 3-OH-THPA each present at 16–17% TRR. Absolute levels of these metabolites in egg white were all < 0.01 mg/kg.

Table 30 Characterisation and identification of radioactive residues in hen tissues and eggs following 14 daily doses of [^{14}C-THP]-flumioxazin (10 ppm in the diet)

METABOLITE	EGG WHITE	EGG YOLK	LIVER	KIDNEY	THIGH	BREAST	FAT	SKIN + FAT

	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)	%TRR (MG/KG)
Flumioxazin	0.65 (0.000)	8.9 (0.039)	6.7 (0.076)	7.2 (0.062)	11.0 (0.020)	10.8 (0.015)	49.0 (0.128)	11.6 (0.072)
THPA	22.7 (0.009)	6.8 (0.029)	9.7 (0.109)	9.8 (0.084)	9.4 (0.017)	8.4 (0.011)	2.3 (0.006)	4.5 (0.028)
TPA	16.4 (0.006)	ND	ND	ND	ND	ND	ND	ND
3-OH-flumioxazin-SA	ND	1.1 (0.005)	6.0 [b] (0.067)	4.3 [b] (0.037)	ND	ND	ND	ND
4-OH-flumioxazin-SA	ND	31.8 (0.139)			ND	ND	ND	ND
4-OH-flumioxazin	ND	5.4 (0.024)	10.8 (0.121)	8.7 (0.075)	10.2 (0.018)	12.3 (0.016)	11.4 (0.030)	10.9 (0.068)
3-OH-flumioxazin	ND	3.6 (0.016)	7.0 (0.079)	7.1 (0.061)	7.7 (0.014)	11.7 (0.016)	9.6 (0.025)	6.4 (0.040)
4-OH-THPA	25.8 (0.009)	7.8 (0.034)	4.4 (0.05)	10.3 (0.088)	7.0 (0.013)	6.4 (0.009)	2.9 (0.008)	3.2 (0.020)
3-OH-THPA	16.7 (0.006)	ND	ND	ND	ND	ND	ND	ND
OH-flumioxazin [a]	0.47 (0.000)	4.9 (0.021)	2.7 (0.030)	3.2 (0.027)	3.7 (0.007)	3.4 (0.004)	5.0 (0.013)	3.3 (0.020)
Maximum single other metabolite	< 1%	< 5%	< 5%	< 5%	< 5%	< 5%	< 5%	–
Unknown	11.1 (0.004)	19.4 (0.085)	29.8 (0.336)	23.4 (0.201)	14.6 (0.026)	13.1 (0.018)	10.0 (0.026)	13.8 (0.087)

Values reported for eggs are from Day 7 Samples

[a] Exact position of hydroxylation not determined

[b] Mixture of two metabolites

The major metabolic pathways proposed for flumioxazin in hens include the hydroxylation of the parent and the subsequent incorporation of sulfonic groups to form 3-OH-flumioxazin-SA and 4-OH-flumioxazin-SA and the cleavage of the imide and amide linkages of the parent molecule to THPA and APF.

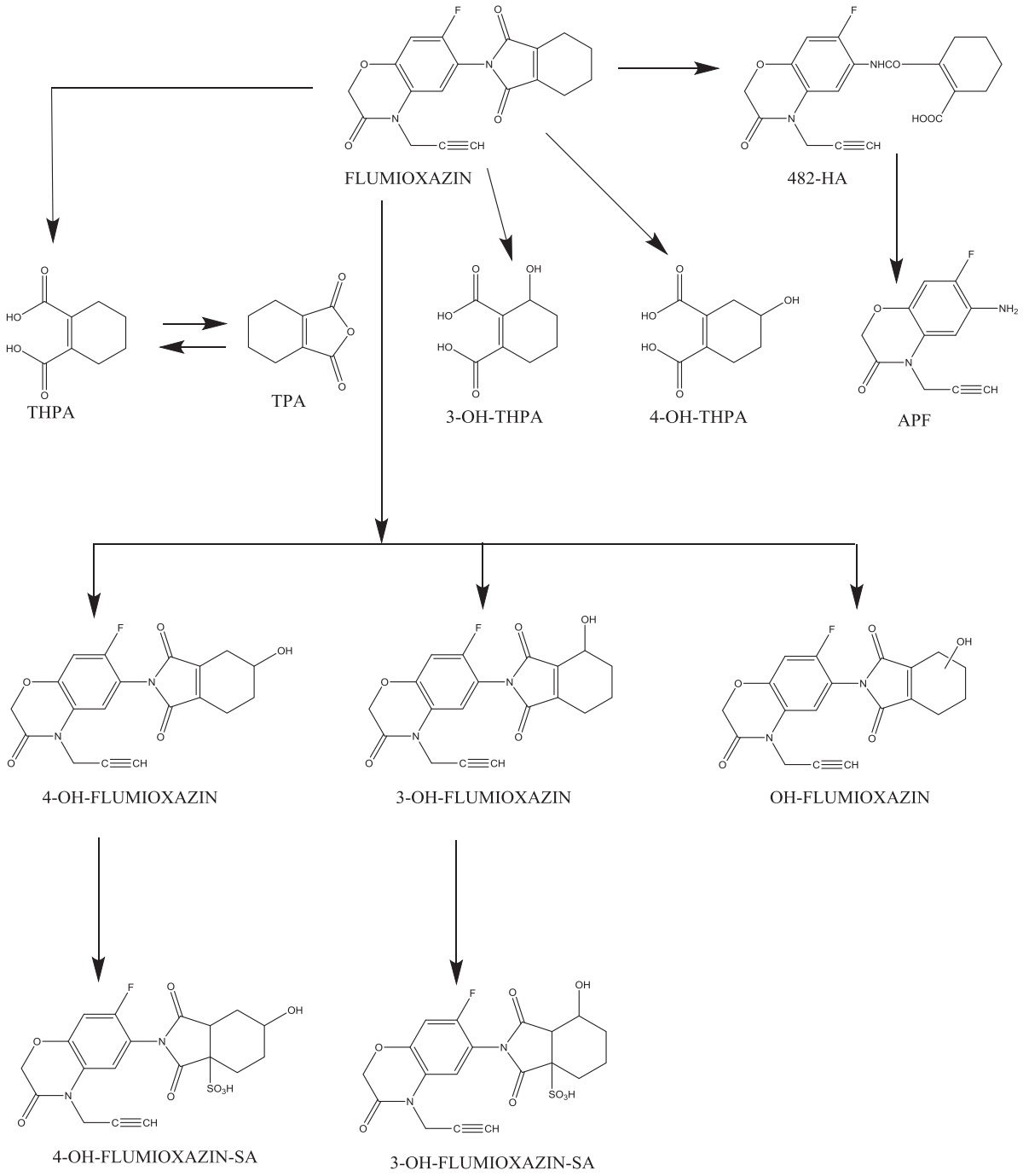


Fig 5 Metabolic Pathway of Flumioxazin in Poultry

Flumioxazin is extensively metabolized with limited absorption into tissues, eggs or milk (less than 0.5% of the administered dose). Flumioxazin was not found at levels above 0.01 mg/kg in goat milk or tissues and in poultry, highest residues found were in fat (0.13 mg/kg, 49% TRR), with lower levels (up to 0.08 mg/kg) in other tissues and egg yolks. The major identified metabolites found above 10% TRR and above 0.01 mg/kg in various matrices were 4-OH-flumioxazin, 4-OH-flumioxazin-SA, 3-OH-flumioxazin and 4-OH-THPA.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The Meeting received analytical method descriptions and validation data for flumioxazin in plant and animal matrices and these are summarized below.

Table 31 Summary of analytical methods for flumioxazin and its 1-OH-HPA metabolite, developed for plant and animal matrices

Matrix	Analyte	Method	Principle	LOQ (mg/kg)	Reference
Fresh and processed plant matrices	Flumioxazin	RM 30A (RM 30A-1) (RM 30A-2) (RM 30A-3)	Acetone/water extraction Dichloromethane partition Hexane/acetonitrile partition Florisil column clean-up GC-MS analysis * RM 30A-1 includes minor modifications to the sample grinding/preparation steps * RM 30A-2 includes minor equipment and text modifications * RM 30A-3 adds confirmatory GC/MS conditions	0.02	SBR-0003
Processed plant oils	Flumioxazin	RM 30B	Hexane/acetonitrile extraction Acetonitrile partition Florisil column clean-up GC-MS analysis	0.02	SBR0019
Eggs Animal tissues Milk	Flumioxazin	ER-MT-9403	Acetone extraction Hexane/acetonitrile partition Florisil column clean-up GC-MS analysis	0.02	SBA-0037
Eggs Animal tissues Milk	Flumioxazin 3-OH-flumioxazin 4-OH-flumioxazin	RM-30T RM-30MK	Acetonitrile & acetonitrile:water extraction (Acetone extraction for milk) Dichloromethane & hexane/acetonitrile partition HPLC-MS/MS analysis	0.02	SBR-0138
Animal feeds	1-OH-HPA including conjugates	RM 30M	Acid hydrolysis extraction Ethyl acetate partition Methylation (dimethyl sulphate) Water/hexane partition Florisil column clean-up CG-MSD analysis (1-OH-HPA-dimethyl ester)	0.02	SBR-0019
Processed plant oils	1-OH-HPA including conjugates	RM 30P	Acid hydrolysis and hexane extraction SPE (ethyl acetate) extraction Methylation (dimethyl sulphate) Water/hexane partition Florisil column clean-up CG-MSD analysis (1-OH-HPA-dimethyl ester)	0.02	SBR-0019

Data collection methods

Method RM 30A (Flumioxazin—fresh and processed plant matrices)

Method 30A, used with minor equipment modifications and sample preparation steps to measure residues of flumioxazin in fresh plant commodities and their processed fractions was first reported by Pensyl, 1992 [Ref: SBR-0003].

In this method, homogenised samples are double-extracted with acetone:water (4:1), partitioned into dichloromethane and after evaporation to dryness, dissolved in hexane:acetonitrile (30:1) then shaken with acetonitrile:hexane (5:1), After separation, the combined acetonitrile extracts are evaporated to dryness, redissolved in ethyl acetate and diluted with hexane and purified using Florisil columns eluted with hexane:ethyl acetate (2:1 v:v). Residues in the eluate are concentrated, reconstituted in acetone, and then analysed by GC/NPD or in some cases, GC/MSD, using an external standard. Validation studies were conducted in parallel with some of the supervised field trials. The LOQ for this method is 0.02 mg/kg for all matrices except grapes, almonds and cotton seed, where acceptable recovery rates were achieved at a lower fortification level of 0.01 mg/kg.

For some commodities, method validation was conducted prior to analysing the samples from the supervised field trials. Recovery validation data from these trials are summarized in the table below.

Table 32 Flumioxazin analytical method (GC-MS) validation recovery rates in plant matrices

COMMODITY	FORTIFICATION (MG/KG)	N	%RECOVERY			METHOD	REFERENCE
			RANGE	MEAN	RSD		
Artichoke	0.02–0.2	6	80–115	96	14.5	RM 30A-1	SBR-0128
Asparagus	0.02–0.2	6	95–102	99	3	RM 30A-3	SBR-0116
Blueberries	0.02–0.2	6	95–109	103	5	RM 30A-3	SBR-0115
Cantaloupe	0.02–0.2	6	92–106	99	7	RM 30A-3	SBR-0112
Peanut hay	0.02–0.10	6	83–86	85	2.9	RM 30A	SBR-0018
Peanut hulls	0.02–0.10	6	90–101	96	2.1	RM 30A	SBR-0018
Peanut nutmeat	0.02–0.10	11	89–91	90	3.3	RM 30A	SBR-0018
Peanut oil	0.02–0.10	6	88–99	94	4.3	RM 30B	SBR-0018
Peanut vines	0.02–0.10	6	90–95	92	4.8	RM 30A-3	SBR-0018
Soya bean forage	0.1	3	90–98	94	4.4	RM 30A-3	SBR-0003
Soya bean hay	0.1	3	95–101	97	2.4	RM 30A-3	SBR-0003
Soya bean	0.1	3	81–84	80	2.5	RM 30A-3	SBR-0003

Method RM 30B (Flumioxazin—processed plant oils)

This method, similar to method RM 30A but without the initial acetone extraction and dichloromethane partitioning steps, used for the determination of flumioxazin in processed oils (maize oil, cottonseed oil and peanut oil) was first reported by Pensyl, 1994 [Ref: SBR-0019]. Samples are dissolved in hexane:acetonitrile (30:1), shaken with acetonitrile:hexane (5:1) and after separation, the acetonitrile extracts are evaporated and samples are cleaned-up using Florisil columns eluted with hexane:ethyl acetate (2:1 v:v). Residues in the eluate are concentrated, reconstituted in acetone, and then analysed by GC/NPD using an external standard. The LOQ of flumioxazin in oil matrices by this method is 0.02 mg/kg.

Method RM 30M (Metabolite 1-OH-HPA—animal feeds)

This method, developed for the determination of residues of the plant metabolite, 1-OH-HPA in animal feed commodities (almond hulls, peanut and soya bean forage/hay, cotton gin trash and sugar matrices) was first reported by Pensyl, 1994 [Ref: SBR-0019]. The metabolite, 1-OH-HPA is extracted from homogenized samples using acid hydrolysis (refluxing for 3 hours in 2.5 N HCl) prior to washing with hexane and partitioning into ethyl acetate. The concentrated extract is refluxed for 30 minutes with acetone, triisopropanolamine and dimethyl sulfate to convert the 1-OH-HPA to its dimethyl ester. The samples are then shaken with water and hexane, and after separation, the hexane extracts are cleaned-up using Florisil columns eluted with hexane:ether (1:2 v:v). Residues in the eluate are concentrated, reconstituted in acetone, and analysed by GC/MSD (m/z 157.2—

quantification and m/z 125.1—qualifier). The LOQs for the method are 0.02 mg/kg (peanut and soya bean forage/hay and sugar matrices) and 0.1 mg/kg (almond hulls and gin trash).

Method RM 30P (Metabolite 1-OH-HPA—processed plant oils)

This method, a modification of Method RM 30M (with an additional hexane partitioning step) to determine 1-OH-HPA in peanut and soya bean oils was reported by Pensyl, 1994 [Ref: SBR-0019]. Samples are hydrolysed in 2.5 N HCl and then partitioned with hexane to remove oils. The 1-OH-HPA is then extracted from the aqueous phase using ethyl acetate via solid phase extraction. The concentrated extract is re-dissolved in acetone and refluxed for 30 minutes with acetone, triisopropanolamine and dimethyl sulphate to convert the 1-OH-HPA to its dimethyl ester. The samples are then shaken with water and hexane, and after separation, are cleaned-up using Florisil columns eluted with hexane:ether (1:2 v:v). Residues in the eluate are concentrated, reconstituted in acetone, and analysed by GC/MSD. The LOQ for the method is 0.02 mg/kg.

Method ER-MT-9403 (Flumioxazin—animal matrices)

A method for determining residues of flumioxazin in milk, eggs and animal tissues was developed by Oishni, 1994 [Ref: SBA-0037]. Homogenised samples are double-extracted with acetone, partitioned into dichloromethane, evaporated to dryness, redissolved in ethyl acetate, diluted with hexane and purified using Florisil columns eluted with hexane:ethyl acetate (2:1 v:v for all tissues except chicken liver, where a 3:1 ratio is used). Meat and fat extracts also undergo an additional partitioning step before the Florisil clean-up, with samples being dissolved in hexane:acetonitrile (30:1), shaken with acetonitrile:hexane (5:1) and after separation, the combined acetonitrile extracts being evaporated to dryness. Residues in the eluate are concentrated, reconstituted in acetone, and then analysed by GC/NPD with a validated LOQ of 0.02 mg/kg for each analyte. Recovery data are summarized in the following table.

Table 33 Flumioxazin analytical method ER-MT-9403 (GC-MS) recovery rates in animal matrices [Ref SBA-0037]

Commodity	%Recovery 0.02 mg/kg fortification		%Recovery 0.1 mg/kg fortification		%Recovery 1.0 mg/kg fortification	
	%Recovery	%Mean	%Recovery	%Mean	%Recovery	%Mean
Meat	97, 96	96	102, 101	102	98, 96	97
Fat	101, 92	97	96, 93	95	94, 92	93
Liver	108, 99	103	100, 96	98	97, 96	96
Kidney	107, 107	107	95, 95	95	101, 99	100
Milk	105, 103	104	101, 98	100	94, 92	93
Poultry meat	96, 100	98	97, 97	97	101, 97	99
Poultry fat	96, 99	98	101, 98	100	98, 96	97
Poultry liver	87, 90	88	91, 88	90	92, 89	91
Poultry gizzard	89, 91	90	96, 96	96	97, 98	97
Eggs	97, 98	98	91, 89	90	96, 96	96

Methods RM- 30T, RM-30MK (Flumioxazin, 3-OH-flumioxazin, 4-OH-flumioxazin—animal matrices)

A method (RM-30T) for determining residues of flumioxazin and the 3-OH and 4-OH metabolites in animal tissues and a modified version (RM-30MK) were reported by Kowalsky, 2006 [Ref: SBR-0138] in an dairy cattle feeding study. Tissue samples are homogenised in acetonitrile, extracted in acetonitrile:water (50:50) acidified with 1% acetic acid. Milk samples are extracted with acetone. Sample extracts are partitioned into dichloromethane, evaporated to dryness, then dissolved in hexane:acetonitrile (30:1), shaken with acetonitrile:hexane (5:1) and after separation, the combined acetonitrile extracts being evaporated to dryness. Residues in the eluate are concentrated, reconstituted

in methanol:water and analysed by LC-MS/MS ((flumioxazin: m/z 355MS/MS 3-OH-flumioxazin: m/z 371OH-flumi and 4-OH-flumioxazin: m/z 371 →and 4-OH with an LOQ of 0.02 mg/kg for each analyte. Recovery data are summarized in the following table.

Table 34 Flumioxazin analytical methods RM-30T, RM-30MK recovery rates in animal matrices [Ref SBR-0138]

	Flumioxazin %Recovery (mean)		3-OH-flumioxazin %Recovery (mean)		4-OH-flumioxazin %Recovery (mean)	
Fortification	0.02 mg/kg	0.1 mg/kg	0.02 mg/kg	0.1 mg/kg	0.02 mg/kg	0.1 mg/kg
Muscle (concurrent)	82, 87, 88 (86)	82	72, 116, 124 (104)	84	83, 100, 108 (97)	83
Fat (concurrent)	77, 94, 103 (91)	79	75, 120, 126 (107)	87	83, 116, 119 (106)	83
Liver (validation)	77, 78, 82 (79)	85, 86, 90, 90, 90, 92 (92)	116, 117, 120 (118)	87, 89, 91, 91, 92, 93 (91)	110, 111, 111 (111)	96, 97, 101, 102, 102, 107 (101)
Liver (concurrent)	84	70	93	76	114	89
Kidney (concurrent)	81, 83, 88 (84)	82	73, 117, 120 (103)	90	81, 113, 114 103)	87
Milk (validation)	88, 89, 89 (89)	74, 78, 79,79,82, 84 (79)	80, 92, 103 (92)	81, 82, 90, 92, 93, 97 (89)	79, 87, 87 (84)	81, 83, 85, 86, 87, 90 (85)
Milk (concurrent)	77, 90, 92 (86)	78, 82, 85 (82)	96, 101, 106 (101)	78, 82, 85 (82)	92, 94, 99 (95)	83, 86, 94 (88)
Cream (concurrent)	84, 85 (85)	83	96, 98 (97)	78	89, 90 (90)	72
Skim milk (concurrent)	98	85	95	83	105	80

Analytical (concurrent) recoveries in supervised crop trials

Analytical recovery rates were measured in all the supervised crop field trials, with control samples being fortified with flumioxazin at 0.01 mg/kg or 0.02 mg/kg and at higher levels that generally reflected the range of expected residues. For each study, average recoveries per fortification level generally fell within the 70–120% range, with a relative standard deviation of 20% or less. A summary of recovery data from the methods used for plant commodities evaluated by the Meeting where one or more individual recovery values were outside the above criteria are presented in the table below.

Table 35 Flumioxazin analytical concurrent recovery rates in studies where one or more individual recovery values were outside the 70–120% range

Commodity	Fortification (mg/kg)	n	%Recovery range	%Recovery mean	%RSD	Method	Determina-tion	Study reference
Alfalfa forage	0.02–0.1	47	78–122	101	10.6	RM 30A-3	GC-MS	SBR-0111
Celery	0.02–0.2	13	90–150	113	17	RM 30A-1	GC-MS	SBR-0122
Cottonseed meal	0.01–0.05	3	101–135	113	16.6	RM 30A-1	GC-MS	SBR-0026
Grapes	0.01–0.05	16	82–123	107	9.6	RM 30A-1	GC-MS	SBR-0025
Maize grain	0.02–0.1	14	85–122	96	9.2	NCL 293	LC/MS/MS	SBR-0078
Olives	0.02–0.2	6	76–122	103	15	RM 30A-3	GC-MS	SBR-0130
Peanut hay	0.02	5	63–79	71	10	RM 30A	GC-MS	SBR-0019
Peppers	0.02–0.2	7	68–117	91	18.4	RM 30A-1	GC-MS	SBR-0118
Soya bean forage	0.02	29	67–120	92	15.3	RM 30A	GC-MS	SBR-0021
Soya bean hay	0.02	19	73–130	89	19.5	RM 30A	GC-MS	SBR-0021
Sugar cane	0.01–0.5	12	67–113	89	16	RM 30A-1	GC-MS	SBR-0022

Commodity	Fortification (mg/kg)	n	%Recovery range	%Recovery mean	%RSD	Method	Determina-tion	Study reference
Wheat grain	0.02–0.5	34	70–122	103	12.9	RM 30A-3	GC-MS	SBR-0092

In some supervised trials, residues of the 1-OH-HPA were also measured, together with analytical recovery rates in control samples fortified with 0.02–0.5 mg/kg 1-OH-HPA. For each study, average recoveries per fortification level generally fell within the 70–120% range, with a relative standard deviation of 20% or less. A summary of recovery data from the methods used for plant commodities evaluated by the Meeting are presented in the table below.

Table 36 Analytical concurrent recovery rates for 1-OH-HPA in plant matrices

Commodity	Fortification (mg/kg)	n	%Recovery range	%Recovery mean	%RSD	Method	Determina-tion	Study reference
Almond hulls	0.1–0.5	10	81–98	90	6.0	RM 30M	GC-MS	SBR-0024
Gin trash	0.1–0.5	14	81–121	99	9.4	RM 30M	GC-MS	SBR-0026
Molasses	0.02, 0.1	2	78, 114	96	–	RM 30M	GC-MS	SBR-0022
Peanut soapstock	0.02–0.1	9	69–87	74	11.6	RM 30P	GC-MS	SBR-0021
Soya bean oil	0.02–0.1	9	85–88	86	8.4	RM 30P	GC-MS	SBR-0021
Soya bean seeds	0.02	14	71–100	81	9.6	RM 30M	GC-MS	SBR-0021
Sugar	0.02, 0.1	2	80, 111	96	–	RM 30M	GC-MS	SBR-0022
Sugar cane	0.02–0.2	10	70–114	96	16.4	RM 30M	GC-MS	SBR-0022

Enforcement methods

FDA Multi-residue method

Nandihalli, 1996 [Ref: SBA-0040] evaluated the suitability of the FDA PAM Multi-residue methods for measuring residues of flumioxazin. Testing according to Protocols A, C and F showed that retention times and sensitivity criteria were not met, and that none of the FDA multi-residue method test procedures are suitable for the regulatory analysis of flumioxazin.

Multi-residue method DFG S19 (plant matrices)

The multi-residue method DFG S19 (revised) was investigated and validated for the determination of flumioxazin in cereals and other dry crops (Rzepka, 2004; SBA-0048), potato (Rzepka and Klimmek, 2006; SBA-0051), and oily crops such as sunflower seeds (Class and Merdian, 2010; SBA-0064). Samples are extracted with acetone:water (2:1 v/v) and the extracts partitioned with 1:1 v/v ethyl acetate:cyclohexane (Module E 2). The organic phase is cleaned up by gel permeation chromatography using ethyl acetate:cyclohexane (1:1, v/v) as the eluent and after concentration, flumioxazin residues are determined by GC-MS (Module D4). The fragment ion m/z 354 was used for quantitation and m/z 287 and m/z 259 were used for confirmation. The LOQ was 0.02 mg/kg for all matrices tested.

The method showed good linearity (correlation coefficients > 0.997 and no significant interferences were detected at the retention time corresponding to flumioxazin in any control samples, although confirmatory analysis of wheat straw samples yielded chromatographic interferences. These were removed by an additional clean-up step using silica gel mini-columns. The mean recoveries for all matrices tested and at all fortification levels ranged from 70 and 110%, within the acceptable range, with relative standard deviations of 20% or less.

Table 37 Multi-residue method DFG S19 analytical recovery rates for flumioxazin

Commodity	Fortification (mg/kg)	Fragment ion (m/z)	% Recovery	%Recovery mean	SD	Study reference

Commodity	Fortification (mg/kg)	Fragment ion (m/z)	% Recovery	%Recovery mean	SD	Study reference
Wheat grain	0.02 0.2	354 354	80, 106, 101, 117, 126 107, 112, 109, 112, 95	106 107	18 7	SBA-0048
Wheat grain	0.02 0.2	287 287	83, 103, 108, 118, 122 107, 113, 109, 112, 95	107 107	15 7.2	SBA-0048
Wheat grain	0.02 0.2	259 259	101, 102, 94, 100, 105 108, 113, 106, 109, 98	100 107	4 5.5	SBA-0048
Wheat straw	0.05 0.5	354 354	77, 71, 62, 73, 66 75, 77, 70, 80, 76	70 76	5.9 3.6	SBA-0048
Wheat straw	0.05 [a] 0.05 [a] 0.05 [a]	354 287 259	95, 98, 103 92, 93, 102 90, 91, 96	99 96 92	4 5.5 3.2	SBA-0048
Potato	0.02 0.2	354 354	107, 110, 112, 113, 102 107, 114, 112, 108	109 110	4.4 3.3	SBA-0051
Potato	0.02 0.2	287 287	100, 93, 108, 100, 103 108, 111, 109, 106	101 109	5.4 2.1	SBA-0051
Potato	0.02 0.2	259 259	97, 83, 112, 101, 110 106, 112, 109, 106, 74	101 101	12 16	SBA-0051
Sunflower seed	0.05 0.5	354 354	99, 102, 100, 101, 101 113, 111, 104, 101, 104	101 107	1 5	SBA-0064
Sunflower seed	0.05 0.5	287 287	99, 102, 100, 100, 101 110, 111, 101, 102, 103	100 105	1 4	SBA-0064
Sunflower seed	0.05 0.5	259 259	102, 103, 101, 101, 98 111, 110, 101, 100, 102	101 105	2 5	SBA-0064

[a] With an additional silica gel mini-column clean-up step

Stability of residues in stored analytical samples

The Meeting received information on the stability of residues of flumioxazin in a wide range of fresh and processed commodities with high water, starch, protein, oil and acid contents, stored at freezer temperatures of –20 °C (or below) for various intervals. Several studies were also provided on the stability of the 1-OH-HPA metabolite. Most of these studies were conducted concurrently with the supervised field trials, and the longest storage intervals reflected those used in the field trials.

Table 38 Stability of flumioxazin residues in a range of fresh and processed plant matrices with high water content, spiked at 0.1–0.5 mg/kg and stored at –20 °C or below

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Alfalfa forage (0.1 mg/kg)	0 131 305 929	105, 107, 106 83, 88 94,96 80, 85	– 86 95 83	106 93 101 100	RM 30A-3	SBR-0111
Alfalfa hay (0.1 mg/kg)	0 131 305 929	95, 99, 111 87, 77 91, 96 67, 73	– 82 94 70	102 79 97 70	RM 30A-3	SBR-0111
Apple juice (0.5 mg/kg)	0 60 119 196 265	88, 101, 92 85, 97, 91 85, 93 101, 102 59, 60, 63	– 91 89 102 61	94 109 93 94 81	RM 30A-3	SBR-0031
Apple wet pomace (0.5 mg/kg)	0 69 197 267	98, 98, 115 92, 98 89, 87 82, 79	– 95 88 81	104 105 87.4 79	RM 30A-3	SBR-0031

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Artichoke (0.2 mg/kg)	252	85, 90, 120	98 [a]	107	RM 30A-1	SBR-0128
Asparagus (0.1 mg/kg)	217	98, 94, 86	93 [a]	105	RM 30A-3	SBR-0116
Cabbage (0.2 mg/kg)	243	120, 120, 105	115 [a]	110	RM 30A-1	SBR-0129
Cantaloupe (0.2 mg/kg)	125	95, 94, 92	94 [a]	106	RM 30A-3	SBR-0112
Celery (0.2 mg/kg)	298	100, 90, 90	93 [a]	108	RM 30A-1	SBR-0122
Cherries (0.1 mg/kg)	0 112 316 354	104, 99, 101 101, 103 88, 92 79, 92	– 102 90 86	101 92 94 86	RM 30A-3	SBR-0027
Cucumber (0.2 mg/kg)	203	70, 85, 70	75 [a]	80	RM 30A-1	SBR-0121
Maize forage (0.1 mg/kg)	0 162 293 417	87, 95, 99 74, 90 93, 84 72, 76	– 82 89 74	94 98 95 76	RM 30A-3	SBR-0078
Maize stover (0.1 mg/kg)	0 165 293 404	98, 103, 105 77, 75 88, 90 73, 75	– 76 89 74	102 85 101 79	RM 30A-3	SBR-0078
Non-bell pepper (0.2 mg/kg)	786	77, 77, 76, 73, 77, 75	76 [a]	111	RM 30A-1	SBR-0118
Onion bulb (0.1 mg/kg)	124	92, 78, 80	83 [a]	80	RM 30A-1	SBR-0083
Peanut hay (0.1 mg/kg)	0 20 41 142 296	92, 94, 101 100, 101 93, 96 117, 128 74, 92	– 101 95 123 83	96 95 84 112 73	RM 30A-1	SBR-0018
Peanut vines (0.1 mg/kg)	0 20 40 147 300	95, 96, 99 97, 97 100, 105 110, 111 92, 100	– 97 103 111 96	97 99 103 100 100	RM 30A-1	SBR-0018
Soya bean forage (0.1 mg/kg)	0 30 92 190 240 360	102, 102, 103 87, 88 77, 79 83, 86 95, 96 112, 112	– 88 78 85 96 112	102 89 81 95 95 121	RM 30A-3	SBR-0003
Soya bean hay (0.1 mg/kg)	0 31 87 182 240 297 360	79, 79, 80 91, 92 76, 90 78, 78 66, 67 91, 92 87, 91	– 92 83 78 67 92 89	79 97 89 87 80 97 90	RM 30A-3	SBR-0003
Sugar cane (0.1 mg/kg)	0 29 64	94, 94, 99 93, 100 100, 99	– 97 100	96 86 92	RM 30A-1	SBR-0022
Sugar (0.1 mg/kg)	0 32 54	90, 97, 99 83, 96 82, 76	– 90 79	95 94 101	RM 30C	SBR-0022

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Summer squash (0.2 mg/kg)	477/479	65, 80, 80	75 [a]	108	RM 30A-1	SBR-0120
Tomato (0.2 mg/kg)	218	110, 115, 115, 125	116 [a]	100	RM 30A-1	SBR-0117

[a] % nominal residue remaining. No analysis of Day-0 sample

Table 39 Stability of flumioxazin residues in a range of of fresh and processed plant matrices matrices with high oil content, spiked at 0.05–1.0 mg/kg and stored at –20 °C or below

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Almond nutmeat (0.05 mg/kg)	0 29 60 92 186	98, 99, 101 117, 116 94, 100 123, 130 83, 79	– 117 97 127 81	99 115 95 119 99	RM 30A-1	SBR-0024
Almond hulls (0.05 mg/kg)	0 29 60 92 186	91, 91, 94 100, 112 89, 88 93, 96 92, 102	– 106 89 95 97	92 103 95 101 78	RM 30A-1	SBR-0024
Pecan (0.1 mg/kg)	0 135	88, 83, 84 100, 96, 90	– 95	85 103	RM 30A-3	SBR-0062
Cotton seed (0.1 mg/kg)	0 90 197 273	121, 126, 129 104, 104 117, 120 76, 87	– 104 119 82	125 95 120 78	RM 30A-1	SBR-0011
Cotton seed (1.0 mg/kg)	0 90 197 273	93, 100, 107 90, 117 104, 104 99, 99	– 104 104 99	100 103 111 85	RM 30A-1	SBR-0011
Cotton seed (0.05 mg/kg)	0 36 61 90 183	76, 82, 83 85, 111 81, 80 82,74 70, 77	– 98 81 78 74	80 114 77 81 97	RM 30A-1	SBR-0026
Cotton gin trash (0.05 mg/kg)	0 34 59 88	70, 70, 70 92, 84 96, 93 101, 85	– 88 95 93	70 83 93 92	RM 30A-1	SBR-0026
Cottonseed hulls (0.05 mg/kg)	0 33 61 93	93, 97, 98 111, 108 119, 111 103, 96	– 110 115 100	96 109 108 94	RM 30A-1	SBR-0026
Cottonseed meal (0.05 mg/kg)	0 33 61 93	106, 107, 108 98, 102 110, 128 113, 95	– 100 119 104	107 67 115 92	RM 30A-1	SBR-0026
Peanut nutmeat (0.1 mg/kg)	0 20 40 147 300	85, 87, 89 84, 86 92, 105 74, 86 93, 92	– 85 99 80 93	87 94 102 105 77	RM 30A-1	SBR-0018

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Peanut hull (0.1 mg/kg)	0 20 41 142 296	88, 89, 105 89, 98 91, 97 91, 93 92, 124	– 94 94 92 108	94 92 95 100 75	RM 30A-1	SBR-0018
Peanut presscake (0.1 mg/kg)	0 30	107, 108, 111 119, 119	– 119	109 96	RM 30A-1	SBR-0018
Peanut soapstock (0.1 mg/kg)	0 15 30 31	96, 98, 104, 108, 109 64, 67 37, 57 44, 44	– 66 47 44	103 111 93 97	RM 30A-1	SBR-0018
Peanut oil (crude) (0.1 mg/kg)	0 31	115, 119, 114 123, 133	– 128	116 98	RM 30B	SBR-0018
Olives (0.2 mg/kg)	526	91, 95, 105	97 [a]	89	RM 30A-3	SBR-0130
Olive oil (0.2 mg/kg)	479	99, 105, 107	104 [a]	109	RM 30A-3	SBR-0130
Mint tops (0.2 mg/kg)	0 82 354	98, 102, 104 99, 93 89, 94	– 96 92	101 98 103	RM 30A-2	SBR-0136
Mint oil (0.2 mg/kg)	0 267	77, 88, 89 84, 83	– 84	85 82	RM 30A-2	SBR-0136

[a] % nominal residue remaining. No analysis of Day-0 sample

Table 40 Stability of flumioxazin residues in soya bean seed (high protein content), spiked at 0.1 mg/kg and stored at -20 °C or below

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Soya bean seed (0.1 mg/kg)	0 30 91 178 240 357	85, 86, 88 97, 103 100, 107 91, 91 99, 101 104, 105	– 100 104 91 100 105	86 96 99 87 93 96	RM 30A-3	SBR-0003

Table 41 Stability of flumioxazin residues in a range of of fresh and processed plant matrices with high starch content, spiked at 0.1–1.0 mg/kg and stored at –20 °C or below

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Maize grain (0.1 mg/kg)	0 162 293 404	108, 110, 103 87, 87 89, 72 85, 82	– 87 81 84	107 87 92 75	RM 30A-3	SBR-0078
Potato tubers (0.1 mg/kg)	0 92 196 274	109, 116, 118 83, 117 92, 92 93, 104	– 100 92 99	114 106 96 111	RM 30A-3	SBR-0011
Potato tubers (1.0 mg/kg)	0 92 196 274	86, 88, 99 88, 89 83, 87 80, 80	– 89 85 80	91 97 113 100	RM 30A-3	SBR-0011

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Potato tubers (0.2 mg/kg)	0 218 279	92, 94, 95 113, 114 89, 93	– 114 91	94 118 104	RM 30A-2	SBR-0091
Potato chips (0.2 mg/kg)	0 279	95, 96, 99 94, 95	– 95	97 98	RM 30A-2	SBR-0091
Potato flakes (0.2 mg/kg)	0 279	91, 89, 91 92, 93	– 93	90 104	RM 30A-2	SBR-0091

Table 42 Stability of flumioxazin residues in a range of of fresh and processed plant matrices with high acid content, spiked at 0.05–0.2 mg/kg and stored at –20 °C or below

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Blueberry (0.1 mg/kg)	0 176	88, 82, 81 100, 102, 102	– 101	84 102	RM 30A-3	SBR-0115
Strawberry (0.2 mg/kg)	252 254	90, 100 100, 100	95 [a] 100 [a]	115 70	RM 30A-1	SBR-0109
Grape (0.05 mg/kg)	0 29 93 198	98, 101, 105 129, 115 93, 93 74, 100	– 122 93 87	101 116 103 95	RM 30A-1	SBR-0025
Grape juice (0.05 mg/kg)	0 30 68	94, 100, 102 111, 113 105, 92	– 112 99	99 99 101	RM 30A-1	SBR-0025
Dried grapes (0.05 mg/kg)	0 30 90 188	105, 114, 114 88, 104 106, 96 94, 83	– 96 101 89	111 99 118 114	RM 30A-1	SBR-0025

[a] % nominal residue remaining. No analysis of Day-0 sample

Table 43 Stability of 1-OH-HPA (flumioxazin metabolite) residues in a range of plant matrices spiked at 0.05–0.5 mg/kg and stored at –20 °C or below

Commodity (fortification)	Storage interval (days)	Residues remaining (%)		Procedural recovery (%)	Analytical method	Study reference
			mean			
Sugar cane (0.1 mg/kg)	0 29 65 93 393	98, 97, 84 95, 99 99, 108 99, 92 82, 87	– 97 104 96 85	93 97 108 101 95	RM 30M	SBR-0023
Sugar (0.1 mg/kg)	0 14 35 78	106, 106, 102 86, 93 78, 69 79, 95	– 90 74 87	105 94 76 94	RM 30M	SBR-0022
Almond hulls (0.5 mg/kg)	0 27 55 131 263	88, 89, 90 94, 97 77, 81 77, 80 70, 72	– 96 79 79 71	89 94 80 76 70	RM 30M	SBR-0024
Cottonseed gin trash (0.05 mg/kg)	0 34 64 140 247	95, 97, 103 113, 114 84, 91 72, 80 104, 106	– 114 88 76 105	98 116 88 78 99	RM-30M	SBR-0026

USE PATTERNS

Information on GAP in the USA was provided to the Meeting on the use of flumioxazin, available as WG, SC or WP formulations, often co-formulated with other herbicides. The Meeting also noted that flumioxazin registrations existed in Australia, Europe, Canada, Latin America and some countries in Asia.

The following table summarizes the representative critical GAPs in the USA for crops relevant to the available residue field trials.

Table 44 Representative registered uses of flumioxazin (510 g ai/kg WG formulations)

Crop	Country	Application (max)		Max/season		PHI (days)	Comments
		kg ai/ha	water L/ha	no	kg ai/ha		
Pome fruit	USA	0.42	140–280		0.84	60	Directed inter-row band sprays, up to pink bud or bud-burst, min 30 day RTI
Stone fruit	USA	0.42	140–280		0.84	60	Directed inter-row band sprays, up to bud break, min 30 day RTI
Bush berries	USA	0.42	140–280		0.42	7	Directed inter-row band sprays. Min 30 day RTI
Grapes	USA	0.42	140–280		0.84	60	Directed inter-row band sprays. Min 30 day RTI. Not after bud-break on table grapes
Strawberries	USA	0.105	140–280		0.105		Pre-plant (at least 30 days before transplanting)
	USA	0.105	140–280		0.105		Broadcast to dormant plants
	USA	0.105	140–280		0.105		Directed inter-row band application up to fruit-set
Olives	USA	0.42	140–280		0.84	60	Directed inter-row band sprays. Min 30 day RTI
Pomegranates	USA	0.42	140–280		0.84	60	Directed inter-row band sprays. Min 30 day RTI
Garlic	USA	0.21	140–280		0.21		Pre-emergent application up to 3 days after planting
Onion, bulb	USA	0.07	140–180		0.105	45	Apply from 2-leaf and 6-leaf stage (BBCH12–16). Min 14 day RTI
Cabbage, head	USA	0.14	140–280		0.28		Pre-plant directed inter-row application (between raised plastic mulched beds)
Cucurbit vegetables	USA	0.14	140–280		0.28		Pre-plant directed inter-row applications (between raised plastic mulched beds), up to 14 days before planting
	USA	0.14	140–280		0.28		Directed inter-row band application up to 21 days after transplanting/emergence, not after start of flowering
Fruiting	USA	0.14	140–280		0.28		Pre-plant directed inter-row applications (between raised plastic mulched beds), up to 14 days before

Crop	Country	Application (max)		Max/season		PHI (days)	Comments
		kg ai/ha	water L/ha	no	kg ai/ha		
vegetables							planting
	USA	0.14	140–280		0.28		Directed inter-row band application up to 21 days after transplanting/emergence. Not after start of flowering
Beans, dry (incl lentils)	USA	0.07	140–280		0.07		Pre-plant or pre-emergent (up to 2 days after sowing)
	USA	0.105	140–560		0.105	5	Apply when crop is mature and at least 80% of pods are yellowing (BBCH 87–89)
Field peas	USA	0.07	140–280		0.07		Pre-plant or pre-emergent (up to 2 days after sowing)
	USA	0.105	140–560		0.105	5	Apply when crop is mature and at least 80% of pods are yellowing (BBCH 87–89)
Soya bean	USA	0.105	140–280		0.105		Pre-plant or pre-emergent (up to 3 days after sowing). No grazing or use for stock feed
Potato	USA	0.053	140–280		0.053		Pre-emergent after hilling or to soil-covered potatoes
Sweet potato	USA	0.105	140–280		0.105		Pre-plant
Artichoke, Globe	USA	0.21	94–280		0.21		Pre-plant (annual varieties) or pre-emergence (perennial varieties)
Asparagus	USA	0.21	140–280		0.21		Broadcast application min 14 days prior to spear emergence (perennial varieties) or fern emergence (annual varieties)
Celery	USA	0.105	140–280		0.105		Pre-transplant
	USA	0.105	140–280		0.105		Broadcast application, 3–7 days after transplanting
Maize	USA	0.105	140–280		0.105		Broadcast application 14–30 days prior to sowing
Wheat	USA	0.07	140–280		0.07		Pre-plant or pre-emergent (up to 2 days after sowing) in minimum tillage fields. No grazing until wheat is 13 cm high
	USA	0.07	min 93 air 47		0.07	10	Apply when crop reaches BBCH 87 (hard dough stage, grain 70% DM)
Sugar cane	USA	0.28	140–280		0.42		Broadcast up to 14 days before planting or broadcast pre-emergent
	USA	0.14	140–280		0.42	90	Directed inter-row band applications after canes are 60 cm height or at layby (canes > 76 cm height). Min 14 day application interval

Crop	Country	Application (max)		Max/season		PHI (days)	Comments
		kg ai/ha	water L/ha	no	kg ai/ha		
Tree nuts	USA	0.42	140–280		0.84	60	Directed inter-row band sprays. Min 60 day RTI
Cotton	USA	0.07	140–280		0.14		Autumn or spring burndown, up to 21 days before planting
	USA	0.07	140–280		0.14	60	Directed inter-row band applications after cotton is 15 cm height or at layby (cotton > 40 cm height). Min 30 day application interval. Use with non-ionic adjuvant
Linseed (flax)	USA	0.105	140–560		0.105	5	Apply when crop is mature and at least 75% of the seed heads are brown in colour (BBCH 87–89). Mix with MSO adjuvant
Sunflower seed Safflower seed	USA	0.105	140–560		0.105	5	Apply when crop is mature (BBCH 86–87—seedheads yellowing and the bracts turning brown). Mix with MSO adjuvant
Peanut	USA	0.105	140		0.105		Pre-plant or pre-emergent (up to 2 days after sowing). With adjuvant. No grazing or use for stock feed
Mints (spearmint, peppermint)	USA	0.14	140–180		0.28	80	Autumn-spring applications to established dormant plants. At least 60 days between applications
Alfalfa	USA	0.14	94–280		0.28	25	After last cut (Autumn) and/or after 1st cut, before crop reaches 15 cm height. PHI is for cutting and grazing

Pome fruit = apple, crabapple, loquat, mayhaw, pear, pear (oriental) and quince

Stone Fruit = apricot, cherries (sweet and tart), nectarine, peach, plum (chickasaw, damson, japanese), plumcot and prune

Bushberries = aronia berry, black currant, blueberry (highbush, rabbit-eye and lowbush), buffalo currant, chilean guava, cranberry (highbush), elderberry, european barberry, gooseberry, honeysuckle (edible), huckleberry, jostaberry, juneberry, lingonberry, native currant, red currant, salal and sea buckthorn

Cucurbits = chayote (fruit); chinese waxgourd (chinese preserving melon); citron melon; cucumber; gherkin; gourd, edible (includes hyotan, cucuzza, hechima, chinese okra); Momordica spp. (includes balsam apple, balsam pear, bittermelon, chinese cucumber); muskmelon (includes cantaloupe); pumpkin; squash, summer; squash, winter (includes butternut squash, calabaza, hubbard squash, acorn squash, spaghetti squash) and watermelon

Fruiting vegetables = eggplant, groundcherry (Physalis spp), okra, pepino, peppers (Capsicum spp incl bell, chili, cooking, pimento & sweet), tomatillo and tomato

Tree nuts = almond, beechnut, betelnut, black walnut, brazil nut, butternut, cashew, chestnut, chinquapin, coconut, english walnut, filbert (hazelnut), ginkgo, heartnut, hickory nut, macadamia nut, oak, pecan, pili nut, pine nut, pistachio and tropical almond

Dry beans = Dried cultivars of bean (Lupinus), bean (Phaseolus) (incl field bean, kidney bean, lima bean (dry), navy bean, pinto bean, tepary bean); bean (Vigna) (incl adzuki bean, blackeyed pea, catjang, cowpea, crowder pea, moth bean, mung bean, rice bean, southern pea, urd bean); broad bean (dry); chickpea; guar; lablab bean, lentil

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials involving soil or foliar treatments of flumioxazin to the following crops.

Group	Crop	Countries	Table no
Pome fruits	Apple, Pear	USA	45
Stone fruits	Cherry, Peach Plum	USA	46
Berries and other small fruit	Blueberry	USA	47
	Grape	USA	48
	Strawberries	USA	49
Assorted tropical and sub-tropical fruits	Olives	USA	50
	Pomegranate	USA	51
Bulb vegetables	Onion, dry bulb	USA	52
Brassica vegetables	Cabbage	USA	53
Fruiting vegetables, Cucurbits	Cucumber	USA	54
	Melons	USA	55
	Summer squash	USA	56
Fruiting vegetables, other than Cucurbits	Peppers	USA	57
	Tomato	USA	58
Pulses	Beans (dry)	USA	59
	Peas (dry)	USA	60
	Soya bean (dry)	USA	61
Root and tuber vegetables	Potato	USA	62
Stalk and stem vegetables	Artichoke, Globe	USA	63
	Asparagus	USA	64
	Celery	USA	65
Cereal grains	Maize	North America	66
	Wheat	USA	67
Grasses for sugar or syrup production	Sugar cane	USA	68
Tree nuts	Almond	USA	69
	Pecan	USA	70
Oilseed	Cottonseed	USA	71
	Rape seed	USA	72
	Peanut	USA	73
	Sunflower seed	USA	74
Herbs	Mint leaves and oil	USA	75
Legume animal feeds	Alfalfa forage and fodder	USA	76, 77
	Peanut vines and fodder	USA	78
	Soya bean forage and fodder	USA	79
Straw, forage, fodder of cereal grains	Maize forage and fodder	USA	80
	Wheat forage, hay and straw	USA	81, 82

The supervised trials were well documented with laboratory and field reports. Laboratory reports included method validation including procedural recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables unless residues in control samples exceeded the LOQ. In such cases, the residues found are noted as "c=nn mg/kg" in the Reference and Comments columns. Residue data are recorded unadjusted for recovery.

Results from replicated field plots are presented as individual values. Residues and application rates have been reported as provided in the study reports, although the results from trials used for the estimation of maximum residue levels (underlined) have been rounded to two significant digits (or if close to the LOQ, rounded to one significant digit) in the Appraisal.

When multiple applications were made to a crop, the application rate, spray concentration and spray volume were not always identical from one application to the next. In most trials, the actual treatment rates were within 10% of the listed 'target' application rates, but if not, the actual treatment rates are listed.

Pome fruits

In supervised trials on pome fruit (12 on apples and six on pears) conducted in the USA during 2002–2003, two inter-row/berm soil treatments of 0.42–0.45 kg ai flumioxazin/ha (WG or SC formulations) were applied using tractor-mounted boom sprayers or back-pack sprayers with hand-held booms. Treatments were applied about 60 days apart, with the last application about 60 days before harvest.

Duplicate samples of mature fruit (min 2 kg or 24 fruit) were frozen within 2 hours and analysed for flumioxazin within 1 year of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 80–115% and the validated LOQ was 0.02 mg/kg.

Table 45 Residues in pome fruit from supervised trials in the USA involving two directed inter-row soil applications of flumioxazin (SC or WG formulations)

POME FRUIT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
PEAR									
USA, 2002 Orefield, PA (Bartlett)	2	0.43 0.44	354 362	0.87	whole fruit	59	< 0.02, < 0.02	< 0.02	SBR-0029 V-24678-02-C
USA, 2002 Soap Lake, WA (Anjou)	2	0.445 0.429	164 205	0.874	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0029 V-24678-02-A
USA, 2002 Ukiah, CA (Bosc)	2	0.427 0.436	186 189	0.863	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0029 V-24678-02-B
USA, 2003 Hood River, OR (Bosc)	2	0.419 0.434	270 311	0.853	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0029 V-24678-03-F
USA, 2003 Ukiah, CA (Bosc)	2	0.434 0.434	189 189	0.868	whole fruit	61	< 0.02, < 0.02	< 0.02	SBR-0029 V-24678-03-D
USA, 2003 White Salmon, WA (Bosc)	2	0.434 0.439	282 314	0.873	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0029 V-24678-03-E
APPLE									

POME FRUIT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2002 Conklin, MI (Red Delicious)	2	0.40 0.431	260 266	0.861	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-C
USA, 2002 Eckert, CO (Yellow Delicious)	2	0.434 0.445	163 167	0.879	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-E
USA, 2002 Ephrata, WA (Rome)	2	0.431 0.432	200 201	0.863	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-F
USA, 2002 Hood River, OR (Jonagold)	2	0.445 0.441	293 294	0.886	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-D
USA, 2002 Monetta, SC (Gala)	2	0.43 0.431	259 255	0.861	whole fruit	56	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-B
USA, 2002 Orefield, PA (Rome)	2	0.43 0.429	354 353	0.859	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-A
USA, 2003 Conklin, MI (Red Delicious)	2	0.431 0.432	270 259	0.863	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-J
USA, 2003 North Rose, NY Golden Delicious)	2	0.441 0.432	287 282	0.873	whole fruit	61	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-H
USA, 2003 Orefield, PA (Rome)	2	0.429 0.445	355 368	0.874	whole fruit	58	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-I
USA, 2003 Parkdale, OR (Jonagold)	2	0.441 0.438	285 314	0.879	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-03-M
USA, 2003 Payette, ID (Rome)	2	0.426 0.423	279 276	0.849	whole fruit	61	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-03-L
USA, 2003 Santa Maria, CA (Fuji)	2	0.424 0.421	276 275	0.845	whole fruit	60	< 0.02, < 0.02	< 0.02	SBR-0031 V-24504-02-K

Stone fruits

Cherry, peach, and plum

In supervised trials on stone fruit (six on cherries, nine on peaches, and six on plums) conducted in the USA during 2002–2003, two inter-row/berm soil treatments of 0.42–0.45 kg ai flumioxazin/ha (WG or SC formulations) were applied using tractor-mounted boom sprayers or back-pack sprayers with hand-held booms. Treatments were applied 50–60 days apart (except in two trials with shorter intervals of 34 and 15 days) with the last application about 60 days before harvest.

Duplicate samples of mature fruit (min 1 kg cherries, 2 kg peaches, plums) were frozen within 2 hours and after removing the stones, were analysed for flumioxazin within 10 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 75–120% and the validated LOQ was 0.02 mg/kg.

Table 46 Residues in stone fruit from supervised trials in the USA involving two directed inter-row soil applications of flumioxazin (SC or WG formulations)

STONE FRUIT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
CHERRY									
USA, 2002 Casnovia, MI (Montmorency)	2	0.432 0.427	256 253	0.859	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0027 V-24694-B
USA, 2002 Conklin, MI (Montmorency)	2	0.427 0.427	261 255	0.854	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0027 V-24694-A
USA, 2002 Ephrata, WA (Van)	2	0.425 0.427	186 187	0.852	fruit without stone	61	< 0.02, < 0.02	< 0.02	SBR-0027 V-24694-F
USA, 2002 Madera, CA (Brooks)	2	0.42 0.425	324 326	0.845	fruit without stone	59	< 0.02, < 0.02	< 0.02	SBR-0027 V-24694-D 34 day RTI
USA, 2002 Orefield, PA (Montmorency)	2	0.435 0.425	356 349	0.86	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0027 V-24694-C 15 day RTI
USA, 2002 Parkdale, OR (Bing)	2	0.435 0.413	321 344	0.848	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0027 V-24694-E
PEACH									
USA, 2002 Athens, GA (Contender)	2	0.423 0.432	319 329	0.855	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-B
USA, 2002 Conklin, MI (Red Heaven)	2	0.435 0.43	245 255	0.865	fruit without stone	59	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-D
USA, 2002 Mexia, TX (Redskins)	2	0.425 0.43	326 330	0.855	fruit without stone	55	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-C
USA, 2002 Orefield, PA (Suncrest)	2	0.445 0.428	365 352	0.873	fruit without stone	59	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-A
USA, 2002 Selma, CA (September Sun)	2	0.445 0.437	192 189	0.882	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-E
USA, 2003 Athens, GA (Contender)	2	0.435 0.435	280 279	0.87	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-F
USA, 2003 Batesburg, SC (Monroe)	2	0.432 0.437	247 254	0.869	fruit without stone	53	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-G
USA, 2003 Gridley, CA (Starn)	2	0.43 0.43	234 234	0.86	fruit without stone	59	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-I
USA, 2003 Selma, CA (September Sun)	2	0.437 0.43	190 187	0.867	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0028 V-24686-H
PLUM									

STONE FRUIT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2002 Conklin, MI (Vision)	2	0.437 0.43	262 249	0.867	fruit without stone	59	< 0.02, < 0.02	< 0.02	SBR-0030 V-24539-B
USA, 2002 Hughson, CA (French)	2	0.428 0.428	375 375	0.856	fruit without stone	46 53 60 68 75	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	SBR-0030 V-24539-F
USA, 2002 Madera, CA (Fortune)	2	0.432 0.423	333 326	0.855	fruit without stone	59	< 0.02, < 0.02	< 0.02	SBR-0030 V-24539-D
USA, 2002 Porterville, CA (Angelino)	2	0.440 0.435	308 325	0.875	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0030 V-24539-C
USA, 2002 Yuba City, CA (French)	2	0.43 0.43	188 187	0.86	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0030 V-24539-E
USA, 2003 Zillah, WA (Autumn Sweet)	2	0.42 0.423	310 314	0.843	fruit without stone	60	< 0.02, < 0.02	< 0.02	SBR-0030 V-24539-H

Berries and other small fruits

Blueberries

In supervised trials on blueberries (six) conducted in the USA during 2003, two inter-row/berm soil treatments of 0.41–0.45 kg ai flumioxazin/ha (WG formulations) were applied using back-pack sprayers with 1–4 nozzle hand-held booms. Treatments were applied 50–113 days apart with the last application 6–8 days before harvest (except in one lowbush trial where a single application was made to dormant bushes).

Duplicate samples of mature fruit (min 1 kg except at two sites) were frozen within 4 hours and analysed for flumioxazin within 6 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 74–113% and the validated LOQ was 0.02 mg/kg.

Table 47 Residues in blueberries from supervised trials in the USA involving 1–2 directed inter-row soil applications of flumioxazin (WG formulations)

BLUEBERRY COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2003 Aurora, OR (Bluecrop)	2	0.44 0.45	287 299	0.89	berries	7	< 0.02, < 0.02	< 0.02	SBR-0115 OR11
USA, 2003 Bridgeton, NJ, (Duke)	2	0.45 0.44	238 231	0.89	berries	7	< 0.02, < 0.02	< 0.02	SBR-0115 NJ16
USA, 2003 Castle Hayne, NC (Croatan)	2	0.42 0.41	274 270	0.83	berries	6	< 0.02, < 0.02	< 0.02	SBR-0115 NC15

BLUEBERRY COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2003 Holt, MI (Jersey)	2	0.43 0.45	192 203	0.88	berries	8	< 0.02, < 0.02	< 0.02	SBR-0115 MI21 56g sample size
USA, 2003 Jonesboro, ME (Wild blueberries) Lowbush	1	0.45	194	0.45	berries	99	< 0.02, < 0.02	< 0.02	SBR-0115 ME02 Dormant bushes
USA, 2003 Onondaga, MI (Bluecrop)	2	0.45 0.44	200 197	0.89	berries	8	< 0.02, < 0.02	< 0.02	SBR-0115 MI22 227g sample size

Grapes

In supervised trials on grapes (12) conducted in the USA during 1999, two directed inter-row/berm soil treatments of 0.4–0.43 kg ai flumioxazin/ha (WG formulations) with added crop oil were applied using tractor-mounted boom sprayers or back-pack sprayers with hand-held booms. Treatments were applied about 60 days apart with the last application about 60 days before harvest.

Duplicate samples of grapes (min 12 bunches or 1 kg) were frozen within 4 hours and analysed for flumioxazin within 6 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.01 and 0.05 mg/kg ranged from 82–123% and the LOQ was 0.01 mg/kg.

Table 48 Residues in grapes from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulations)

GRAPES COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1999 Breinigsville, PA (Vidal 256)	2	0.421 0.419	187 187	0.84	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-B
USA, 1999 Dundee, NY (Delaware)	2	0.416 0.408	185 181	0.824	bunches	59	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-A
USA, 1999 Dunnigan, CA (Symphony)	2	0.419 0.418	186 186	0.837	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-C
USA, 1999 Hughson, CA (Thompson seedless)	2	0.421 0.427	234 238	0.848	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-N
USA, 1999 Hughson, CA (Thompson seedless)	2	0.86 0.844	240 235	1.704	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-N 2x
USA, 1999 Kerman, CA (Thompson seedless)	2	0.42 0.425	184 187	0.845	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-L

GRAPES COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1999 Madera, CA (Thompson seedless)	2	0.418 0.423	186 188	0.841	bunches	59	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-D
USA, 1999 Orland, CA (Zinfindel)	2	0.422 0.42	218 223	0.842	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-G
USA, 1999 Orland, CA (Zinfindel)	2	0.83 0.826	214 219	1.656	bunches	60	< 0.01, < 0.01	0.01	SBR-0025 V-20108-G 2x
USA, 1999 Poplar, CA (Thompson seedless)	2	0.42 0.422	186 187	0.842	bunches	59	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-E
USA, 1999 San Luis Obispo, CA (Chardonnay)	2	0.426 0.42	238 234	0.846	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-H
USA, 1999 Temecula, CA (Merlot)	2	0.424 0.434	189 191	0.858	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-I
USA, 1999 Trinidad, W (Gamay Noir)	2	0.422 0.419	188 187	0.841	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-J
USA, 1999 Watsonville, CA (Pinot Noir)	2	0.398 0.428	209 224	0.826	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-F
USA, 1999 Watsonville, CA (Pinot Noir)	2	0.836 0.828	219 217	1.664	bunches	60	< 0.01, < 0.01	< 0.01	SBR-0025 V-20108-F 2x

Strawberry

In supervised trials on strawberries (five) conducted in the USA during 2002, one inter-row soil treatment of 0.1–0.11 kg ai flumioxazin/ha (WG formulations) was applied 1–2 days before harvest using shielded back-pack sprayers with 1–4 nozzle mini-booms. In three additional trials, two applications of 0.1 kg ai/ha flumioxazin were made, the first being a broadcast application to dormant strawberries and the second as an inter-row shielded application 1–2 days before harvest.

Duplicate samples of at least 1 kg mature fruit (with sepals removed) were frozen within 4 hours and analysed for flumioxazin within 7 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 100–120% and the validated LOQ was 0.02 mg/kg.

Table 49 Residues in strawberries from supervised trials in the USA involving 1–2 inter-row soil applications of flumioxazin (WG formulations)

STRAWBERRIES COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2002 Bridgeton, NJ (Early Glow)	2	0.109 0.104	215 253	0.213	berries	1	< 0.02, < 0.02	< 0.02	SBR-0109 08063.02-NJ04

STRAWBERRIES COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2002 Clinton, NC (Camarosa)	1	0.108	187	0.108	berries	1	< 0.02, < 0.02	< 0.02	SBR-0109 08063.02-NC06
USA, 2002 Holt, MI (Mira)	2	0.104 0.110	178 187	0.214	berries	1	0.034, 0.021	0.03	SBR-0109 08063.02-MI04
USA, 2002 Live Oak, FL (Sweet Charlie)	1	0.108	187	0.108	berries	1	< 0.02, < 0.02	< 0.02	SBR-0109 08063.02-FL08
USA, 2002 Madera, CA (Hecker)	1	0.106	281	0.106	berries	1	< 0.02, < 0.02	< 0.02	SBR-0109 08063.02-CA26
USA, 2002 Mt. Vernon, WA (Totem)	2	0.108 0.113	187 196	0.221	berries	1	< 0.02, < 0.02	< 0.02	SBR-0109 08063.02-WA36
USA, 2002 Salinas, CA (Diamonte)	1	0.108	327	0.108	berries	2	0.034, 0.036	0.04	SBR-0109 08063.02-CA*24
USA, 2002 Watsonville, CA (Camarosa)	1	0.105	346	0.105	berries	1	0.036, 0.05	0.04	SBR-0109 08063.02-CA*25

Assorted tropical and sub-tropical fruits

Olives

In supervised trials on olives (five) conducted in the USA during 2008, two directed inter-row/berm soil treatments of 0.4–0.43 kg ai flumioxazin/ha (WG formulations) with added crop oil were applied using back-pack sprayers with hand-held 3-nozzle minibooms. Treatments were applied about 60 days apart with the last application 56–59 days before harvest.

Duplicate samples of olives were stored refrigerated for up to 2 days before pitting, with the pitted olives (min 0.5 kg) frozen within 2.5 hours and analysed for flumioxazin within 18 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 76–122% and the validated LOQ was 0.02 mg/kg.

Table 50 Residues in olives from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulations)

OLIVE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2008 Orange Cove, CA (Manzanillo)	2	0.415 0.421	326 330	0.841	fruit without pits	59	< 0.02, < 0.02	< 0.02	SBR-0130 CA94
USA, 2008 Orange Cove, CA (Manzanillo)	2	0.424 0.423	232 240	0.852	fruit without pits	59	< 0.02, < 0.02	< 0.02	SBR-0130 CA95 not independent

OLIVE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2008 Glenn, CA (Korondiki 1-38 clone)	2	0.435 0.437	224 224	0.874	fruit without pits	57	< 0.02, < 0.02	< 0.02	SBR-0130 CA92
USA, 2008 Glenn, CA (Arbosama 1-43 line)	2	0.424 0.408	218 210	0.829	fruit without pits	57	< 0.02, < 0.02	< 0.02	SBR-0130 CA93 not independent
USA, 2008 Glenn, CA (Arbegnina 1-18 clone)	2	0.423 0.432	217 222	0.852	fruit without pits	56	< 0.02, < 0.02	< 0.02	SBR-0130 CA91 not independent

Pomegranate

In supervised trials on pomegranates (three) conducted in the USA during 2008, two directed inter-row/berm soil treatments of 0.4–0.43 kg ai flumioxazin/ha (WG formulations) with adjuvant were applied using back-pack sprayers with hand-held 3-nozzle minibooms. Treatments were applied about 60 days apart with the last application 57–59 days before harvest.

Duplicate samples of fruit (min 24 fruit, 6 kg) were frozen within 4 hours and analysed for flumioxazin within 17 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 80–103% and the validated LOQ was 0.02 mg/kg.

Table 51 Residues in pomegranates from supervised trials in the USA involving two inter-row soil of flumioxazin (WG formulations)

POMEGRANATE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
GAP:USA	2	0.42	140–280	0.84		60	Directed inter-row soil sprays, min 30 day RTI		
USA, 2008 Kettleman City, CA (Wonderful)	2	0.42 0.423	305 333	0.841	whole fruit	59	< 0.02, < 0.02	< 0.02	SBR-0131 CA82
USA, 2008 Kettleman City, CA (Wonderful)	2	0.429 0.427	240 239	0.852	whole fruit	59	< 0.02, < 0.02	< 0.02	SBR-0131 CA83 not independent
USA, 2008 Gridley, CA (Wonderful)	2	0.407 0.411	291 294	0.818	whole fruit	57	< 0.02, < 0.02	< 0.02	SBR-0131 CA96

Bulb vegetables

Onion, dry bulb

In supervised trials on bulb onions (nine) conducted in the USA during 2001, two foliar broadcast sprays of 0.1–0.11 kg ai flumioxazin/ha (WG formulations) with added adjuvant were applied using tractor-mounted, wheeled or back-pack sprayers with 3–6 nozzle minibooms. The first applications were made when the onions were at or about the 2-leaf stage, re-treatment intervals were 29–78 days and the last applications were 42–49 days before harvest. Phytotoxicity was observed in most trials.

Duplicate samples of topped and trimmed dry onion bulbs (min 12 bulbs, 1.3 kg) were frozen within 1 hour and analysed for flumioxazin within 2 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 70–120% and the validated LOQ was 0.02 mg/kg.

Table 52 Residues in onion bulbs from supervised trials in the USA involving two broadcast foliar applications of flumioxazin (WG formulations)

ONION, BULB COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2001 Bridgeton, NJ (Santana)	2	0.11 0.102	250 272	0.212	bulb	42	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-NJ02 RTI 62 days
USA, 2001 Celeryville, OH (Burgos)	2	0.102 0.108	317 365	0.21	bulb	42	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-OH*02 RTI 37 days
USA, 2001 Fort Collins, CO (Vision)	2	0.115 0.101	206 178	0.216	bulb	43	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-CO01 RTI 64 days
USA, 2001 Freeville, NY (F1 Candy)	2	0.109 0.108	285 285	0.216	bulb	44	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-NY01 RTI 51 days Includes 11 d field drying
USA, 2001 Fresno, CA (Cimarron)	2	0.114 0.11	391 304	0.224	bulb	44	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-CA128 RTI 72 days
USA, 2001 Laingsburg, MI USA, 2001 (Hustler F1)	2	0.11 0.109	191 194	0.219	bulb	44	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-MI02 RTI 33 days
USA, 2001 Prosser, WA (Teton)	2	0.104 0.103	148 147	0.207	bulb	45	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-WA*04 RTI 33 days
USA, 2001 Salinas, CA (Tahoe)	2	0.112 0.105	325 312	0.217	bulb	49	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-CA*03 RTI 29 days Includes 9 d field drying
USA, 2001 Weslaco, TX (Cougar)	2	0.106 0.105	208 219	0.212	bulb	48	< 0.02, < 0.02	< 0.02	SBR-0083 07389.01-TX01 RTI 78 days

Brassica vegetables

Cabbage

In supervised trials on cabbage (eight) conducted in the USA during 2006, one pre-plant broadcast soil treatment of 0.05 kg ai/ha or 0.1–0.11 kg ai flumioxazin/ha (WG formulations) were applied using tractor-mounted or back-pack sprayers with 3–8 nozzle booms. Phytotoxicity was observed in most trials, particularly in the plots treated at the higher rate.

Duplicate samples of mature cabbage heads with wrapper leaves (min 12 heads) from most plots were quartered or halved in the field (to give sample sizes of at least 1.8 kg). In three trials, smaller sample sizes were taken because of reduced plant numbers. Samples were frozen within 2 hours and analysed for flumioxazin within 8 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 85–90% and the validated LOQ was 0.02 mg/kg.

Table 53 Residues in cabbage heads (with wrapper leaves) from supervised trials in the USA involving one pre-plant broadcast soil application of flumioxazin (WG formulation)

CABBAGE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2006 Freeville, NY (Bobcat)	1	0.052	277	0.052	head with wrapper leaves	94	< 0.02, < 0.02	< 0.02	SBR-0129 NY08 Subsampled in the field
	1	0.107	286	0.107	head with wrapper leaves	94	< 0.02, < 0.02	< 0.02	
USA, 2006 Bridgeton, NJ (Wisconsin Golden Acre)	1	0.051	327	0.051	head with wrapper leaves	55	< 0.02, < 0.02	< 0.02	SBR-0129 NJ15 Subsampled in the field
	1	0.107	337	0.107	head with wrapper leaves	55	< 0.02, < 0.02	< 0.02	
USA, 2006 Clinton, NC (Bravo)	1	0.053	326	0.053	head with wrapper leaves	67	< 0.02, < 0.02	< 0.02	SBR-0129 NC10 Subsampled in the field
	1	0.106	326	0.106	head with wrapper leaves	67	< 0.02, < 0.02	< 0.02	
USA, 2006 Citra, FL (Bravo)	1	0.054	286	0.054	head with wrapper leaves	67	< 0.02, < 0.02	< 0.02	SBR-0129 FL24 Subsampled in the field
	1	0.108	286	0.108	head with wrapper leaves	67	< 0.02, < 0.02	< 0.02	
USA, 2006 Arlington, WI (Blue Vantage)	1	0.053	271	0.053	head with wrapper leaves	87	< 0.02, < 0.02	< 0.02	SBR-0129 WI15 Subsampled in the field
	1	0.106	271	0.106	head with wrapper leaves	87	< 0.02, < 0.02	< 0.02	
USA, 2006 Wesalco, TX (Blue Vantage)	1	0.053	284	0.053	head with wrapper leaves	98	< 0.02, < 0.02	< 0.02	SBR-0129 TX*26 Subsampled in the field
	1	0.107	284	0.107	head with wrapper leaves	98	< 0.02, < 0.02	< 0.02	
USA, 2006 Brighton, CO (Blue Dynasty)	1	0.054	190	0.054	head with wrapper leaves	84	< 0.02, < 0.02	< 0.02	SBR-0129 CO06
	1	0.106	187	0.106	whole plants [a]	84	< 0.02, < 0.02	< 0.02	
USA, 2006 Holtville, CA (Grenadier)	1	0.054	240	0.054	head with wrapper leaves	104	< 0.02, < 0.02	< 0.02	SBR-0129 CA61 Subsampled in the field
	1	0.108	242	0.108	head with wrapper leaves	104	< 0.02, < 0.02	< 0.02	

[a] Reduced sample size—only two whole plants able to be collected

Fruiting vegetables, cucurbits

Supervised trials on fruiting vegetables, cucurbits were conducted in the USA between 2003 and 2005.

Cucumber

In eight trials on cucumbers, two treatments of 0.14–0.17 kg ai flumioxazin/ha (WG formulations) were applied as inter-row shielded applications, the first application about 14 days before crop emergence or before transplanting and the second application about 21 days after transplanting or 28 days after the crop emergence (at or before flowering). Treatments were made using tractor-mounted or backpack sprayers with 1–4 shielded nozzle minibooms.

Duplicate samples of mature fruit (min 12 units, 1.8 kg). In two trials, samples were quartered or halved in the field. Samples were frozen within 2.5 hours and analysed for flumioxazin within 5 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 70–120% and the validated LOQ was 0.02 mg/kg.

Table 54 Residues in cucumbers from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

CUCUMBER COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2005 Salisbury, MD (Genuine)	2	0.142 0.14 [a]	279 273	0.282	whole fruit	6	< 0.02, < 0.02	< 0.02	SBR-0121 MD08
USA, 2005 Charleston, SC (Poinsett 76)	2	0.141 0.173	265 289	0.313	whole fruit	15	< 0.02, < 0.02	< 0.02	SBR-0121 SC*02
USA, 2005 Holt, MI (Journey)	2	0.151 0.149	201 199	0.3	whole fruit	15	< 0.02, < 0.02	< 0.02	SBR-0121 MI05
USA, 2005 Citra, FL (Dasher II)	2	0.145 0.143	192 191	0.288	whole fruit	7	< 0.02, < 0.02	< 0.02	SBR-0121 FL19
USA, 2005 Arlington, WI (Zapata)	2	0.14 0.141	312 317	0.281	whole fruit	29	< 0.02, < 0.02	< 0.02	SBR-0121 WI07
USA, 2005 Clinton, NC (Dasher II)	2	0.14 0.141	204 205	0.281	whole fruit	11	< 0.02, < 0.02	< 0.02	SBR-0121 NC29
USA, 2005 Tifton, GA (Diva)	2	0.137 0.137 [a]	193 192	0.273	whole fruit	21	0.024, 0.027	0.03	SBR-0121 GA*07
USA, 2005 Wesalco, TX (Poinsett 76)	2	0.139 0.142	271 214	0.281	whole fruit	31	< 0.02, < 0.02	< 0.02	SBR-0121 TX*17

[a] 2nd application after the start of flowering

Melon (cantaloupe)

In eight trials on cantaloupes, two treatments of 0.14–0.15 kg ai flumioxazin/ha (WG formulations) were applied as inter-row shielded applications, the first application about 10–14 days before transplanting or 4–7 days before sowing and the second application about 21 days after transplanting or 28 days after the crop emergence (at or before flowering). Treatments were made using tractor-mounted or backpack sprayers with 1–2 shielded nozzle minibooms.

Duplicate samples of mature fruit (min 12 units) were subsampled in the field (2–5 cm longitudinal sections) to give sample sizes of at least 1.8 kg. Samples were frozen within 3 hours and analysed for flumioxazin within 4 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 72–108% and the validated LOQ was 0.02 mg/kg.

Table 55 Residues in melons from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

MELON COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2003 Clinton, NC (Athena)	2	0.14 0.14	206 202	0.28	whole fruit	41	< 0.02, < 0.02	< 0.02	SBR-0112 NC13
USA, 2003 Five Points, CA (Gold Express)	2	0.14 0.14	202 219	0.28	whole fruit	47	< 0.02, < 0.02	< 0.02	SBR-0112 CA72
USA, 2003 Holt, MI (Athena)	2	0.14 0.15	190 195	0.29	whole fruit	69	< 0.02, < 0.02	< 0.02	SBR-0112 MI20
USA, 2003 Mesilla, NM (Top Mark SR)	2	0.14 0.14	231 206	0.289	whole fruit	53	< 0.02, < 0.02	< 0.02	SBR-0112 NM07
USA, 2003 Mesilla, NM (Top Mark SR)	2	0.14 0.14	230 229	0.28	whole fruit	51	< 0.02, < 0.02	< 0.02	SBR-0112 NM08
USA, 2003 Parlier, CA (Top Mark)	2	0.14 0.15	291 286	0.29	whole fruit	36	< 0.02, < 0.02	< 0.02	SBR-0112 CA73
USA, 2003 Wesalco, TX (Cruiser)	2	0.14 0.14	298 275	0.28	whole fruit	47	< 0.02, < 0.02	< 0.02	SBR-0112 TX*23
USA, 2003 Wesalco, TX USA, 2003 (Primo)	2	0.14 0.14	223 225	0.28	whole fruit	52	< 0.02, < 0.02	< 0.02	SBR-0112 TX22

Summer squash

In eight trials on summer squash, two treatments of 0.14–0.15 kg ai flumioxazin/ha (WG formulations) were applied as inter-row shielded applications, the first application about 10–14 days before planting or before crop emergence and the second application about 20–26 days after transplanting or 29–30 days after the crop emergence (at or before flowering or fruiting). Treatments were made using tractor-mounted or backpack sprayers with 1–4 shielded nozzle minibooms.

Duplicate samples of mature fruit (min 12 units, 1.8 kg). In three trials, samples were quartered or halved in the field. Samples were frozen within 25 minutes and analysed for

flumioxazin within 12 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 70–120% (except for one recovery at 130%) and the validated LOQ was 0.02 mg/kg.

Table 56 Residues in summer squash from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

SUMMER SQUASH COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2004 Citra, FL (Gentry)	2	0.14 0.142	235 238	0.282	whole fruit	30	< 0.02, < 0.02	< 0.02	SBR-0120 FL12
USA, 2004 Davis, CA (Straight Neck Early Prolific)	2	0.145 0.141	292 318	0.286	whole fruit	14	< 0.02, < 0.02	< 0.02	SBR-0120 CA30
USA, 2004 Freeville, NY (Revune)	2	0.151 0.144	301 288	0.295	whole fruit	34	< 0.02, < 0.02	< 0.02	SBR-0120 NY04
USA, 2004 Holt, MI (Black Beauty)	2	0.144 0.149	193 200	0.294	whole fruit	16	< 0.02, < 0.02	< 0.02	SBR-0120 MI03
USA, 2004 Prosser, WA (Early Summer Crookneck)	2	0.139 0.141	273 277	0.28	whole fruit	25	< 0.02, < 0.02	< 0.02	SBR-0120 WA03
USA, 2004 Salisbury, MD (Seneca Prolific)	2	0.144 0.145	133 134	0.289	whole fruit	7	< 0.02, < 0.02	< 0.02	SBR-0120 MD03
USA, 2004 Tifton, GA (Crookneck Early Summer)	2	0.144 0.142 [a]	193 191	0.286	whole fruit	11	< 0.02, < 0.02	< 0.02	SBR-0120 GA*02
USA, 2004 Wesalco, TX (Golide)	2	0.143 0.143 [a]	257 239	0.286	whole fruit	12	< 0.02, < 0.02	< 0.02	SBR-0120 TX08

[a] 2nd application after the start of flowering

Fruiting vegetables other than cucurbits

Supervised trials on fruiting vegetables other than cucurbits were conducted in the USA in 2003.

Peppers (sweet, chili)

In nine trials on peppers (bell and non-bell/chilli), two treatments of 0.14–0.15 kg ai flumioxazin/ha (WG formulations) with added adjuvant were applied as inter-row shielded applications, the first application at transplanting or shortly after emergence and the second application from 15–21 days before harvest. Treatments were made using tractor-mounted or backpack sprayers with 1–2 shielded nozzle minibooms.

Duplicate samples of mature fruit (min 12 units, 1.8 kg) were frozen within 6 hours and analysed for flumioxazin within 27 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 86–121% (0.02 mg/kg spike level) and 59–111% (0.2 mg/kg spike level). The validated LOQ was 0.02 mg/kg.

Table 57 Residues in peppers from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

PEPPERS COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
BELL PEPPER									
USA, 2003 Citra, FL (Camelot)	2	0.143 0.144	193 194	0.286	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0118 03-FL25
USA, 2003 Clinton, NC (Camelot)	2	0.14 0.136	194 188	0.276	whole fruit	19	< 0.02, < 0.02	< 0.02	SBR-0118 03-NC09
USA, 2003 Holtville, CA (Valiant)	2	0.143 0.146	108 110	0.29	whole fruit	20	< 0.02, < 0.02	< 0.02	SBR-0118 03-CA48
USA, 2003 Parlier, CA (Valiant)	2	0.145 0.143	149 147	0.287	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0118 03-CA49
USA, 2003 Tifton, GA (Capistrano)	2	0.144 0.145	192 194	0.289	whole fruit	15	< 0.02, < 0.02	< 0.02	SBR-0118 03-GA*10
USA, 2003 Wesalco, TX (Capistrano)	2	0.14 0.144	244 206	0.284	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0118 03-TX*16
NON-BELL PEPPER									
USA, 2003 Mesilla, NM (Big Jim Chile)	2	0.144 0.14	190 192	0.284	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0118 03-NM11
USA, 2003 Rocky Ford, CO (Joe Parker)	2	0.143 0.147	191 197	0.29	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0118 03-CO11
USA, 2003 Wesalco, TX (TAM Veracruz)	2	0.142 0.145	222 210	0.286	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0118 03-TX15

Tomato

In twelve trials on tomatoes, two treatments of 0.14–0.15 kg ai flumioxazin/ha (WG formulations) with added adjuvant were applied as inter-row shielded applications, the first application at transplanting or shortly after emergence and the second application from 15–21 days before harvest. Treatments were made using tractor-mounted or backpack sprayers with 1–2 shielded nozzle minibooms.

Duplicate samples of mature fruit (min 12 units, 1.8 kg) were frozen within 2.3 hours and analysed for flumioxazin within 7 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 70–120% (except for one recovery at 130%) and the validated LOQ was 0.02 mg/kg.

Table 58 Residues in tomatoes from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

TOMATO COUNTRY, YEAR	APPLICATION	MATRIX	DAT	RESIDUES (MG/KG)	REFERENCE & COMMENTS

LOCATION (VARIETY)	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2003 Arlington, WI (Capri VF)	2	0.145 0.142	272 264	0.287	whole fruit	19	< 0.02, < 0.02	< 0.02	SBR-0117 03-WI06
USA, 2003 Charleston, SC (Sunleaper)	2	0.138 0.136	271 237	0.273	whole fruit	15	< 0.02, < 0.02	< 0.02	SBR-0117 03-SC*03
USA, 2003 Citra, FL (FLA47)	2	0.146 0.142	197 192	0.288	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0117 03-FL23
USA, 2003 Citra, FL (FLA47)	2	0.147 0.14	198 189	0.287	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0117 03-FL24 not independent
USA, 2003 Davis, CA (Hypeel 303)	2	0.149 0.141	299 236	0.29	whole fruit	19	< 0.02, < 0.02	< 0.02	SBR-0117 03-CA155
USA, 2003 Freeville, NY (Celebrity)	2	0.15 0.14	290 259	0.284	whole fruit	20	< 0.02, < 0.02	< 0.02	SBR-0117 03-NY04
USA, 2003 Glenn, CA (H-8892)	2	0.141 0.143	208 258	0.284	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0117 03-CA45
USA, 2003 Madera, CA (Rio Grande)	2	0.141 0.145	236 242	0.286	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0117 03-CA46
USA, 2003 Parlier, CA (Heinz 3155)	2	0.144 0.139	235 247	0.282	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0117 03-CA44
USA, 2003 Parlier, CA (Quality 21)	2	0.145 0.141	150 154	0.287	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0117 03-CA43
USA, 2003 Porterville, CA (Better Boy)	2	0.138 0.14	239 236	0.278	whole fruit	21	< 0.02, < 0.02	< 0.02	SBR-0117 03-CA42 not independent
USA, 2003 Porterville, CA (UC82-L)	2	0.139 0.139	234 233	0.278	whole fruit		< 0.02, < 0.02	< 0.02	SBR-0117 03-CA41

Pulses

Supervised trials on pulses (beans, peas and soya beans) were conducted in North America between 1989 and 2009.

Beans (dry)

In twelve trials on beans, one foliar broadcast spray of 0.1–0.11 kg ai flumioxazin/ha (WG formulations) with added adjuvant was applied as a pre-harvest desiccant and harvest aid using tractor-mounted or back-pack sprayers with 4–11 nozzle booms.

Duplicate samples were harvested, allowed to dry in the field for up to 13 days before being shelled (manually or mechanically) to obtain minimum samples of 1 kg dry seeds. These samples were frozen within 5 hours and analysed for flumioxazin within 10 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 79–119% and the validated LOQ was 0.02 mg/kg.

Table 59 Residues in beans, dry from supervised trials in the USA involving one pre-harvest foliar application of flumioxazin (WG formulation)

BEANS, DRY COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2004 Fargo, ND (Navigator)	1	0.104	111	0.104	seeds	5 + 2	< 0.02, < 0.02	< 0.02	SBR-0114 ND11
USA, 2004 Fort Collins, CO (Bill Z)	1	0.106	190	0.106	seeds	5 + 1	0.02, 0.02	0.02	SBR-0114 CO13
USA, 2004 Fort Collins, CO (Ohello)	1	0.108	194	0.108	seeds	4 + 3	0.02, < 0.02	0.02	SBR-0114 CO12
USA, 2004 Freeville, NY (Cabarnet)	1	0.107	286	0.107	seeds	6 + 8	< 0.02, < 0.02	< 0.02	SBR-0114 NY18
USA, 2004 Fremont, OH (Midnight Black)	1	0.107	324	0.107	seeds	4 + 13	< 0.02, < 0.02	< 0.02	SBR-0114 OH*12
USA, 2004 Fremont, OH (Topaz)	1	0.106	323	0.106	seeds	4 + 13	< 0.02, < 0.02	< 0.02	SBR-0114 OH*13 not independent
USA, 2004 Holtville, CA (Apache)	1	0.106	162	0.106	seeds	5	< 0.02, < 0.02	< 0.02	SBR-0114 CA128
USA, 2004 Kimberly, ID (Othello)	1	0.102	183	0.102	seeds	5 + 10	0.02, < 0.02	0.02	SBR-0114 ID09
USA, 2004 Minot, ND (Maverick)	1	0.106	94	0.106	seeds	4 + 3	< 0.02, < 0.02	< 0.02	SBR-0114 ND09 not independant
USA, 2004 Minot, ND (Maverick)	1	0.103	93	0.103	seeds	4 + 3	0.02, < 0.02	0.02	SBR-0114 ND10
USA, 2004 Minot, ND (Maverick)	1	0.104	93	0.104	seeds	4	< 0.02, < 0.02	< 0.02	SBR-0114 ND14
USA, 2004 Scottsbluff, NE (Beryl)	1	0.106	205	0.106	seeds	6 + 6	0.04, 0.03	0.04	SBR-0114 NE03 not independent
USA, 2004 Scottsbluff, NE (Kelly Bean 99124)	1	0.103	203	0.103	seeds	6 + 6	0.04, 0.05	0.05	SBR-0114 NE04

DAT = Interval from last application to cutting + field drying interval (in days)

Peas (dry)

In thirteen trials on field peas, one foliar broadcast spray of 0.11 kg ai flumioxazin/ha (WG formulations) with added adjuvant was applied as a pre-harvest desiccant and harvest aid using tractor-mounted or back-pack sprayers with 4–8 nozzle booms.

Duplicate plant samples were collected using small plot combines or cut and harvested using a stationary combine to obtain minimum samples of 1 kg dry seeds. These samples were frozen within 6 hours and analysed for flumioxazin within 4.5 months of harvest using method

RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 96–112% and the validated LOQ was 0.02 mg/kg.

Table 60 Residues in peas, dry from supervised trials in North America involving one pre-harvest foliar application of flumioxazin (WG formulation)

PEAS, DRY COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
Canada, 2009 Blaine Lake, Saskatchewan (Golden)	1	0.106	199	0.106	seeds	5	< 0.02, < 0.02	< 0.02	SBR-0124 V-32901-H
Canada, 2009 Boissevain, Manitoba (Golden)	1	0.106	158	0.106	seeds	5	< 0.02, < 0.02	< 0.02	SBR-0124 V-32901-F
Canada, 2009 Carberry, Manitoba (Golden)	1	0.105	195	0.105	seeds	6	0.03, 0.01	0.02	SBR-0124 V-32901-A
Canada, 2009 Elgin, Manitoba (Golden)	1	0.109	162	0.109	seeds	6	< 0.02, < 0.02	< 0.02	SBR-0124 V-32901-I
Canada, 2009 Hepburn, Saskatchewan (Golden)	1	0.111	184	0.111	seeds	6	< 0.02, < 0.02	< 0.02	SBR-0124 V-32901-G
Canada, 2009 Justice, Manitoba (Golden)	1	0.108	200	0.108	seeds	6	0.03, 0.02	0.03	SBR-0124 V-32901-B
Canada, 2009 Waldheim, Saskatchewan (Admiral)	1	0.107	201	0.107	seeds	6	< 0.02, < 0.02	< 0.02	SBR-0124 V-32901-D
USA, 2009 Northwood, ND (Admiral)	1	0.107 +NIS	184	0.107	seeds	5	< 0.02, < 0.02	< 0.02	SBR-0125 V-32857-A
	1	0.108 +MSO	186	0.108	seeds	5	< 0.02, < 0.02	< 0.02	
USA, 2009 Norwich, ND (Golden)	1	0.109	140	0.109	seeds	4	< 0.02, < 0.02	< 0.02	SBR-0125 V-32857-C
	1	0.216	141	0.216	seeds	4	0.02, 0.02	0.02	
USA, 2009 Parkdale, OR (Bluebird)	1	0.112	188	0.112	seeds	5	0.04, 0.02	0.03	SBR-0125 V-32857-E
USA, 2009 Payette, ID (Austrian Winter Pea)	1	0.108	186	0.108	seeds	5+2	0.05, 0.07	0.06	SBR-0125 V-32857-F
	1	0.216	188	0.216	seeds	5	0.07, 0.09	0.08	

PEAS, DRY COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Sharon, ND (Admiral)	1	0.108	186	0.108	seeds	1 3 5 7	0.02, 0.03 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	0.03 < 0.02 <u>≤ 0.02</u> < 0.02	SBR-0125 V-32857-B
USA, 2009 Velva, ND, (Golden)	1	0.109 +NIS	141	0.109	seeds	4	0.02, 0.02	0.02	SBR-0125 V-32857-D
	1	0.11 +MSO	141	0.11	seeds	4	0.02, 0.02	0.02	

DAT = Interval from last application to cutting + field drying interval (in days)

NIS = Non-ionic surfactant

MSO = Methylated seed oil surfactant

Soya beans

In supervised trials on <u>soya beans</u> conducted in the USA between 1989 and 1993, single broadcast soil applications of 0.1–0.11 kg ai flumioxazin/ha (WG, FL or WP formulations) were applied using back-pack or tractor-mounted boom sprayers, either as pre-plant treatments (with or without soil incorporation) or just after sowing, before crop emergence.

Duplicate samples of seed (min 1 kg) were frozen within 24 hours and stored for up to 9 months before analysis for flumioxazin using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 mg/kg ranged from 71–112% in seeds, with a validated LOQ of 0.02 mg/kg.

In the trials conducted in 1992–93, seeds were also analysed for the 1-OH-HPA metabolite, using method RM 30M (GC-MS), with an LOQ of 0.02 mg/kg and recovery rates of 71–100% in samples spiked with 0.02 mg/kg.

Table 61 Residues in soya bean seeds from supervised trials in the USA involving one broadcast pre-plant or pre-emergence soil application of flumioxazin

SOYA BEAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	TYPE	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1989 Dallas Center, IA (Asgrow 1937)	PE	0.101	94	0.101	seed	139	< 0.02, < 0.02	< 0.02	SBR-0003 T-7262
USA, 1989 Dallas Center, IA (Wells II)	PE	0.101	187	0.101	seed	132	< 0.02, < 0.02	< 0.02	SBR-0003 T-7370 no cultivation
USA, 1989 Geneseo, IL (Pioneer 9271)	PE	0.101	187	0.101	seed	133	< 0.02, < 0.02	< 0.02	SBR-0003 T-7374
USA, 1989 Greenville, MS (Forrest)	PE	0.101	187	0.101	seed	141	< 0.02, < 0.02	< 0.02	SBR-0003 T-7373
USA, 1989 Hollandale, MN (NK523-12)	PE	0.101	187	0.101	seed	123	< 0.02, < 0.02	< 0.02	SBR-0003 T-7260
USA, 1989 Lanoke, AR (Asgrow 5980)	PE	0.101	94	0.101	seed	131	< 0.02, < 0.02	< 0.02	SBR-0003 T-7263
USA, 1989 Leonard, MO (Williams 82)	PE	0.101	374	0.101	seed	128	< 0.02, < 0.02	< 0.02	SBR-0003 T-7368
USA, 1989 Metcalfe, MS (Forrest)	PE	0.101	187	0.101	seed	128	< 0.02, < 0.02	< 0.02	SBR-0003 T-7375
USA, 1989 New Holland, OH (Pioneer 9361)	PE	0.101	365	0.101	seed	128	< 0.02, < 0.02	< 0.02	SBR-0003 T-7369
USA, 1989 Noblesville IN (Pioneer 9361)	PE	0.101	206	0.101	seed	138	< 0.02, < 0.02	< 0.02	SBR-0003 T-7261
USA, 1989 Rosa, LA (Forrest)	PE	0.101	212	0.101	seed	149	< 0.02, < 0.02	< 0.02	SBR-0003 T-7372
USA, 1989 York, NE (Hack)	PE	0.101	187	0.101	seed	138	< 0.02, < 0.02	< 0.02	SBR-0003 T-7371
USA, 1990 Clarence, MO (Williams 82)	PE	0.101	187	0.101	seed	126	< 0.02, < 0.02	< 0.02	SBR-0003 T-7512
USA, 1990 Cloverport, TN (FFR 562)	PE	0.101	187	0.101	seed	121	< 0.02, < 0.02	< 0.02	SBR-0003 T-7501
USA, 1990 Dallas Center, IA (Asgrow 2187)	PE	0.101	187	0.101	seed	136	< 0.02, < 0.02	< 0.02	SBR-0003 T-7507
USA, 1990 Dallas Center, IA (Asgrow 2187)	PP	0.101	187	0.101	seed	131	< 0.02, < 0.02	< 0.02	SBR-0003 T-7509
USA, 1990 Elwood, IL (Pioneer 9202)	PP	0.101	196	0.101	seed	138	< 0.02, < 0.02	< 0.02	SBR-0003 T-7508

SOYA BEAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	TYPE	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1990 Geneseo, IL (Pioneer 9272)	PE	0.101	187	0.101	seed	111	< 0.02, < 0.02	< 0.02	SBR-0003 T-7502
USA, 1990 Greenville, MS (Forrest)	PE	0.101	187	0.101	seed	130	< 0.02, < 0.02	< 0.02	SBR-0003 T-7506
USA, 1990 Hollandale, MN (Agri Pro 1776)	PE	0.101	187	0.101	seed	133	< 0.02, < 0.02	< 0.02	SBR-0003 T-7511
USA, 1990 Hollendale, MN (Agri Pro1776)	PE	0.101	187	0.101	seed	133	< 0.02, < 0.02	< 0.02	SBR-0003 T-7500 no cultivation
USA, 1990 New Holland, OH (Pioneer 9391)	PE	0.101	243	0.101	seed	128	< 0.02, < 0.02	< 0.02	SBR-0003 T-7510
USA, 1990 Noblesville, IN (Pioneer 9361)	PE	0.101	253	0.101	seed	125	< 0.02, < 0.02	< 0.02	SBR-0003 T-7503
USA, 1990 Proctor, AR (DPL 105)	PE	0.101	187	0.101	seed	140	< 0.02, < 0.02	< 0.02	SBR-0003 T-7513
USA, 1992 Goldsboro, NC (Ransom)	PP	0.105	187	0.105	seed	154	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-A
USA, 1992 Greenville, MS (Pioneer 9641)	PE	0.105	187	0.105	seed	127	<< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-H
USA, 1992 Leonard, MO (Pioneer 9443)	PP	0.102	187	0.102	seed	126	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-M
USA, 1992 Little Rock, AR (Hutcheson)	PP	0.105	187	0.105	seed	146	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-C
USA, 1992 New Holland, OH (GL 2910)	PE	0.105	150	0.105	seed	132	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-G
USA, 1992 Noblesville, IN (Pioneer 9361)	PP	0.105	234	0.105	seed	131	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-D
USA, 1992 Seymour, IL (Asgrow 2543)	PP	0.105	187	0.105	seed	130	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-B
USA, 1992 Seymour, IL (Asgrow 2543)	PE	0.105	187	0.105	seed	126	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-J
	PE	0.526	187	0.526	seed	126	< 0.02, < 0.02	< 0.02	
USA, 1992 Waukee, IA (Asgrow 2543)	PP	0.105	187	0.105	seed	129	< 0.02, < 0.02	< 0.02	SBR-0021 V-1039-F
USA, 1993 Jamesville, NC (Hutcheson)	PP	0.108	253	0.108	seed	160	< 0.02, < 0.02	< 0.02	SBR-0021 V-10719-A

SOYA BEAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	TYPE	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1993 Leonard, MO (Linford)	PP	0.107	271	0.107	seed	123	< 0.02, < 0.02	< 0.02	SBR-0021 V-10719-E
USA, 1993 Noblesville, IN (Pioneer 9361)	PP	0.11	206	0.11	seed	138	< 0.02, < 0.02	< 0.02	SBR-0021 V-10719-D
USA, 1993 Seymour, IL (Asgrow 2506)	PE	0.536	187	0.536	seed	112	< 0.02, < 0.02	< 0.02	SBR-0021 V-10719-K
USA, 1993 Theilman, MN (Pioneer 9061)	PP	0.107	187	0.107	seed	160	< 0.02, < 0.02	< 0.02	SBR-0021 V-10719-B
USA, 1993 Webster City, IA (L-1700)	PP	0.108	206	0.108	seed	112	< 0.02, < 0.02	< 0.02	SBR-0021 V-10719-F
USA, 1993 York, NE (Hack)	PP	0.107	187	0.107	seed	126	< 0.02, < 0.02	< 0.02	SBR-0021 V-10719-C

The 1992–1993 supervised trials also analysed for residues of the metabolite, 1-OH HPA in seeds. Residues in all samples were below the LOQ of 0.02 mg/kg (18 trials).

PE = pre-emergence application (within 5 days after sowing)

PP = pre-plant application

Root and tuber vegetables

Potato

In supervised trials on potatoes (14) conducted in the USA during 2001, single broadcast soil applications of 0.13–0.15 kg ai flumioxazin/ha (WG formulations) were applied using back-pack, wheeled or tractor-mounted sprayers with 2–12 nozzle booms after the last hilling operation, before potato emergence. In several trials, transitory phytotoxicity and stunting was observed.

Duplicate samples of at least 1.8 kg potatoes were wiped, brushed or rinsed to remove adhering soil, frozen within 2.5 hours and analysed for flumioxazin within 9 months of harvest using method RM 30A-2 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 77–118% and the validated LOQ was 0.02 mg/kg.

Table 62 Residues in potatoes from supervised trials in the USA involving one broadcast pre-emergent soil application of flumioxazin (WG formulations)

POTATO COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2001 Aberdeen, ID (Russet Burbank)	1	0.138	279	0.138	Tuber	118	< 0.02, < 0.02	< 0.02	SBR-0091 ID01
USA, 2001 Clinton, NC (Atlantic)	1	0.138	184	0.138	Tuber	62	< 0.02, < 0.02	< 0.02	SBR-0091 NC05
USA, 2001 E. Corinth, ME (Atlantic)	1	0.132	177	0.132	Tuber	105	< 0.02, < 0.02	< 0.02	SBR-0091 ME01

POTATO COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2001 Fort Collins, CO (Russet Norkotah)	1	0.139	183	0.139	Tuber	96	< 0.02, < 0.02	< 0.02	SBR-0091 CO02
USA, 2001 Fort Collins, CO (Russet Norkotah)	1	0.139	182	0.139	Tuber	91	< 0.02, < 0.02	< 0.02	SBR-0091 CO03 not independent
USA, 2001 Freemont, OH (Yukon Gold)	1	0.148	258	0.148	Tuber	92	< 0.02, < 0.02	< 0.02	SBR-0091 OH*03
USA, 2001 Freeville, NY (Atlantic)	1	0.127	254	0.127	Tuber	111	< 0.02, < 0.02	< 0.02	SBR-0091 NY03
USA, 2001 Gainesville, FL (Red La Soda)	1	0.123	247	0.123	Tuber	67	< 0.02, < 0.02	< 0.02	SBR-0091 FL09
USA, 2001 Holtville, CA (California White)	1	0.141	309	0.141	Tuber	104	< 0.02, < 0.02	< 0.02	SBR-0091 CA09
USA, 2001 Prosper, ND (Red La Soda)	1	0.14	156	0.14	Tuber	101	< 0.02, < 0.02	< 0.02	SBR-0091 ND04
USA, 2001 Prosper, ND (Russet Burbank)	1	0.147	164	0.147	Tuber	101	< 0.02, < 0.02	< 0.02	SBR-0091 ND05 not independent
USA, 2001 Prosser, WA (Russet Burbank)	1	0.147	252	0.147	Tuber	126	< 0.02, < 0.02	< 0.02	SBR-0091 WA05
USA, 2001 Prosser, WA (Russet Burbank)	1	0.141	268	0.141	Tuber	126	< 0.02, < 0.02	< 0.02	SBR-0091 WA06 not independent
USA, 2001 Prosser, WA (Russet Burbank)	1	0.141	149	0.141	Tuber	107	< 0.02, < 0.02	< 0.02	SBR-0091 WA*07

Stem and stalk vegetables

Supervised trials on stem and stalk vegetables (asparagus, Globe artichoke and celery) were conducted in North America between 2003 and 2007.

Artichoke, Globe

In three supervised trials on Globe artichokes, single broadcast soil applications of 0.21 kg ai flumioxazin/ha (WG formulations) were applied 1–4 days before transplanting using back-pack sprayers with hand-held 5-nozzle minibooms.

Duplicate samples of flower heads (12 units, min 2.7 kg) were frozen within 1 hour and analysed for flumioxazin within 7.5 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 70–115% and the validated LOQ was 0.02 mg/kg.

Table 63 Residues in Globe artichokes from supervised trials in the USA involving one broadcast pre-plant soil application of flumioxazin (WG formulations)

ARTICHOKE,	APPLICATION	MATRIX	DAT	RESIDUES (MG/KG)	REFERENCE &

GLOBE COUNTRY, YEAR LOCATION (VARIETY)	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	COMMENTS
GAP:USA		0.21	94–280	0.21			Before planting or cut-back		
USA, 2007 Castroville, CA (F1 1855)	1	0.214	280	0.214	Head	147	< 0.02, < 0.02	< 0.02	SBR-0128 CA37
USA, 2007 Watsonville, CA (F1 41)	1	0.21	367	0.21	Head	134	< 0.02, < 0.02	< 0.02	SBR-0128 CA38
USA, 2007 Castroville, CA (F1 1855)	1	0.214	468	0.214	Head	126	< 0.02, < 0.02	< 0.02	SBR-0128 CA39

Asparagus

In eight supervised trials on asparagus, single broadcast soil applications of 0.21–0.22 or 0.43–0.45 kg flumioxazin/ha (WG formulations) were applied using back-pack, ATV or tractor-mounted 3–6 nozzle booms about 2 weeks before spear emergence. Phytotoxicity was observed in several trials.

Duplicate samples of at least 1.3 kg spears were brushed (if necessary) to remove adhering soil, frozen within 4.5 hours and analysed for flumioxazin within 3.5 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 90–114% and the validated LOQ was 0.02 mg/kg.

Table 64 Residues in asparagus from supervised trials in the USA involving one broadcast soil application of flumioxazin (WG formulations)

ASPARAGUS COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
GAP:USA		0.21	140–280	0.21		14	Pre-emergent		
USA, 2004 Porterville, CA (UC157)	1	0.22	231	0.22	spears	15	< 0.02, < 0.02	< 0.02	SBR-0116 CA74
		0.43	229	0.43	spears	15	< 0.02, < 0.02	< 0.02	
USA, 2004 Stockton, CA (UC157)	1	0.22	198	0.22	spears	14	< 0.02, < 0.02	< 0.02	SBR-0116 CA75 min 0.5kg sample
		0.43	198	0.43	spears	14	< 0.02, < 0.02	< 0.02	
USA, 2004 Stockton, CA (UC157)	1	0.22	295	0.22	spears	14	< 0.02, < 0.02	< 0.02	SBR-0116 CA76
		0.44	924	0.44	spears	14	< 0.02, < 0.02	< 0.02	
USA, 2003 Holt, MI (Jersey Knight)	1	0.22	192	0.22	spears	14	< 0.02, < 0.02	< 0.02	SBR-0116 MI23
		0.43	191	0.43	spears	14	< 0.02, < 0.02	< 0.02	
USA, 2003 East Lansing, MI (Jersey Giant)	1	0.22	193	0.22	spears	14	< 0.02, < 0.02	< 0.02	SBR-0116 MI24
		0.44	196	0.44	spears	14	< 0.02, < 0.02	< 0.02	
USA, 2003 Bridgeton, NJ (New Jersey hybrids)	1	0.22	217	0.22	spears	15	< 0.02, < 0.02	< 0.02	SBR-0116 NJ17
		0.45	227	0.45	spears	15	< 0.02, < 0.02	< 0.02	
USA, 2003 Prosser, WA (Jersey Giant)	1	0.21	343	0.21	spears	15	< 0.02, < 0.02	< 0.02	SBR-0116 WA09
		0.43	343	0.43	spears	15	< 0.02, < 0.02	< 0.02	

ASPARAGUS COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2003 Moxee, WA (Mary Washington)	1	0.22	261	0.22	spears	20	< 0.02, < 0.02	< 0.02	SBR-0116 WA*10
		0.44	260	0.44	spears	20	< 0.02, < 0.02	< 0.02	

Celery

In eight supervised trials on celery, single broadcast soil applications of 0.1–0.11 or 0.2–0.22 kg ai flumioxazin/ha (WG formulations) were applied 0–2 days before transplanting using back-pack plot sprayers with 3–4 nozzle minibooms or tractor-mounted 9-nozzle boom sprayers. Phytotoxicity was reported in several of the high-rate plots.

Duplicate samples of 12 untrimmed bunches (12 units, min 1.8 kg) were brushed or rinsed if necessary to remove adhering soil, frozen within 5 hours and analysed for flumioxazin within 9 months of harvest using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 90–120% (except for one recovery at 150%) and the validated LOQ was 0.02 mg/kg.

Table 65 Residues in celery from supervised trials in the USA involving one broadcast pre-plant soil application of flumioxazin (WG formulations)

CELERY COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
GAP:USA		0.105	140–280	0.105			Before or 3–7 days after transplanting		
USA, 2004 Citra, FL (M-9)	1	0.105	278	0.105	Stalk	104	< 0.02, < 0.02	< 0.02	SBR-0122 FL10
	1	0.212	282	0.212	Stalk	104	< 0.02, < 0.02	< 0.02	
USA, 2004 Citra, FL (M-9)	1	0.107	285	0.107	Stalk	108	< 0.02, < 0.02	< 0.02	SBR-0122 FL11
	1	0.216	287	0.216	Stalk	108	< 0.02, < 0.02	< 0.02	
USA, 2004 Laingsburg, MI (Dutchess)	1	0.107	191	0.107	Stalk	73	< 0.02, < 0.02	< 0.02	SBR-0122 MI02 subsampled in the field
	1	0.221	196	0.221	Stalk	73	< 0.02, < 0.02	< 0.02	
USA, 2004 Salinas, CA (Dutchess)	1	0.112	367	0.112	Stalk	98	< 0.02, < 0.02	< 0.02	SBR-0122 CA*18 subsampled in the field
	1	0.214	358	0.214	Stalk	98	< 0.02, < 0.02	< 0.02	
USA, 2004 Paso Robles, CA (Conquistado)	1	0.107	283	0.107	Stalk	95	< 0.02, < 0.02	< 0.02	SBR-0122 CA19
	1	0.204	272	0.204	Stalk	95	< 0.02, < 0.02	< 0.02	
USA, 2004 Camarillo, CA (BSM2)	1	0.107	283	0.107	Stalk	127	< 0.02, < 0.02	< 0.02	SBR-0122 CA20
	1	0.211	282	0.211	Stalk	127	< 0.02, < 0.02	< 0.02	
USA, 2004 Irvine, CA (Conquistador 1703)	1	0.105	233	0.105	Stalk	112	< 0.02, < 0.02	< 0.02	SBR-0122 CA21
	1	0.21	235	0.21	Stalk	112	< 0.02, < 0.02	< 0.02	
USA, 2004 Salinas, CA (Challenger)	1	0.104	318	0.104	Stalk	90	< 0.02, < 0.02	< 0.02	SBR-0122 CA*22 subsampled in the field
	1	0.21	322	0.21	Stalk	90	< 0.02, < 0.02	< 0.02	

Cereal grains

Supervised trials on cereal grains (maize and wheat) were conducted in North America between 2005 and 2010.

Maize

In twenty-one supervised trials on maize, single broadcast soil applications of 0.1–0.11 or 0.2–0.22 kg ai flumioxazin/ha (WG formulations) with added surfactant were applied 6–14 days before sowing, using back-pack plot sprayers, wheeled or tractor-mounted boom sprayers (3–9 nozzles).

Duplicate samples of kernels (min 1 kg) were taken at maturity, frozen within 2 hours and analysed for flumioxazin within 14 months using method RM 30A-3 (GC-MS) in the 2005 trials and method NCL 293 (HPLC-MS/MS) in the 2006 trials. Recoveries from control kernel samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 85–122% in the two methods and the validated LOQ was 0.02 mg/kg.

Table 66 Residues in maize from supervised trials in North America involving one broadcast pre-plant soil application of flumioxazin (WG formulations)

MAIZE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
GAP:USA		0.105	140–280	0.105			14–30 days before sowing		
USA, 2005 New Holland, OH (Syngenta N73-F7)	1	0.107	191	0.107	grain	148	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-A
	1	0.211	188	0.211	grain	148	< 0.02, < 0.02	< 0.02	
USA, 2005 Carlyle, IL (FS 6455)	1	0.107	190	0.107	grain	171	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-B
	1	0.212	187	0.212	grain	171	< 0.02, < 0.02	< 0.02	
USA, 2005 Clarence, MO (Pioneer 35P12)	1	0.107	191	0.107	grain	154	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-C
USA, 2005 Greenville, MS (69-71 757 HXJINX)	1	0.104	185	0.104	grain	135	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-D
USA, 2006 North Rose, NY (Dairyland Stealth 8711)	1	0.108	191	0.108	grain	131	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-E
USA, 2006 Elko, SC (Pioneer 31R87)	1	0.105	180	0.105	grain	158	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-F
Canada, 2006 City of Hamilton, Ontario (Pioneer 38B84)	1	0.107	187	0.107	grain	166	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-G
	1	0.211	184	0.211	grain	166	< 0.02, < 0.02	< 0.02	
USA, 2006 Conklin, MI (N45-M2 Field Corn)	1	0.106	189	0.106	grain	138	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-H
USA, 2006 Carlyle, IL (DKC-65-16)	1	0.108	185	0.108	grain	168	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-I
USA, 2006 Bellmore, IN (Wyffels 5531)	1	0.104	185	0.104	grain	136	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-J
USA, 2006 York, NE (NK N70-F1)	1	0.106	184	0.106	grain	155	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-K
USA, 2006 Richland, IA (Pioneer 33P65)	1	0.105	189	0.105	grain	151	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-L
USA, 2006 Geneva, MN (Pioneer 38H66)	1	0.106	180	0.106	grain	156	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-M
USA, 2006 Fairmount, ND (Dekalb 35-02)	1	0.106	188	0.106	grain	145	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-N

MAIZE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2006 Campbell, MN (Pioneer 39H83)	1	0.106	188	0.106	grain	155	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-O
USA, 2006 Hudson, KS (Midwest Seed Genetics 8127RB)	1	0.106	188	0.106	grain	134	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-P
Canada, 2006 Portage la Prairie, Manitoba (Roundup Ready-Monsanto)	1	0.102	181	0.102	grain	154	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-Q
USA, 2006 Arkansaw, WI (Pioneer 38B85)	1	0.106	188	0.106	grain	137	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-R
Canada, 2006 St. Pie, Quebec (NK 3030 BT)	1	0.101	176	0.101	grain	156	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-S
USA, 2006 Dill City, OK (DKC48-53)	1	0.107	193	0.107	grain	130	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-T
USA, 2006 Clarence, MO (Pioneer 34B20)	1	0.107	187	0.107	grain	165	< 0.02, < 0.02	< 0.02	SBR-0078 V-28566-U

Wheat

In twenty supervised trials on wheat, single foliar broadcast sprays of 0.07–0.075 kg ai flumioxazin/ha (WG formulations) with added adjuvants were applied as pre-harvest desiccants and harvest aids using tractor-mounted or back-pack sprayers with 4–8 nozzle booms.

Duplicate samples were collected using small plot combines or cut and harvested using a stationary combine to obtain minimum samples of 1 kg dry seeds. Samples were frozen within 5 hours and analysed for flumioxazin within 17 months of harvest using method RM 30A-3 (GC-MS). Concurrent recoveries from control grain samples fortified with flumioxazin at levels of 0.02–0.5 mg/kg ranged from 70–122% and the validated LOQ was 0.02 mg/kg.

Table 67 Residues in wheat grain from supervised trials in the USA involving one pre-harvest foliar application of flumioxazin (WG formulations)

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Lexington, GA (USG 3592)	1	0.071 + NIS	193	0.071	grain	10	0.03, 0.04	0.04	SBR-0092 V-33037-A
	1	0.071 + MSO	192	0.071	grain	10	0.04, 0.06	0.05	
USA, 2009 Leland, MS (Gore)	1	0.071 + MSO	185	0.071	grain	3 7 10 13	0.04, 0.08 0.05, 0.05 0.11, 0.11 0.07, 0.11	0.06 0.05 0.11 0.09	SBR-0092 V-33037-B
USA, 2009	1	0.072 + MSO	199	0.072	grain	10	0.07, 0.08	0.08	SBR-0092

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
Carlyle, IL (Branson)	1	0.145 + MSO	200	0.145	grain	10	0.22, 0.24	0.23	V-33037-C
USA, 2009 York, NE (Traverse Hard red Spring)	1	0.071 + NIS	184	0.071	grain	10	0.04, 0.05	0.05	SBR-0092 V-33037-D
	1	0.071 + MSO	186	0.071	grain	10	0.03, 0.04	0.04	
USA, 2009 Rockville, IN (Becks 164)	1	0.072 + NIS	148	0.072	grain	11	0.08, 0.13	0.11	SBR-0092 V-33037-E
	1	0.072 + MSO	148	0.072	grain	11	0.07, 0.09	0.08	
USA, 2009 Clarence, MO (Ernie)	1	0.074 + NIS	193	0.074	grain	10	0.11, 0.15	0.13	SBR-0092 V-33037-F
	1	0.07 + MSO	183	0.07	grain	10	0.09, 0.13	0.11	
USA, 2009 Bagley, IA (Briggs hrS)	1	0.071 + NIS	148	0.071	grain	10	0.18, 0.28	0.23	SBR-0092 V-33037-G
	1	0.072 + MSO	150	0.072	grain	10	0.13, 0.19	0.16	
USA, 2009 Ulvade, TX (Fannin)	1	0.07 + NIS	138	0.07	grain	9	0.11, 0.12	0.12	SBR-0092 V-33037-H
	1	0.072 + MSO	142	0.072	grain	9	0.08, 0.1	0.09	
USA, 2009 Grand Island, NE (Traverse Hard Red Spring)	1	0.072 + NIS	189	0.072	grain	10	0.05, 0.07	0.06	SBR-0092 V-33037-I not independent
	1	0.071+MSO	186	0.071	grain	10	0.06, 0.06	0.06	
USA, 2009 Velva, ND (Faller)	1	0.072 + MSO	141	0.072	grain	10	0.06, 0.08	0.07	SBR-0092 V-33037-J
USA, 2009 Grand Island, NE (Kelby Hard Red Spring)	1	0.071 + NIS	185	0.071	grain	10	0.08, 0.09	0.09	SBR-0092 V-33037-K
USA, 2009 Norwich, ND (Faller)	1	0.072 + MSO	172	0.072	grain	10	0.09, 0.11	0.1	SBR-0092 V-33037-L
	1	0.146 + MSO	143	0.146	grain	10	0.13, 0.14	0.14	
USA, 2009 Malta, MT (McNeal)	1	0.069 + NIS	181	0.069	grain	10	0.3, 0.31	0.31	SBR-0092 V-33037-AM
USA, 2009 Levelland, TX (TAM 105)	1	0.072 + NIS	189	0.072	grain	9	0.1, 0.16	0.13	SBR-0092 V-33037-N
USA, 2009 Wellington, TX (TAM 111)	1	0.072 + MSO	165	0.072	grain	9	0.03, 0.06	0.05	SBR-0092 V-33037-O
	1	0.142 + MSO	163	0.142	grain	9	0.03, 0.05	0.04	
USA, 2009 Larned, KS (Jagger)	1	0.074 + NIS	212	0.074	grain	10	0.07, 0.13	0.10	SBR-0092 V-33037-P
USA, 2009 Hinton, OK (Jagger)	1	0.07 + MSO	164	0.07	grain	4 7 10 13	0.07, 0.09 0.16, 0.16 0.06, 0.08 0.15, 0.2	0.08 0.16 0.07 <u>0.18</u>	SBR-0092 V-33037-Q
USA, 2009 Cordell, OK (Fuller)	1	0.072 + MSO	172	0.072	grain	11	0.07, 0.12	0.1	SBR-0092 V-33037-R
USA, 2009 Jerome, ID (AC Andrew)	1	0.071 + NIS	181	0.071	grain	10	0.04, 0.05	0.05	SBR-0092 V-33037-S
USA, 2009	1	0.071 + MSO	165	0.071	grain	10	0.05, 0.06	0.06	SBR-0092

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
Hinton, OK (Deliver)	1	0.344 + MSO	152	0.344	grain	10	0.37, 0.35	0.36	V-33037-T not independent

NIS = Non-ionic surfactant

MSO = Methylated seed oil surfactant

Grasses for sugar production

Sugar cane

In supervised trials on sugar cane (nine) conducted in the USA during 1998, single broadcast applications of 0.4–0.42 kg ai flumioxazin/ha (WG formulations) with added crop oil were applied over the top of 2–2.5 m high canes using back-pack sprayers with elevated 6-nozzle booms or extended single-nozzle hand lances.

Duplicate samples of at least 12 canes with leaves attached (min 5 kg) were frozen within 10 hours and analysed for flumioxazin within 5 months of harvest using method RM 30A-1 (GC-MS). Samples were also analysed within 7 months of harvest for the 1-OH-HPA metabolite using method RM-30C (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.01–0.5 mg/kg ranged from 67–113% and in samples fortified with 0.02–0.2 mg/kg 1-OH-HPA, recoveries were 70–114%. The validated LOQs for both compounds were both 0.02 mg/kg.

Table 68 Residues in sugar cane from supervised trials in the USA involving one broadcast foliar application of flumioxazin (WG formulation)

SUGAR CANE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1998 Clewiston, FL (CP-70-1133)	1	0.424	160	0.424	cane	89	< 0.02, < 0.02	< 0.02	SBR-0022 V-11945-A
USA, 1998 Clewiston, FL (CP-72-2086)	1	0.416	157	0.416	cane	89	0.02, 0.03	0.03	SBR-0022 V-11945-B
USA, 1998 Canal Point, FL (CP80-1827)	1	0.409	154	0.409	cane	89	0.04, < 0.02	0.03	SBR-0022 V-11945-C
USA, 1998 Clewiston, FL (CL77-79786)	1	0.421	159	0.421	cane	89	< 0.02, < 0.02	< 0.02	SBR-0022 V-11945-D
USA, 1998 Washington, LA (La 384)	1	0.423	143	0.423	cane	91	0.07, 0.11	0.09	SBR-0022 V-11945-E
USA, 1998 Raymondville, TX (1210)	1	0.415	139	0.415	cane	90	0.02, 0.09	0.06	SBR-0022 V-11945-F
USA, 1998 LeBeau, LA (La 384)	1	0.408	143	0.408	cane	90	0.07, 0.07	0.07	SBR-0022 V-11945-G

SUGAR CANE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1998 Spreckelsville, HI (78-4153)	1	0.421	187	0.421	cane	90	< 0.02, < 0.02	< 0.02	SBR-0022 V-11945-H
USA, 1998 Washington, LA (CP-845)	1	0.417	146	0.417	cane	90	0.03, 0.06	0.05	SBR-0022 V-11945-J
	1	1.254	147	1.254	cane	90	0.33, 0.14	0.23	

Residues of 1-OH-HPA < 0.02 mg/kg in all samples

Tree nuts

Supervised trials on tree nuts (almonds and pecans) were conducted in the US during 1999 and 2003, respectively.

Almonds

In supervised trials on almonds (five), two inter-row/berm broadcast soil treatments of 0.42 kg ai flumioxazin/ha (WG formulations) with added crop oil were applied using tractor-mounted 4–8 nozzle boom sprayers. Treatments were applied about 60 days apart, with the last application about 60 days before harvest.

Duplicate samples of mature nuts (min 1 kg) shaken from the trees, shelled in the field, frozen within 3 hours and analysed for flumioxazin within 6 months of harvest using method RM 30A-1 (GC-MS). Samples of almond hulls were also analysed within 8.5 months of harvest for the 1-OH-HPA metabolite using method RM-30M (GC-MS).

Recoveries from control samples fortified with flumioxazin at levels of 0.01 and 0.05 mg/kg ranged from 89–114% in nutmeat and 71–96% in hulls. In hull samples fortified with 0.1 or 0.5 mg/kg 1-OH-HPA, recoveries were 81–98%.The validated LOQs were 0.01 mg/kg (flumioxazin) and 0.1 mg/kg for the 1-OH-HPA metabolite.

Table 69 Residues in almonds (nutmeat and hulls) from supervised trials in the USA involving two broadcast soil application of flumioxazin (WG formulation)

ALMOND COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
GAP:USA		0.42	140–280	0.84		60	Directed inter-row band applications, min 60 day RTI		
USA, 1999 Chico, CA (Carmel)	2	0.419 0.425	168 168	0.844	nutmeat hulls	60	< 0.01, < 0.01 0.01, 0.01	< 0.01 0.01	SBR-0024 V-20116-A
	2	0.838 0.847	168 168	1.685	nutmeat hulls	60	< 0.01, < 0.01 0.03, 0.03	< 0.01 0.03	
USA, 1999 Hughson, CA (Carmel)	2	0.425 0.424	234 234	0.849	nutmeat hulls	60	< 0.01, < 0.01 0.49, 0.62	< 0.01 0.55	SBR-0024 V-20116-B
USA, 1999 Kerman, CA (Carmel)	2	0.417 0.419	187 187	0.836	nutmeat hulls	60	< 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01	SBR-0024 V-20116-C

ALMOND COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1999 Madera, CA (Non-pareil)	2	0.424 0.418	187 187	0.842	nutmeat hulls	60	< 0.01, < 0.01 0.04, 0.04	< 0.01 0.04	SBR-0024 V-20116-D
USA, 1999 Terra Bella CA (Carmel)	2	0.421 0.419	187 224	0.84	nutmeat hulls	60	< 0.01, < 0.01 0.06, 0.07	< 0.01 0.06	SBR-0024 V-20116-E

Residues of 1-OH-HPA all < 0.05 mg/kg in almond hulls

Pecans

In supervised trials on pecans (five), two inter-row/berm broadcast soil treatments of 0.42–0.43 kg ai flumioxazin/ha (WG formulations) were applied using knapsack or wheeled sprayers with 3–4 nozzle booms. Treatments were applied about 60 days apart, with the last application about 60 days before harvest.

Duplicate samples of mature nuts (min 1.2 kg) were shaken from the trees, shelled within 2 days of harvest, with nutmeat samples frozen within 6.25 hours of shelling and analysed for flumioxazin within 3.3 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 77–99% and the validated LOQ was 0.02 mg/kg.

Table 70 Residues in pecans from supervised trials in the USA involving one broadcast soil application of flumioxazin (WG formulation)

PECAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2003 Roseboro, NC (Pawnee)	2	0.419 0.426	290 299	0.845	nutmeat	59	< 0.02, < 0.02	< 0.02	SBR-0062 NC19
USA, 2003 Roseboro, NC (Kiowah)	2	0.422 0.427	299 309	0.849	nutmeat	61	< 0.02, < 0.02	< 0.02	SBR-0062 NC20
USA, 2003 Neches, TX (Desirable)	2	0.425 0.42	206 206	0.845	nutmeat	42	< 0.02, < 0.02	< 0.02	SBR-0062 TX31
USA, 2003 Shreveport, LA (Cape Fear)	2	0.421 0.42	206 206	0.841	nutmeat	61	< 0.02, < 0.02	< 0.02	SBR-0062 TX32
USA, 2003 Mesilla, NM (Western Shleigh)	2	0.419 0.433	196 215	0.852	nutmeat	61	< 0.02, < 0.02	< 0.02	SBR-0062 NM10

Oilseeds

Supervised trials on oilseeds (oilseed rape, cotton seed, sunflower seed and peanuts) were conducted in the USA between 1992 and 2009.

Cotton seed

In supervised trials on cotton seed (13), two foliar broadcast sprays of 0.1–0.11 kg ai flumioxazin/ha (WG formulations) with added crop oil were applied using tractor-mounted boom sprayers. The first applications were made about 90 days before harvest using shielded nozzles to minimise spray contact with the plants and the second applications were made about 60 days before harvest as directed inter-row sprays at layby, with spray contacting only the lower 5–10 cm cotton stems.

Duplicate samples of cotton seed, either ginned in the field (min 1 kg) or unginned (min 20 kg), were frozen within 4 hours (undelinted seed) or within 24 hours (unginned cotton). The cotton seed samples were stored frozen for up to 30 days before being ginned to separate the undelinted seed and gin trash and refrozen. All samples were analysed for flumioxazin within 3 months using method RM 30A-1 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.01 and 0.05 mg/kg ranged from 76–106% (cottonseed) and 70–102% in gin trash. The validated LOQs were 0.01 mg/kg for cotton seed and gin trash.

Samples of gin trash were also analysed within 8 months of harvest for the 1-OH-HPA metabolite using method RM-30M (GC-MS) with recoveries from control samples fortified with 1-OH-HPA at levels of 0.1 and 0.5 mg/kg ranging from 81–121% and the validated LOQ was 0.1 mg/kg.

Table 71 Residues in cotton seed and gin trash from supervised trials in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

COTTONSEED COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1999 Brookshire, TX (DPL 50B)	2	0.107 0.109	218 219	0.216	seed	60	< 0.01, < 0.01	< 0.01	SBR-0026 V-20124-E
USA, 1999 Greenville, MS (ST474)	2	0.105 0.106	186 190	0.211	seed gin trash	59 59	< 0.01, < 0.01 0.19, 0.3	< 0.01 0.25	SBR-0026 V-20124-L
USA, 1999 Greenville, MS (Stoneville 474)	2	0.107 0.106	148 143	0.213	seed gin trash	61 61	< 0.01, < 0.01 0.03, 0.03	< 0.01 0.03	SBR-0026 V-20124-C not independent
USA, 1999 Jamesville, NC (Stoneville 474)	2	0.117 0.107	236 258	0.224	seed gin trash	62 62	< 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01	SBR-0026 V-20124-A
USA, 1999 Kerman, CA (Maxxa)	2	0.109 0.112	193 198	0.221	seed	62	< 0.01, < 0.01	< 0.01	SBR-0026 V-20124-K
USA, 1999 Levelland, TX (PM 2200 RR)	2	0.106 0.106	187 187	0.212	seed gin trash	60	< 0.01, < 0.01 0.18, 0.13	< 0.01 0.16	SBR-0026 V-20124-H
USA, 1999 Littlefield, TX (DP 2379)	2	0.107 0.107	188 188	0.214	seed gin trash	61 61	< 0.01, 0.01 0.48, 0.48	0.01 0.48	SBR-0026 V-20124-F
USA, 1999 Madera, CA (Maxxa)	2	0.107 0.104	216 210	0.211	seed	60	< 0.01, 0.01	0.01	SBR-0026 V-20124-J
USA, 1999 Maricopa, AZ (Delta Pine 50B)	2	0.107 0.106	188 187	0.213	seed	60	< 0.01, < 0.01	< 0.01	SBR-0026 V-20124-I

COTTONSEED COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1999 Newport, AR (Paymaster 1220RR)	2	0.107 0.107	143 140	0.214	seed	60	< 0.01, < 0.01	< 0.01	SBR-0026 V-20124-B
USA, 1999 Ulvade, TX (PM 2326)	2	0.107 0.107	188 187	0.214	seed gin trash	59 59	< 0.01, < 0.01 0.03, 0.05	< 0.01 0.04	SBR-0026 V-20124-N
	2	0.213 0.211	190 188	0.424	seed gin trash	59 59	< 0.01, < 0.01 0.06, 0.1	< 0.01 0.08	
USA, 1999 Washington, LA (DLP Nuc.33B)	2	0.106 0.107	203 146	0.213	seed	60	< 0.01, < 0.01	< 0.01	SBR-0026 V-20124-D
USA, 1999 Wolfforth, TX (HS 26)	2	0.106 0.107	187 188	0.213	seed gin trash	62	< 0.01, < 0.01 0.24, 0.23	< 0.01 0.24	SBR-0026 V-20124-G

Residues of 1-OH-HPA all < 0.1 mg/kg in gin trash (8 trials, including one at 2× rate)

Oilseed rape

In supervised trials on oilseed rape (eight), single foliar broadcast sprays of 0.1–0.11 kg ai flumioxazin/ha (WG formulations) were applied with added adjuvant as pre-harvest desiccants and harvest aids using tractor-mounted or back-pack sprayers with 4–8 nozzle booms.

Duplicate samples of seed (min 0.5 kg) were collected using small plot combines, frozen within 4 hours and analysed for flumioxazin within 14 months of harvest using method RM 30A-3 (GC-MS). Concurrent recoveries from control samples fortified with flumioxazin at levels of 0.02–1.0 mg/kg ranged from 74–120% and the validated LOQ was 0.02 mg/kg.

Table 72 Residues in rape seed from supervised trials in the USA involving one pre-harvest foliar application of flumioxazin (WG formulation)

OILSEED RAPE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Stephens, GA (Sumner)	1	0.104 + MSO	198	0.104	seed	5	0.04, 0.04	0.04	SBR-0123 V-32833-A
		0.105 + NIS	201	0.105	seed	5	0.05, 0.05	0.05	
USA, 2009 Campbell, MN (Hyola 357 RR Mag)	1	0.109 + MSO	188	0.109	seed	1 3 5 8	0.15, 0.17 0.16, 0.16 0.15, 0.17 0.04, 0.04	0.16 0.16 0.16 0.04	SBR-0123 V-32833-B
USA, 2009 Norwich, ND (Invigor 5550)	1	0.11 + MSO	141	0.11	seed	4	0.05, 0.06	0.05	SBR-0123 V-32833-C
	1	0.218 + MSO	142	0.218	seed	4	0.16, 0.16	0.16	
USA, 2009 Carrington, ND (Pioneer 45H26)	1	0.108 + MSO	186	0.108	seed	5 + 16	0.02, 0.03	0.03	SBR-0123 V-32833-D
	1	0.108 + NIS	186	0.108	seed	5 + 16	0.04, 0.04	0.04	

OILSEED RAPE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Scobey, MT (Xceed 8571)	1	0.109+ NIS	186	0.109	seed	4	0.03, 0.12	0.07	SBR-0123 V-32833-E
	1	0.11 + MSO	187	0.11	seed	4	0.3, 0.21	0.25	
USA, 2009 Payette, ID (Hyola 308)	1	0.109 + MSO	188	0.109	seed	5	0.04, 0.04	0.04	SBR-0123 V-32833-F
	1	0.214 + MSO	186	0.214	seed	5	0.09, 0.1	0.1	
USA, 2009 Minidoka, ID (46A76)	1	0.113 + MSO	160	0.113	seed	5 + 6	0.05, 0.09	0.07	SBR-0123 V-32833-G
USA, 2009 Ephrata, WA (71-45 RR)	1	0.109 + MSO	187	0.109	seed	5 + 9	0.05, 0.06	0.06	SBR-0123 V-32833-H
	1	0.541 + MSO	188	0.541	seed	5 + 9	0.6, 0.66	0.63	

DAT = Interval from last application to cutting + field drying interval (in days)

In trials V-32833-D, V-32833-G and V-32833-H, vines were cut and allowed to dry for up to 16 days before seeds were collected.

Peanuts

In fifteen supervised trials on peanuts, single broadcast soil applications of 0.1–0.11 kg ai flumioxazin/ha (WG formulations) were applied either as pre-plant broadcast sprays (with shallow soil incorporation) within 7 days before sowing or as pre-emergent broadcast sprays within 5 days after sowing, using tractor-mounted boom sprayers (8–13 nozzles).

Duplicate samples of whole peanuts were collected after 3–19 days of field drying, shelled in the field and samples of nutmeat (min 2.2 kg) and hulls (min 0.22 kg) were taken for analysis. All samples were kept in frozen storage up to 210 days before analysis for flumioxazin using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02–0.1 mg/kg ranged from 80–105% (nutmeat) and 70–101% (hulls), and the validated LOQs were 0.02 mg/kg.

Table 73 Residues in peanuts (nutmeat and hulls) from supervised trials in the USA involving one broadcast pre-plant or pre-emergent soil application of flumioxazin (WG formulations)

PEANUT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1992 Grangerburg, AL (Florunner)	1	0.109	187	0.109	Nutmeat Hull	140 + 8	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-A PPSI
USA, 1992 Pattison, TX (Spanish)	1	0.105	187	0.105	Nutmeat Hull	110 + 10	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-B PPSI
USA, 1992 Hawkinsville, GA	1	0.108	215	0.108	Nutmeat Hull	134 + 7	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-C

PEANUT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
(Florunner)	1	0.539	215	0.539	Nutmeat Hull	134 + 7	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	PE
USA, 1992 Hobgood, NC (NC-7)	1	0.105	187	0.105	Nutmeat Hull	148 + 10	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-E PE
USA, 1993 Columbia, AL (Florunner)	1	0.108	185	0.108	Nutmeat Hull	135 + 4	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-A PPSI
USA, 1993 Melrose, FL (Florunner)	1	0.109	238	0.109	Nutmeat Hull	148 + 3	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-B PPSI
USA, 1993 Goldsboro NC (NC-7)	1	0.11	193	0.11	Nutmeat Hull	127 + 6	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-C PPSI
USA, 1993 Pattison, TX (Spanish)	1	0.111	271	0.111	Nutmeat Hull	97 + 5	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-D PE
USA, 1993 Hawkinsville, GA (Florunner)	1	0.106	215	0.106	Nutmeat Hull	152 + 8	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-F PE
USA, 1993 Pattison, TX (STARR Spanish)	1	0.549	268	0.549	Nutmeat Hulls	101	< 0.02, < 0.02 0.04, 0.04	< 0.02 0.04	SBR-0019 V-10716-I PE
USA, 1996 Levelland, TX (Valonica McRan)	1	0.108	187	0.108	Nutmeat	154 + 7	< 0.02, < 0.02	< 0.02	SBR-0020 V-11438-B PE
USA, 1996 Unadilla, GA (Georgia Runner)	1	0.107	196	0.107	Nutmeat	139 + 4	< 0.02, < 0.02	< 0.02	SBR-0020 V-11438-C PE
USA, 1996 Columbia, AL (Southern Runner)	1	0.107	242	0.107	Nutmeat	154 + 6	< 0.02, < 0.02	< 0.02	SBR-0020 V-11438-D PE
USA, 1996 Malone, FL (GK-7)	1	0.102	243	0.102	Nutmeat	138 + 19	< 0.02, < 0.02	< 0.02	SBR-0020 V-11438-E PE
USA, 1996 Dill City, OK (Spanco)	1	0.11	131	0.11	Nutmeat	131 + 5	< 0.02, < 0.02	< 0.02	SBR-0020 V-11438-F PE

DAT = Interval from last application to cutting + field drying interval (in days)

PPSI = pre-plant soil incorporation

PE = pre-emergent broadcast soil treatment

Sunflower seed

In supervised trials on sunflowers (eight), single foliar broadcast sprays of 0.11 kg ai flumioxazin/ha (WG formulations) were applied with added adjuvant as pre-harvest desiccants and harvest aids using tractor-mounted or back-pack sprayers with 4–8 nozzle booms.

Duplicate samples of seed (min 1 kg from 12 flower heads) were frozen within 3.5 hours and analysed for flumioxazin within 11 months of harvest using method RM 30A-3 (GC-MS).

Concurrent recoveries from control samples fortified with flumioxazin at levels of 0.02–3.0 mg/kg ranged from 82–102% and the validated LOQ was 0.02 mg/kg.

Table 74 Residues in sunflower seed from supervised trials in the USA involving one pre-harvest foliar application of flumioxazin (WG formulation)

SUNFLOWER SEED COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Northwood, ND (Pioneer 63M80)	1	0.11 + NIS	189	0.11	seed	5	0.03, 0.05	0.04	SBR0126 V-32835-A
	1	0.109 + MSO	187	0.109	seed	5	0.03, 0.03	0.03	
USA, 2009 Campbell, MN (Jaguar)	1	0.109 + MSO	187	0.109	seed	1 3 5 7	0.05, 0.05 0.06, 0.07 0.03, 0.04 0.02, 0.03	0.05 0.06 <u>0.04</u> 0.03	SBR0126 V-32835-B
USA, 2009 Stafford, KS (Pioneer 63M91)	1	0.105 + MSO	200	0.105	seed	5	0.05, 0.05	0.05	SBR0126 V-32835-C
	1	0.216 + MSO	207	0.216	seed	5	0.14, 0.15	0.14	
USA, 2009 Norwich, ND (Mycogen 8N358CL)	1	0.108 + MSO	187	0.108	seed	5	0.09, 0.1	0.1	SBR0126 V-32835-D
	1	0.219 + MSO	191	0.219	seed	5	0.14, 0.2	0.17	
USA, 2009 Velva, ND (Mycogen 8N358CL)	1	0.109 + NIS	188	0.109	seed	5	0.13, 0.15	0.14	SBR0126 V-32835-E
	1	0.11 + MSO	189	0.11	seed	5	0.17, 0.2	0.18	
USA, 2009 Grand Island, NE (3080 DMR NS)	1	0.108 + NIS	187	0.108	seed	4 + 1	0.18, 0.18	0.18	SBR0126 V-32835-F
	1	0.108 + MSO	186	0.108	seed	4 + 1	0.06, 0.07	0.07	
USA, 2009 Malta, MT (Croplan Genetics)	1	0.108 + NIS	187	0.108	seed	5	0.11, 0.12	0.12	SBR0126 V-32835-G
USA, 2009 Hinton, OK (Mycogen 8N435DM)	1	0.111 + MSO	144	0.111	seed	5	0.23, 0.34	0.29	SBR0126 V-32835-H

DAT = Interval from last application to cutting + field drying interval (in days)

Herbs

Mints

In supervised trials on <u>mint</u> (six) conducted in the USA during 2001, two foliar broadcast sprays of 0.28 or 0.42 kg ai flumioxazin/ha (WG formulations) were applied to dormant mint plants (February-April). The intervals between treatments were not reported in the study report. Duplicate samples of mint tops (leaves and stems) were stored frozen for up to 9 months before analysis for flumioxazin using method RM 30A-2 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 72–113% and the validated LOQ was 0.02 mg/kg.

In two of the trials, mint oil was extracted on the same day of harvest and stored frozen for up to 8 months before dilution with acetone and analysis for flumioxazin using method RM 30A-2 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 91–111% and the validated LOQ was 0.02 mg/kg.

Table 75 Residues in mint leaves and oil from supervised trials in the USA involving two foliar applications of flumioxazin (WG formulation)

MINT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
GAP:USA		0.14	140–180	0.28		80	Foliar sprays to dormant plants		
USA, 2001 Roza Unit C-9, WA (Mint)	2	0.28		0.56	leaves oil	112	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0136 WA*01
	2	0.42		0.84	leaves oil	112	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	
USA, 2001 Paterson, WA (Peppermint)	2	0.28		0.56	leaves	80	< 0.02, < 0.02	< 0.02	SBR-0136 WA*02
	2	0.42		0.84	leaves	80	< 0.02, < 0.02	< 0.02	
USA, 2001 Paterson, WA (Spearmint)	2	0.28		0.56	leaves	80	0.02, 0.02	0.02	SBR-0136 WA*03
	2	0.42		0.84	leaves	80	0.03, 0.03	0.03	
USA, 2001 Portage, WI (Peppermint)	2	0.28		0.56	leaves oil	112	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0136 WI-01
	2	0.42		0.84	leaves oil	112	< 0.02, < 0.02	< 0.02	
USA, 2001 Portage, WI (Spearmint)	2	0.28		0.56	leaves	79	< 0.02, < 0.02	< 0.02	SBR-0136 WI-02
	2	0.42		0.84	leaves	79	< 0.02, 0.02	0.02	
USA, 2001 Portage, WI (Spearmint)	2	0.28		0.56	leaves	79	< 0.02, < 0.02	< 0.02	SBR-0136 WI-03 not independent
	2	0.42		0.84	leaves	79	< 0.02, < 0.02	< 0.02	

Legume animal feeds

Alfalfa forage and fodder

In supervised trials on alfalfa (six) conducted in the USA during 2003, two foliar broadcast sprays of 0.14–0.15 kg ai flumioxazin/ha (WG formulations) were applied 24–26 days before the first cutting (with added surfactant) and to the alfalfa regrowth 6–8 days after the first cutting using back pack or tractor-mounted boom sprayers (6–9 nozzles). Retreatment intervals ranged from 30–33 days. In further trials conducted in 2005, single foliar broadcast sprays of 0.14 kg ai flumioxazin/ha (with added surfactants) were applied to alfalfa regrowth 7–9 days after the first cutting using back pack or tractor-mounted boom sprayers (4–8 nozzles).

Duplicate samples of forage (min 1 kg) were taken 6–26 days after the second application and fodder (hay) samples (min 0.5 kg) were taken after a further 2–8 days drying in the field. Samples were frozen within 6 hours and analysed for flumioxazin within 15 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02–2.0 mg/kg (forage) and 0.02–7.0 mg/kg (fodder) ranged from 71–120% and the validated LOQ was 0.02 mg/kg.

Table 76 Residues in alfalfa forage from supervised trials in the USA involving one or two foliar applications of flumioxazin (WG formulation)

ALFALFA	APPLICATION	MATRI	DAT	RESIDUES (MG/KG)	REFEREN

FORAGE COUNTRY, YEAR LOCATION (VARIETY)	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON	X		FLUMIOXAZIN	MEAN	CE & COMMENTS
USA, 2003 Germansville, PA (WL-325)	2	0.14 0.144	234 241	0.284	forage	25 60 113	0.09, 0.12 < 0.02, < 0.02 < 0.02, < 0.02	0.11 < 0.02 < 0.02	SBR-0111 V-25814-A
	2	0.279 0.284	234 237	0.563	forage	25 60 113	0.36, 0.44 0.03, 0.04 < 0.02, < 0.02	0.4 0.04 < 0.02	
USA, 2003 Columbia, MO (Cody)	2	0.141 0.15	239 220	0.291	forage	6 15 24 35 65 107	2.2, 2.3 0.33, 0.37 0.08, 0.16 0.03, 0.06 0.02, 0.03 < 0.02, < 0.02	2.3 0.35 0.12 0.05 0.03 < 0.02	SBR-0111 V-25814-B
USA, 2003 York, NE (Haymark)	2	0.14 0.14	187 187	0.28	forage	25 50 97	0.12, 0.12 0.07, 0.1, 0.18, 0.19 < 0.02, < 0.02	0.12 0.14 < 0.02	SBR-0111 V-25814-C
USA, 2003 Britton, SD (Dekalb DK 122)	2	0.14 0.14	187 187	0.28	forage	25 55 90	0.03, 0.03 0.02, 0.02 < 0.02 (3), 0.06 (2), 0.09	0.03 0.02 0.04	SBR-0111 V-25814-D
USA, 2003 Clarence, MO (UNS Missouri Certified Seed)	2	0.14 0.139	187 186	0.279	forage	25 61 104	0.09, 0.11 < 0.02, < 0.02 < 0.02, < 0.02	0.1 < 0.02 < 0.02	SBR-0111 V-25814-E
USA, 2003 Eden, AZ (Mesa Circi)	2	0.14 0.137	190 186	0.277	forage	25 60 101	0.35, 0.43 0.02, 0.02 < 0.02, < 0.02	0.39 0.02 < 0.02	SBR-0111 V-25814-G
USA, 2005 Franklin, GA (Emerald)	1	0.141	208	0.141	forage	25 70 128	0.79, 0.8 < 0.02, < 0.02 < 0.02, < 0.02	0.8 < 0.02 < 0.02	SBR-0111 V-25814-H
	1	0.283	208	0.283	forage	25 70	1.1, 1.7 < 0.02, < 0.02	1.4 < 0.02	
USA, 2005 New Holland, OH (Rocket)	1	0.144	144	0.144	forage	24 62 87	0.02, 0.03 < 0.02, < 0.02 < 0.02, < 0.02	0.03 < 0.02 < 0.02	SBR-0111 V-25814-I
USA, 2005 Carlyle, IL (Buffalo)	1	0.138	148	0.138	forage	24 49 76	0.07, 0.13 < 0.02, < 0.02 < 0.02, < 0.02	0.1 < 0.02 < 0.02	SBR-0111 V-25814-J
	1	0.278	149	0.278	forage	24 49 76	< 0.02, 0.26 < 0.02, < 0.02 < 0.02, < 0.02	0.14 < 0.02 < 0.02	
USA, 2005 Velva, ND (Vernal)	1	0.139	139	0.139	forage	24 56 99	0.05, 0.06 < 0.02, < 0.02 < 0.02, < 0.02	0.06 < 0.02 < 0.02	SBR-0111 V-25814-K
USA, 2005 Live Oak, CA (Achiever)	1	0.136	137	0.136	forage	25 45 71	0.22, 0.24 0.03, 0.03 < 0.02, < 0.02	0.23 0.03 < 0.02	SBR-0111 V-25814-L
USA, 2005 Payette, ID (Unknown Pioneer variety)	1	0.141	236	0.141	forage	26 57 97	0.14, 0.21 < 0.02, 0.02 < 0.02, < 0.02	0.18 0.02 < 0.02	SBR-0111 V-25814-M

Table 77 Residues in alfalfa fodder (hay) from supervised trials in the USA involving one or two foliar applications of flumioxazin (WG formulation)

ALFALFA FODDER COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2003 Germansville, PA (WL-325)	2	0.14 0.144	234 241	0.284	fodder	25 + 4 60 + 7 113 + 7	0.36, 0.34 < 0.02, 0.02 < 0.02, < 0.02	0.35 0.02 < 0.02	SBR-0111 V-25814-A
		0.279 0.284	234 237	0.563	fodder	25 + 4 60 + 7 113 + 7	2.1, 2.2 0.06, 0.08 < 0.02, < 0.02	2.2 0.07 < 0.02	
USA, 2003 Columbia, MO (Cody)	2	0.141 0.15	239 220	0.291	fodder	6 + 2 15 + 2 24 + 2 35 + 2 65 + 4 107 + 5	5.4, 5.0 1.2, 1.4 0.27, 0.27 0.08, 0.12 0.04, 0.05 < 0.02, < 0.02	5.2 1.3 0.27 0.10 0.05 < 0.02	SBR-0111 V-25814-B
USA, 2003 York, NE (Haymark)	2	0.14 0.14	187 187	0.28	fodder	25 + 4 50 + 5 97 + 4	0.29, 0.17 0.04, 0.03, 0.05, 0.09 < 0.02, < 0.02	0.23 0.05 < 0.02	SBR-0111 V-25814-C
USA, 2003 Britton, SD (Dekalb DK 122)	2	0.14 0.14	187 187	0.28	fodder	25 + 3 55 + 4 90 + 4	0.07, 0.06 0.02, 0.03 0.10, 0.14, 0.13, 0.15	0.07 0.03 0.13	SBR-0111 V-25814-D
USA, 2003 Clarence, MO (UNS Missouri Certified Seed)	2	0.14 0.139	187 186	0.279	fodder	25 + 4 61 + 2 104 + 5	0.23, 0.18 0.02, 0.02 < 0.02, < 0.02	0.21 0.02 < 0.02	SBR-0111 V-25814-E
USA, 2003 Eden, AZ (Mesa Circi)	2	0.14 0.137	190 186	0.277	fodder	25 + 3 60 + 4 101 + 3	1.1, 1.3 0.02, 0.04 < 0.02, < 0.02	1.2 0.03 < 0.02	SBR-0111 V-25814-G
USA, 2005 Franklin, GA (Emerald)	1	0.141	208	0.141	fodder	25 + 7 70 + 4	1.4, 1.6 0.03, < 0.02	1.5 0.02	SBR-0111 V-25814-H
	1	0.283	208	0.283	fodder	25 + 7 70 + 4	5.5, 3.0 0.03, 0.03	4.3 0.03	
USA, 2005 New Holland, OH (Rocket)	1	0.144	144	0.144	fodder	24 + 3 62 + 1 87 + 3	0.11, 0.11 < 0.02, < 0.02 < 0.02, < 0.02	0.11 < 0.02 < 0.02	SBR-0111 V-25814-I
USA, 2005 Carlyle, IL (Buffalo)	1	0.138	148	0.138	fodder	24 + 3 49 + 9 76 + 3	0.23, 0.36 0.03, 0.04 < 0.02, < 0.02	0.3 0.04 < 0.02	SBR-0111 V-25814-J
	1	0.278	149	0.278	fodder	24 + 3 49 + 9 76 + 3	0.94, 0.51 0.07, 0.07 < 0.02, < 0.02	0.73 0.07 < 0.02	
USA, 2005 Velva, ND (Vernal)	1	0.139	139	0.139	fodder	24 + 1 56 + 4 99 + 2	0.22, 0.25 0.02, < 0.02 < 0.02, < 0.02	0.24 0.02 < 0.02	SBR-0111 V-25814-K
USA, 2005 Live Oak, CA (Achiever)	1	0.136	137	0.136	fodder	25 + 4 45 + 4 71 + 2	0.47, 0.45 0.07, 0.09 < 0.02, < 0.02	0.46 0.08 < 0.02	SBR-0111 V-25814-L
USA, 2005 Payette, ID (Unknown Pioneer variety)	1	0.141	236	0.141	fodder	26 + 5 57 + 3 97 + 8	0.88, 0.84 0.04, 0.05 < 0.02, < 0.02	0.86 0.05 < 0.02	SBR-0111 V-25814-M

DAT = Interval from last application to cutting + field drying interval (in days)

Peanut forage and fodder

In fifteen supervised trials on peanuts, single broadcast soil applications of 0.1–0.11 kg ai flumioxazin/ha (WG formulations) were applied either as pre-plant broadcast sprays (with shallow soil incorporation) within 7 days before sowing or as pre-emergent broadcast sprays within 5 days after sowing, using tractor-mounted boom sprayers (8–13 nozzles).

Duplicate samples of peanut vines were collected immediately after digging, and samples of hay (min 0.45 kg) were collected after 3–19 days of field drying and kept in frozen storage up to 210 days before analysis for flumioxazin using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02–0.1 mg/kg ranged from 76–113% (vines) and 63–86% (hay) and the validated LOQs were 0.02 mg/kg.

Table 78 Residues in peanut vines and hay from supervised trials in the USA involving one broadcast pre-plant or pre-emergent soil application of flumioxazin (WG formulations)

PEANUT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1992 Alabama (Florunner)	1	0.109	187	0.109	Vines Hay	132	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-A
USA, 1992 Georgia (Florunner)	1	0.108	215	0.108	Vines Hay	134	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-C
USA, 1992 North Carolina (NC-7)	1	0.105	187	0.105	Vines Hay	148	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-E
USA, 1992 Texas (Spanish)	1	0.105	187	0.105	Vines Hay	110	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0018 V-1040-B
USA, 1993 Alabama (Florunner)	1	0.108	185	0.108	Vines Hay	135	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-A
USA, 1993 Florida (Florunner)	1	0.109	238	0.109	Vines Hay	148	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-B
USA, 1993 Georgia (Florunner)	1	0.106	215	0.106	Vines Hay	152 14 21 28 152	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	SBR-0019 V-10716-F
USA, 1993 North Carolina (NC-7)	1	0.11	193	0.11	Vines Hay	127 21 28	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02	SBR-0019 V-10716-C
USA, 1993 Texas (Spanish)	1	0.111	271	0.111	Vines Hay	97	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0019 V-10716-D

Soya bean forage and fodder

In supervised trials on soya beans conducted between 1989 and 1993, single broadcast soil application of 0.1–0.11 kg ai flumioxazin/ha (WG, FL or WP formulations) were applied using back-pack or

tractor-mounted boom sprayers, either as pre-plant treatments (with or without soil incorporation) or just after sowing, before crop emergence.

Duplicate samples of forage (min 0.9 kg) and hay (min 0.45 kg) were frozen within 24 hours and stored for up to 13 months (forage) and 11 months (hay) before analysis for flumioxazin using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 mg/kg ranged from 67–120% in forage and 73–130% in hay, with a validated LOQ of 0.02 mg/kg.

Table 79 Residues in soya bean forage and fodder from supervised trials in the USA involving one broadcast soil application of flumioxazin (WG formulations)

SOYA BEAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
GAP:USA		0.105	140–280	0.105			pre-plant or pre-emergent		
USA, 1989 Dallas Center, IA (Asgrow 1937)	1	0.101	94	0.101	forage hay	40 111	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7262
USA, 1989 Dallas Center, IA (Wells II)	1	0.101	187	0.101	forage hay	40 103	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7370 no cultivation
USA, 1989 Geneseo, IL (Pioneer 9271)	1	0.101	187	0.101	forage hay	40 103	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7374
USA, 1989 Greenville, MS (Forrest)	1	0.101	187	0.101	forage hay seed	40 113	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7373
USA, 1989 Hollandale, MN (NK523-12)	1	0.101	187	0.101	forage hay	67 95	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7260
USA, 1989 Lanoke, AR (Asgrow 5980)	1	0.101	94	0.101	forage hay	40 102	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7263
USA, 1989 Leonard, MO (Williams 82)	1	0.101	374	0.101	forage hay	40 100	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7368
USA, 1989 Metcalfe, MS (Forrest)	1	0.101	187	0.101	forage hay	40 100	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7375
USA, 1989 New Holland, OH (Pioneer 9361)	1	0.101	365	0.101	forage hay	40 100	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7369
USA, 1989 Noblesville IN (Pioneer 9361)	1	0.101	206	0.101	forage hay	40 110	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7261
USA, 1989 Rosa, LA (Forrest)	1	0.101	212	0.101	forage hay	64 149	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7372
USA, 1989 York, NE (Hack)	1	0.101	187	0.101	forage hay	40 90	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7371
USA, 1990 Clarence, MO (Williams 82)	1	0.101	187	0.101	forage hay	40 91	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7512
USA, 1990 Cloverport, TN (FFR 562)	1	0.101	187	0.101	forage hay	40 79	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7501

SOYA BEAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1990 Dallas Center, IA (Asgrow 2187)	1	0.101	187	0.101	whole plant whole plant whole plant whole plant whole plant whole plant hay	8 15 29 40 60 90 90	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	SBR-0003 T-7507 pre-emergence
USA, 1990 Dallas Center, IA (Asgrow 2187)	1	0.101	187	0.101	forage hay	40 99	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7509 pre-plant
USA, 1990 Elwood, IL (Pioneer 9202)	1	0.101	196	0.101	forage hay	40 107	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7508
USA, 1990 Geneseo, IL (Pioneer 9272)	1	0.101	187	0.101	forage hay	40 40	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7502
USA, 1990 Greenville, MS (Forrest)	1	0.101	187	0.101	whole plant whole plant whole plant whole plant whole plant whole plant hay	7 15 30 39 60 90 90	 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	0.07 0.06 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	SBR-0003 T-7506
USA, 1990 Hollandale, MN (Agri Pro 1776)	1	0.101	187	0.101	forage hay	40 102	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7511
USA, 1990 Hollendale, MN (Agri Pro1776)	1	0.101	187	0.101	forage hay	40 102	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7500 no cultivation
USA, 1990 New Holland, OH (Pioneer 9391)	1	0.101	243	0.101	forage hay	41 93	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7510
USA, 1990 Noblesville, IN (Pioneer 9361)	1	0.101	253	0.101	forage hay	40 72	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7503
USA, 1990 Proctor, AR (DPL 105)	1	0.101	187	0.101	forage hay	40 110	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0003 T-7513
USA, 1992 Goldsboro, NC (Ransom)	1	0.105	187	0.105	forage forage forage hay seed	22 29 41 123	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-1039-A
USA, 1992 Greenville, MS (Pioneer 9641)	1	0.105	187	0.105	forage forage forage forage hay	13 20 28 39 99	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-1039-H
USA, 1992 Leonard, MO (Pioneer 9443)	1	0.102	187	0.102	forage forage forage hay	21 28 40 91	 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	0.03 < 0.02 < 0.02 < 0.02	SBR-0021 V-1039-M
USA, 1992 Little Rock, AR (Hutcheson)	1	0.105	187	0.105	forage forage forage hay	21 28 40 110	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-1039-C

SOYA BEAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1992 New Holland, OH (GL 2910)	1	0.105	150	0.105	forage forage forage hay	22 29 42 106	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-1039-G
USA, 1992 Noblesville, IN (Pioneer 9361)	1	0.105	234	0.105	forage forage hay	28 40 97	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02	SBR-0021 V-1039-D
USA, 1992 Seymour, IL (Asgrow 2543)	1	0.105	187	0.105	forage forage forage hay	21 28 40 102	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-1039-B pre-plant
USA, 1992 Seymour, IL (Asgrow 2543)	1	0.105	187	0.105	forage forage forage hay	21 28 40 98	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-1039-J pre-emergence
USA, 1992 Waukee, IA (Asgrow 2543)	1	0.105	187	0.105	forage forage forage forage hay	14 21 28 39 98	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-1039-F
USA, 1993 Greenville, MS (Asgrow 5979)	1	0.109	187	0.109	forage forage forage forage hay	14 21 28 41 98	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-10719-H
USA, 1993 Jamesville, NC (Hutcheson)	1	0.108	253	0.108	forage forage forage hay	21 28 40 122	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-10719-A
USA, 1993 Leonard, MO (Linford)	1	0.107	271	0.107	forage forage forage hay	21 28 40 88	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-10719-E
USA, 1993 New Holland, OH (Madison GL 2910)	1	0.107	196	0.107	forage forage forage hay	22 31 40 88	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-10719-G
USA, 1993 Noblesville, IN (Pioneer 9361)	1	0.11	206	0.11	forage forage forage hay	21 27 40 108	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-10719-D
USA, 1993 Seymour, IL (Asgrow 2506)	1	0.107	187	0.107	forage forage forage forage hay	14 22 28 40 83	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 0.02 < 0.02 < 0.02	SBR-0021 V-10719-J
USA, 1993 Theilman, MN (Pioneer 9061)	1	0.107	187	0.107	forage forage hay	28 40 101	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02	SBR-0021 V-10719-B
USA, 1993 Webster City, IA (L-1700)	1	0.108	206	0.108	forage forage hay	28 41 80	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02	SBR-0021 V-10719-F
USA, 1993 York, NE (Hack)	1	0.107	187	0.107	forage forage forage hay	21 28 40 85	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-10719-C

The 1992–1993 supervised trials also analysed for residues of the metabolite, 1-OH HPA in seeds and the results also showed levels below the LOQ of 0.02 mg/kg.

Straw, forage, fodder of cereal grains

Maize forage and fodder

In twenty-one supervised trials on maize, single broadcast soil applications of 0.1–0.11 or 0.2–0.22 kg ai flumioxazin/ha (WG formulations) with added surfactant were applied up to 7 days before sowing, using back-pack plot sprayers, wheeled or tractor-mounted boom sprayers (3–9 nozzles).

Duplicate samples of forage (min 12 units) were taken at the late dough/early dent growth stage (about BBCH 86) and stover samples (min 12 units) were taken at grain harvest. Samples were all frozen within 2 hours and analysed for flumioxazin within 14 months using method RM 30A-3 (GC-MS) in the 2005 trials and method NCL 293 (HPLC-MS/MS) in the 2006 trials. Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 87–117% (forage) and 79–118% (stover) in the two methods and the validated LOQs were 0.02 mg/kg.

Table 80 Residues in maize forage and fodder from supervised trials in the USA involving one broadcast pre-plant soil application of flumioxazin (WG formulations)

MAIZE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2005 New Holland, OH (Syngenta N73-F7)	1	0.107	191	0.107	forage stover	103 148	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-A
	1	0.211	188	0.211	forage stover	103 148	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	
USA, 2005 Carlyle, IL (FS 6455)	1	0.107	190	0.107	forage stover	102 171	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-B
	1	0.212	187	0.212	forage stover	102 171	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	
USA, 2005 Clarence, MO (Pioneer 35P12)	1	0.107	191	0.107	forage stover	119 154	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-C
USA, 2005 Greenville, MS (69-71 757 HXJINX)	1	0.104	185	0.104	forage stover	118 135	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-D
USA, 2006 North Rose, NY (Dairyland Stealth 8711)	1	0.108	191	0.108	forage stover	93 131	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-E
USA, 2006 Elko, SC (Pioneer 31R87)	1	0.105	180	0.105	forage stover	106 158	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-F
Canada, 2006 City of Hamilton, Ontario (Pioneer 38B84)	1	0.107	187	0.107	forage stover	107 166	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-G
	1	0.211	184	0.211	forage stover	107 166	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	
USA, 2006 Conklin, MI (N45-M2 Field Corn)	1	0.106	189	0.106	forage stover	97 138	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-H

MAIZE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2006 Carlyle, IL (DKC-65-16)	1	0.108	185	0.108	forage stover	112 168	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-I
USA, 2006 Bellmore, IN (Wyffels 5531)	1	0.104	185	0.104	forage stover	106 136	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-J
USA, 2006 York, NE (NK N70-F1)	1	0.106	184	0.106	forage stover	117 155	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-K
USA, 2006 Richland, IA (Pioneer 33P65)	1	0.105	189	0.105	forage stover	109 151	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-L
USA, 2006 Geneva, MN (Pioneer 38H66)	1	0.106	180	0.106	forage stover	110 163	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-M
USA, 2006 Fairmount, ND (Dekalb 35-02)	1	0.106	188	0.106	forage stover	100 145	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-N
USA, 2006 Campbell, MN (Pioneer 39H83)	1	0.106	188	0.106	forage stover	100 155	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-O
USA, 2006 Hudson, KS (Midwest Seed Genetics 8127RB)	1	0.106	188	0.106	forage stover	104 134	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-P
Canada, 2006 Portage la Prairie, Manitoba (Roundup Ready-Monsanto)	1	0.102	181	0.102	forage stover	114 154	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-Q
USA, 2006 Arkansaw, WI (Pioneer 38B85)	1	0.106	188	0.106	forage stover	110 137	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-R
Canada, 2006 St. Pie, Quebec (NK 3030 BT)	1	0.101	176	0.101	forage stover	122 156	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-S
USA, 2006 Dill City, OK (DKC48-53)	1	0.107	193	0.107	forage stover	98 130	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-T
USA, 2006 Clarence, MO (Pioneer 34B20)	1	0.107	187	0.107	forage stover	105 165	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-U
USA, 2006 Clarence, MO (Pioneer 34B20)	1	0.536	187	0.536	forage stover	105 165	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0078 V-28566-U (Processing)

Wheat forage, hay and straw

In three supervised trials on wheat, single pre-plant broadcast soil applications of 0.07 or 0.14 kg ai flumioxazin/ha (WG formulations) with added surfactant were applied 7 or 14 days before sowing respectively, using tractor-mounted boom sprayers (4–8 nozzles).

Duplicate samples of wheat forage (from plants about 13 cm tall) and hay (sampled at BBCH 61–85 and allowed to dry to 10–20% moisture content) were frozen within 2 hours and analysed for flumioxazin within 38 days of harvest using method RM 30A-3 (GC-MS). Concurrent recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 93–109% in forage, 89–120% in hay and the validated LOQ was 0.02 mg/kg.

Table 81 Residues in wheat forage and hay from supervised trials in the USA involving one broadcast pre-plant soil application of flumioxazin (WG formulations)

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2010 Leland, MS (Gore)	1	0.07	182	0.07	forage hay	129 172 + 4 d dry	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0127 V-37119-A
	1	0.14	184	0.14	forage hay	129 172 + 4 d dry	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	
USA, 2010 Levelland, TX (TAM 112)	1	0.71	186	0.71	forage hay	85 247 + 4 d dry	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0127 V-37119-B
		0.144	188	0.144	forage hay	79 241 + 4 d dry	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	
USA, 2010 Larned, KS (Santa Fe Winter Wheat)	1	0.72	188	0.72	forage hay	71 262 + 5 d dry	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0127 V-37119-C
	1	0.143	186	0.143	forage hay	64 255 + 5 d dry	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	

In twenty supervised trials on wheat, single foliar broadcast sprays of 0.07–0.075 kg ai flumioxazin/ha (WG formulations) with added adjuvants were applied as pre-harvest desiccants (harvest aids) using tractor-mounted or back-pack sprayers with 4–8 nozzle booms.

Duplicate samples of straw were collected using small plot combines or cut and harvested using a stationary combine, frozen within 5 hours and analysed for flumioxazin within 17 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control straw samples fortified with flumioxazin at levels of 0.02–5.0 mg/kg ranged from 70–115% and the validated LOQ was 0.02 mg/kg.

Table 82 Residues in wheat straw from supervised trials in the USA involving one pre-harvest foliar application of flumioxazin (WG formulation)

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Lexington, GA (USG 3592)	1	0.071 + NIS	193	0.071	straw	10	1.88, 1.74	1.82	SBR-0092 V-33037-A
	1	0.071 + MSO	192	0.071	straw	10	3.46, 3.95	3.71	
USA, 2009 Leland, MS (Gore)	1	0.071 + MSO	185	0.071	straw	3 7 10 13	3.53, 2.85 1.18, 1.42 2.4, 2.69 1.14, 0.92	3.19 1.30 2.55 1.03	SBR-0092 V-33037-B
USA, 2009 Carlyle, IL (Branson)	1	0.072 + MSO	199	0.072	straw	10	0.86, 0.66	0.76	SBR-0092 V-33037-C
		0.145 + MSO	200	0.145	straw	10	2.11, 2.62	2.37	
USA, 2009 York, NE (Traverse Hard red Spring)	1	0.071 + NIS	184	0.071	straw	10	0.88, 0.99	0.94	SBR-0092 V-33037-D
	1	0.071 + MSO	186	0.071	straw	10	1.91, 1.75	1.83	
USA, 2009 Rockville, IN (Becks 164)	1	0.072 + NIS	148	0.072	straw	11	1.79, 1.13	1.46	SBR-0092 V-33037-E
	1	0.072 + MSO	148	0.072	straw	11	1.42, 1.1	1.26	
USA, 2009 Clarence, MO (Ernie)	1	0.074 + NIS 0.07 + MSO	193 183	0.074 0.07	straw	10 10	2.01, 1.67 2.5, 2.19	1.84 2.35	SBR-0092 V-33037-F
USA, 2009 Bagley, IA (Briggs hrS)	1	0.71 + NIS	148	0.071	straw	10	0.49, 1.2	0.85	SBR-0092 V-33037-G
	1	0.072 + MSO	150	0.072	straw	10	1.34, 1.83	1.59	
USA, 2009 Ulvade, TX (Fannin)	1	0.07 + NIS	138	0.07	straw	9	2.9, 3.53	3.22	SBR-0092 V-33037-H
	1	0.072 + MSO	142	0.072	straw	9	3.32, 3.48	3.40	
USA, 2009 Grand Island, NE (Traverse Hard Red Spring)	1	0.072 + NIS	189	0.072	straw	10	1.64, 1.49	1.57	SBR-0092 V-33037-I
	1	0.071 + MSO	186	0.071	straw	10	2.0, 1.48	1.74	
USA, 2009 Velva, ND (Faller)	1	0.072 + MSO	141	0.072	straw	10	3.1, 3.3	3.2	SBR-0092 V-33037-J
USA, 2009 Grand Island, NE (Kelby Hard Red Spring)	1	0.071 + NIS	185	0.071	straw	10	0.99, 1.13	1.06	SBR-0092 V-33037-K not independent
USA, 2009 Norwich, ND (Faller)	1	0.072 + MSO	172	0.072	straw	10	1.73, 1.36	1.55	SBR-0092 V-33037-L
	1	0.146 + MSO	143	0.146	straw	10	4.21, 2.48	3.35	
USA, 2009 Malta, MT (McNeal)	1	0.069 + NIS	181	0.069	straw	10	2.48, 3.63	3.19	SBR-0092 V-33037-M
USA, 2009 Levelland, TX (TAM 105)	1	0.072 + NIS	189	0.072	straw	9	1.39, 2.03	1.71	SBR-0092 V-33037-N
USA, 2009	1	0.072 + MSO	165	0.072	straw	9	1.64, 2.02	1.83	SBR-0092

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
Wellington, TX (TAM 111)	1	0.142 + MSO	163	0.142	straw	9	3.33, 3.93	3.63	V-33037-O
USA, 2009 Larned, KS (Jagger)	1	0.074 + NIS	212	0.074	straw	10	0.21, 0.25	0.23	SBR-0092 V-33037-P
USA, 2009 Hinton, OK (Jagger)	1	0.07 + MSO	164	0.07	straw	4 7 10 13	2.09, 2.53 2.85, 2.35 1.93, 2.18 1.9, 1.91	2.31 2.60 2.06 1.91	SBR-0092 V-33037-Q
USA, 2009 Cordell, OK (Fuller)	1	0.072 + MSO	172	0.072	straw	11	1.2, 1.94	1.57	SBR-0092 V-33037-R
USA, 2009 Jerome, ID (AC Andrew)	1	0.071 + NIS	181	0.071	straw	10	1.33, 1.4	1.37	SBR-0092 V-33037-S
USA, 2009 Hinton, OK (Deliver)	1	0.071 + MSO	165	0.071	straw	10	1.49, 1.93	1.71	SBR-0092 V-33037-T not independent
	1	0.344 + MSO	152	0.344	straw	10	7.66, 9.29	8.48	

NIS = Non-ionic surfactant

MSO = Methylated seed oil surfactant

Fate of residues in storage and processing

The meeting received processing studies on apples, plums, grapes, olives, soya beans, potatoes, sugar cane, maize, wheat, sugar cane, oilseed rape, sunflower seed, peanuts and mint. In all cases, fresh commodity samples collected from supervised trials at exaggerated rates were processed simulating commercial practices.

Apple

In a supervised trial on apples conducted in the USA and reported by Stearns, 2004 [Ref: SBR-0031], two inter-row/berm soil treatments of 0.86 kg ai flumioxazin/ha (SC formulation) were applied using an ATV-mounted boom sprayer (six nozzles). Treatments were applied 60 days apart, with the last application 60 days before harvest.

Duplicate samples of 30 kg mature fruit were frozen within 1 hours and stored for 5 days before processing into apple wet pomace and juice, simulating commercial practices. Unwashed apples were ground using a hammer mill, then pressed in a hydraulic press to provide apple juice and wet pomace. The processed samples were stored frozen for up to 9 months before analysis for flumioxazin using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02–0.1 mg/kg ranged from 79–110% (apples) and 90–98% (wet pomace). Recoveries in juice spiked with 0.005–0.5 mg/kg were 95–103%. In juice the validated LOQ was 0.02 mg/kg.

Table 83 Residues in apples, pomace and juice from a supervised trial in the USA involving two directed inter-row soil applications of flumioxazin (SC formulation)

APPLE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	

APPLE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2002 Ephrata, WA (Rome)	2	0.86 0.86	200 201	1.723	whole fruit wet pomace juice	60	< 0.02, < 0.02 < 0.02, < 0.02 < 0.005, < 0.005	< 0.02 < 0.02 < 0.005	SBR-0031 V-24504-02-G

Plum

In a supervised trial on plums conducted in the USA and reported by Kowalsky, 2004 [Ref: SBR-0030], two inter-row/berm soil treatments of 0.86 kg ai flumioxazin/ha (SC formulation) were applied using a tractor-mounted boom sprayer (six nozzles). Treatments were applied 64 days apart, with the last application 60 days before harvest.

Duplicate samples of mature plums (33 kg) were processing into prunes on the day of harvest by removing stems and leaves, washing the fruits with a hose, and air dying in drying tunnels for about 19 at 86 °C and allowed to cool for 24 hours. Duplicate samples were analysed for flumioxazin within 9 months of processing using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 83–98% (plums) and 105–109% (prunes) and the validated LOQ was 0.02 mg/kg.

Table 84 Residues in plums and prunes from a supervised trial in the USA involving two directed inter-row soil applications of flumioxazin (SC formulation)

PLUM COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2002 Hughson, CA (French)	2	0.86 0.864	375 375	1.72	whole fruit prunes	60	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0030 V-24539-G

Grape

In a supervised trial on grapes conducted in the USA and reported by Schreier, 2000 [Ref: SBR-0025], two directed inter-row/berm soil treatments of 2.1 kg ai flumioxazin/ha (WG formulation) with added crop oil were applied using a back-pack sprayer with hand-held 4-nozzle boom. Treatments were applied 60 days apart with the last application 60 days before harvest. Duplicate samples of grapes (9 kg for juice processing and 56 kg for raisin processing) were processed into juice and raisins within 24 hours of sampling.

Juice was prepared by washing the grape bunches with water then hand feeding them into a crusher/stemmer machine. The grape pulp was separated from the stems and seeds and transferred to a hydraulic fruit press. The fresh juice collected from the press was filtered to remove coarse solids prior to freezing and storage for up to 1.6 months before analysis for flumioxazin using method RM 30A-1.

Grapes were processed into raisins by sun-drying on trays in the field for about a month before being screened to remove loose dirt, stems and debris and hand sorted to remove the cap stems and any additional unacceptable product. The raisins were batch washed for 10–15 seconds, re-hydrated to approximately 18% moisture, frozen and stored for up to 5 months before analysis for flumioxazin using method RM 30A-1.

Recoveries from control samples fortified with flumioxazin at levels of 0.01 and 0.05 mg/kg ranged from 82–123% (grapes), 95–115% (juice) and 96–106% (raisins). The validated LOQs were 0.01 mg/kg.

Table 85 Residues in fresh and processed grapes from a supervised trial in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

GRAPES COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1999 Kerman, CA (Thompson seedless)	2	2.13 2.12	184 187	4.25	grapes washed grapes raisins juice	60	< 0.01, < 0.01 < 0.01, < 0.01 < 00.1, < 0.01 < 0.01, < 0.01	< 0.01 < 001 < 0.01 < 0.01	SBR-0025 V-20108-L

Olive

In a supervised trial on olives conducted in the USA and reported by Arsenovic and Leonard, 2011 [Ref: SBR-0130], two directed inter-row/berm soil treatments of 2.1 kg ai flumioxazin/ha (WG formulation) with added crop oil were applied using a back-pack sprayer with a hand-held 3-nozzle miniboom. Treatments were applied 62 days apart with the last application 56 days before harvest.

Duplicate samples (22 kg) of olives were refrigerated overnight and sent to the processing facility where the samples were cleaned of extraneous materials and then warmed in an oven for about 20 minutes at 24–29 °C. Warmed olives were ground in a food chopper to produce a paste, which was then placed in a mixer and transferred into a filter press. Pressure was applied to remove oil from the paste. The oil was filtered, collected and stored frozen for up to 17 months before analysis for flumioxazin using method RM 30A-03 (GC-MDS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 76–122% (olives) and 82–116% (oil) and the validated LOQ was 0.02 mg/kg.

Table 86 Residues in olives and oil from a supervised trial in the USA involving two inter-row soil applications of flumioxazin (WG formulations)

OLIVE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2008 Glenn, CA (Arbegnina 1-18 clone)	2	2.05 2.07	211 212	4.13	fruit without pits oil	56	< 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02	SBR-0130 CA91

Soya bean

In a supervised trial on soya beans conducted in the USA and reported by Pensyl, 1996 [Ref: SBR-0021], one broadcast soil applications of 0.536 kg ai flumioxazin/ha (WG formulation) was applied using a tractor-mounted boom sprayer (six nozzles), immediately after sowing.

Duplicate samples of seed (min 22 kg) were frozen within 24 hours and shipped overnight to the processing facility where the samples were dried, aspirated and screening before being mechanically cracked. Aspiration was used to separate the hull and kernel fractions and the kernels were heat-conditioned, flaked, expanded into collets, and solvent extracted to obtain the crude oil. The crude oil was degummed, refined, bleached, and deodorized.

Samples were stored for up to 13 months before analysis for flumioxazin using method RM 30A-3 (GC-MS) and also for the 1-OH-HPA metabolite, using method RM 30M (GC-MS). Recovery rates in samples spiked with 0.02 mg/kg flumioxazin ranged from 75–113% in seed and the processed commodities and were 71–100% in samples spiked with the 1-OH-HPA metabolite.

Table 87 Residues in soya bean seeds and processed commodities from a supervised trial in the USA involving one broadcast pre-emergence soil application of flumioxazin (WG formulation)

SOYA BEAN COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1993 Seymour, IL (Asgrow 2506)	1	0.536	187	0.536	seed hulls extracted meal crude oil crude lecithin refined oil soapstock	112	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	SBR-0021 V-10719-K

The 1992–1993 studies also analysed for residues of the metabolite, 1-OH HPA. Residues in all samples were below the LOQ of 0.02 mg/kg.

Potato

In a supervised trial on potatoes conducted in the USA and reported by Arsenovic, 2003 [Ref: SBR-0091], one broadcast soil applications of 0.14 kg ai flumioxazin/ha (WG formulation) was applied using an ATV-mounted boom sprayer (five nozzles) after the last hilling operation, before potato emergence.

Duplicate samples of 22 kg potatoes were cool-stored for 2 days before processing into wet peel, chips, and flakes. Potato tubers were cleaned, washed, peeled with an abrasive peeler and sliced into chips using a food cutter. The slices of potato were rinsed in warm water to remove free starch, and then fried. The oil was drained and the chips salted, packed and stored. Potato flakes were prepared from cleaned potato tubers, which were washed, peeled using a steam peeler, inspected and trimmed. The potato peel was collected and pressed hydraulically in a fruit press. The pressed peel was then blended with trim waste and placed in a freezer. The peeled potatoes were cut into slabs and spray-washed with cold water to remove free starch. The slabs were then pre-cooked at 70–77 °C in a steam-jacketed kettle. The pre-cooked slabs were cooled to less than 32 °C. A small amount of the cooled slabs was removed and steam-cooked at atmospheric pressure at 94–100 °C for 40 minutes. The cooked potato slabs were then mashed and fed into a dryer to produce a thin sheet, which was initially broken into large flakes by hand. The flakes were then fed into a hammermill for uniform milling of the flakes.

Samples were stored frozen for up to 8 months before analysis for flumioxazin using method RM 30A-2 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 77–118% (tubers) and 98–111% in the processed commodities. The validated LOQ was 0.02 mg/kg.

Table 88 Residues in potatoes and processed commodities from a supervised trial in the USA involving one broadcast pre-emergent soil application of flumioxazin (WG formulation)

POTATO COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	

POTATO COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2001 Prosser, WA (Russet Burbank)	1	0.141	149	0.141	Tuber wet peel chips flakes	107	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	SBR-0091 WA*07

Maize

In a supervised trial on maize conducted in the USA and reported by Kowalsky, 2007 [Ref: SBR-0078] one broadcast soil application of 0.536 kg ai flumioxazin/ha (WG formulation) with added surfactant was applied 7 days before sowing, using a tractor-mounted boom sprayer (six nozzles).

Duplicate samples of kernels (min 230 kg) were taken at maturity, frozen within 2 hours and stored for up to 4 months before processing (11 months for processing into refined oil) by dry milling (to obtain grits, meal, flour and refined oil) and by wet milling (to obtain starch and refined oil).

For the dry mill processing, samples were conditioned to 21% moisture content and tempered for 2.5 hours. The kernels were cracked in a mill and corn stock from the mill was dried in an oven at 54–71 °C. Dried corn stock was screened to separate germ, bran, grits, meal and flour. The germ material was heated to 71–79 °C, flaked and triple-extracted with hexane (at 50–60 °C). The spent flakes were exposed to ambient air to remove residual hexane. The resulting fractions were miscella (crude oil and hexane) and solvent extracted germ meal. The miscella was passed through a vacuum evaporator and heated to 73–90 °C to remove hexane from the crude oil which was then mixed with sodium hydroxide in a water bath and centrifuged, decanted, filtered to produce refined oil.

For the wet mill processing, samples of kernels were steeped in 49–54 °C water containing 0.1–0.2% sulphur dioxide for 22–48 hours and passed through a disc mill and centrifuged to remove most of the germ and hulls. After drying to 5–10% moisture content, the remaining germ and hull were separated by aspiration and screening. Corn stock (without germ and hull) was ground in a disc mill, passed over a 325 mesh screen. Material on top of the screen was discarded. Process water passing through the screen was separated into starch and gluten by centrifugation. Germ samples were conditioned to 12% moisture content, heated to 88–104 °C, flaked and pressed in an expeller to liberate part of the crude oil. The presscake was double-extracted with hexane (at 50–60 °C). The spent presscake were exposed to ambient air to remove residual hexane. The resulting fractions were miscella and solvent extracted presscake (germ cake). The miscella was passed through a vacuum evaporator and heated to 73–90 °C to remove hexane from the crude oil. The expelled and extracted crude oil samples were filtered, combined and mixed with sodium hydroxide in a water bath and centrifuged, decanted, filtered to produce refined oil.

The processed samples were analysed for flumioxazin within one month using the GC-MS methods RM 30A-3 and RM-30B for the dry milled samples. Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 85–100% (kernels) and 71–117% in the processed commodities. The validated LOQs were 0.02 mg/kg.

Table 89 Residues in maize and processed commodities from a supervised trial in the USA involving one broadcast pre-plant soil application of flumioxazin (WG formulation)

MAIZE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2006 Clarence, MO (Pioneer 34B20)	1	0.536	187	0.536	Grain Starch Refined oil Grits Flour Meal Refined oil	166	< 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02 < 0.02, < 0.02	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	SBR-0078 V-28566-U

Wheat

In one supervised trial on wheat conducted in the USA and reported by Kowalsky, 2011 [Ref: SBR-0092], a single foliar broadcast spray of 0.344 kg ai flumioxazin/ha (WG formulation) with added adjuvant was applied as a pre-harvest desiccant/harvest aid using an ATV-mounted boom sprayer (eight nozzles).

Duplicate samples of wheat grain (400 kg) were collected using small plot combines, frozen within 2 days after harvest and stored for up to 3.5 months before processing into bran, flour, middlings, shorts, germ, and aspirated grain fractions.

Grain samples were aspirated to remove grain dust with the materials passing through a 2360 µm sieve being collected as the aspirated grain fraction. The cleaned grain samples were adjusted to 16% moisture content, milled and passed through a 34 mesh sieve to separate the bran from the germ fraction. This bran sample was further sieved through a number of 128 µm screens, with the material passing through the screen being collected as “shorts” and the retained material was collected as “bran”. The germ fraction (with endosperm) was passed through a reduction mill and again sifted to separate the germ from the endosperm. Cleaned grain samples (conditioned by 16.5% moisture content) were also milled to crack the grains and passed through sifter screens, with material passing through a 140 µm screen being collected as “break flour”. Material passing through an 800 µm screen was collected as “middlings” and the retained material was collected as “bran”. The “middlings” sample was subjected to further milling and sieving with material passing through a 160 µm screen being collected as “reduction flour” and the retained material collected as “shorts”. The “break flour” and “reduction flour” were mixed together to produce the flour samples.

Samples were analysed for flumioxazin within 14.5 months of harvest using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02–0.5 mg/kg ranged from 96–114% (grain) and 79–120% in the processed fractions. The validated LOQs were 0.02 mg/kg.

Table 90 Residues in wheat grain and processed commodities from a supervised trial in the USA involving one pre-harvest foliar application of flumioxazin (WG formulation)

WHEAT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Hinton, OK (Deliver)	1	0.344+MSO	152	0.344	grain bran flour middling shorts germ asp grain fraction	10	0.37, 0.35 0.35, 0.33 0.05, 0.05 0.08, 0.08 0.11, 0.1 0.38, 0.36 117, 105	0.36 0.34 0.05 0.08 0.11 0.37 111	SBR-0092 V-33037-T

Middlings = The larger particles coming from the floury part (endosperm) of the grain during milling, possibly including small bits of bran

Shorts = A low-grade mill product containing principally germ and fine bran particles, used for animal feed

Sugar cane

In one supervised trial on sugar canes conducted in the USA and reported by Schreier, 1999 [Ref: SBR-0022], a single broadcast application of 1.25 kg ai flumioxazin/ha (WG formulation) with added crop oil was applied over the top of 2–2.5 m high canes using back-pack sprayers with an extended single-nozzle hand lance.

Duplicate samples of canes were frozen within 2 days of harvest and stored for up to 2 months before being processed into refined sugar and blackstrap molasses in a way that simulated commercial practices as closely as possible. Refined sugar was obtained by chopping the cane stalks, pressing out the juice, clarifying, and concentrating the juice to syrup. Syrup, water and seed sugar were vacuum-concentrated to massecuite, which was then centrifuged to produce raw sugar and 'final' or 'blackstrap' molasses. The raw sugar was dissolved in distilled water, adjusted to a pH to 7.2 with calcium hydroxide and heated. The resulting solution was filtered, decolorized with bone char, and filtered again before boiling under vacuum to crystallize out the sugar which was centrifuged and washed with a water spray to produce refined sugar.

Samples were stored frozen for up to 2 months before analysis for flumioxazin using method RM 30C (GC-MS) and for up to 2.7 months before analysis for the 1-OH-HPA metabolite using method RM-30M (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 67–113% in the canes and 97–110% in the processed commodities. In samples fortified with 0.02–0.2 mg/kg 1-OH-HPA, recoveries were 70–114% in canes and 78–114% in the processed commodities. The validated LOQs for both compounds were both 0.02 mg/kg.

Table 91 Residues in sugar cane and processed commodities from a supervised trial in the USA involving one broadcast foliar application of flumioxazin (WG formulation)

SUGAR CANE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	no	kg ai/ha	water (L/ha)	kg ai/ha/ season			flumioxazin	mean	
USA, 1998 Spreckelsville, HI (78-4153)	1	1.263	187	1.263	cane (field) cane (bulk) molasses sugar	90	0.08, 0.09 0.11 0.055 < 0.02	0.08 0.11 0.055 < 0.02	SBR-0022 V-11945-I

Residues of 1-OH-HPA < 0.02 mg/kg in sugar cane and sugar, 0.037 mg/kg in molasses

Oilseed rape

In a supervised trial on oilseed rape conducted in the USA and reported by Stearns, 2011 [Ref: SBR 0123], one foliar broadcast spray of 0.54 kg ai flumioxazin/ha (WG formulation) was applied with added adjuvant as a pre-harvest desiccant/harvest aid using a tractor-mounted boom sprayer (seven nozzles).

Duplicate samples of seed (min 22 kg) were collected 9 days after cutting using a small plot combine, frozen within 1 hour and stored for 3 months until processed into oil and meal using simulated commercial practice. After conditioning to a moisture content of 7–10%, samples of seed were cleaned by aspiration and screening, flaked and heated to 82–90 °C, then pressed in an expeller to remove a portion of the crude oil. The residual oil in the presscake was extracted twice with hexane (50–60 °C) to produce miscella and after evaporating the remaining solvent, the resulting presscake fraction was collected as rape seed meal.

Miscella was passed through a vacuum extractor (90–96 °C) to separate the crude oil and hexane. Crude oil recovered from the expeller and solvent extraction was combined, filtered and refined by adding 85% phosphoric acid and mixing with sodium hydroxide for 20 minutes at 40–44 °C then 10 minutes at 65–70 °C. Neutralized oil was centrifuged to extract the refined oil which was then decanted, filtered, bleached (at 249 °C with bleaching earth) and deodorised with citric acid.

Samples were stored for up to 5.5 months before analysis for flumioxazin using method RM 30A-3 (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.02–1.0 mg/kg ranged from 92–101% (seed) and from 89–108% in the processed commodities. The validated LOQs were 0.02 mg/kg.

Table 92 Residues in oilseed rape from supervised trials in the USA involving one pre-harvest foliar application of flumioxazin (WG formulation)

OILSEED RAPE COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	no	kg ai/ha	water (L/ha)	kg ai/ha/ season			flumioxazin	mean	
USA, 2009 Ephrata, WA (71-45 RR)	1	0.541 + MSO	188	0.541	seed (field) seed (bulk) oil meal	5 + 9 [a]	0.6, 0.66 0.5, 0.53 < 0.02, < 0.02 0.05, 0.06	0.63 0.51 < 0.02 0.06	SBR-0123 V-32833-H

[a] Vines were cut and allowed to dry for 9 days before seeds were collected.

Cotton seed

In one supervised trial on cotton seed in the USA, reported by Schreier, 2001 [Ref: SBR-0026], two foliar broadcast sprays of 0.1–0.11 kg ai flumioxazin/ha (WG formulation) with added crop oil were applied using tractor-mounted boom sprayers. The first application was made 89 days before harvest using shielded nozzles to minimise spray contact with the plants and the second application were made 59 days before harvest as a directed inter-row spray at layby, with spray contacting only the lower 5–10 cm cotton stems.

Duplicate samples of 22 kg unginned cotton seed were frozen for up to 3 weeks before processing into cotton seed hull, meal and oil. The seed cotton samples were tower-dried, extracted (to remove burrs, sticks, and other plant parts), ginned and delinted. A huller was used to obtain the fractions kernels and hulls. The kernels were flaked and the flakes washed with hexane, dissolved and oil recovered with a precision laboratory evaporator. The oil was then refined by adding sodium hydroxide while stirring at 20–24 °C and then allowing the oil to settle at a temperature 60–65 °C. The oil was then refrigerated and filtered to obtain the refined oil and soapstock fractions.

Samples stored frozen for up to 3 months before analysis for flumioxazin using method RM 30A-1 (GC-MS) or in the case of the oil, method RM 30B (GC-MS). Recoveries from control samples fortified with flumioxazin at levels of 0.01 and 0.05 mg/kg ranged from 76–106% (cottonseed) and 97–135% in the processed commodities. The validated LOQs were 0.01 mg/kg.

Table 93 Residues in cotton seed and processed commodities from a supervised trial in the USA involving two inter-row soil applications of flumioxazin (WG formulation)

COTTONSEED COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1999 Ulvade, TX (PM 2326)	1	0.433	188	0.433	Seed Meal Hulls Oil	59	< 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	SBR-0026 V-20124-N

Sunflower seed

In a supervised trial on sunflower conducted in the USA and reported by Stearns, 2011 [Ref: SBR-0126], one foliar broadcast spray of 0.54 kg ai flumioxazin/ha (WG formulation) was applied with added adjuvant as a pre-harvest desiccant/harvest aid using a back-pack sprayer with a 5-nozzle boom.

Duplicate samples of seed (20 kg) were frozen within 1 hour and stored for up to 3 months before being processed into sunflower oil and meal using a procedure similar to that described above for rape seed. (Stearns, 2011; SBR-0126). Processing was done simulating commercial practices as closely as possible. The procedure was very similar to that used in the processing of oilseed rape, involving conditioning, aspiration, flaking and crude oil extraction by pressing followed by hexane double-extraction of the remaining oil from the presscake, with the combined crude oil extracts being filtered, refined by heating with phosphoric acid and sodium hydroxide and the resulting neutral oil being centrifuged, decanted bleached and deodorized with citric acid.

Samples were stored frozen for up to 10 months before analysis for flumioxazin using method RM 30A-3 (GC-MS). Concurrent recoveries from control samples fortified with flumioxazin at levels of 0.02–3.0 mg/kg ranged from 90–101% (seed) and 70–110% in the processed commodities. The validated LOQs were 0.02 mg/kg.

Table 94 Residues in sunflower seed, oil and meal from supervised trials in the USA involving one pre-harvest foliar application of flumioxazin (WG formulation)

SUNFLOWER SEED COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2009 Hinton, OK (Mycogen 8N435DM)	1	0.545+ MSO	143	0.545	seed oil meal		2.31, 2.31 < 0.02, < 0.02 0.16, 0.15	2.31 < 0.02 0.15	SBR0126 V-32835-H

MSO = Methylated seed oil

Peanut

In two supervised trials on peanuts conducted in the USA and reported by Pensyl, 1994 [Ref: SBR-0018] and Pensyl, 1996 [Ref: SBR-0019], single broadcast soil applications of about 0.53 kg ai flumioxazin/ha (WG formulations) were applied as pre-emergent broadcast sprays within 5 days after sowing, using tractor-mounted boom sprayers (6–13 nozzles).

Duplicate samples of 22–27 kg whole peanuts were collected and processed within 7 days to produce presscake, crude oil, refined oil, soapstock, bleached oil, and deodorized oil.

Peanut samples were dried and then cleaned by aspiration and screening. A sheller was used to mechanically crack the hull surrounding the kernel (nutmeat). Aspiration was used to separate the hull and kernel fractions. The raw peanut kernels were heat-conditioned and pressed in an expeller to extract most of the crude oil. After pressing, the presscake was flaked and the remaining oil was extracted from the flake with hexane. The hexane in the solvent-extracted presscake was evaporated. The crude oil recovered from the expeller and solvent extraction was combined, refined, bleached and deodorized.

Samples were kept in frozen storage up to 2.6 months before analysis for flumioxazin using method RM 30A-3 (GC-MS) and method RM 30B (GC-MS) for peanut oil. Residues of 1-OH-HPA were determined in one of these studies using method RM 30M (GC-MS) for nutmeats, hulls and presscake and method RM 30P (GC-MS) for all oil samples. Recoveries from control samples fortified with flumioxazin at levels of 0.02 and 0.1 mg/kg ranged from 80–99% (nutmeat) and 72–125% (other processed commodities), and in samples spiked with the 1-OH-HPA (0.02 mg/kg) recoveries were 84% in nutmeat and 63-119% in the processed commodities. The validated LOQs were 0.02 mg/kg.

Table 95 Residues in peanuts and processed commodities from supervised trials in the USA involving one broadcast pre-plant or pre-emergent soil application of flumioxazin (WG formulations)

PEANUT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 1992 Hobgood, NC (NC-7)	1	0.524	187	0.524	Whole nuts	148	< 0.02, < 0.02	< 0.02	SBR-0018 V-1040-E PREM
					Hulls		< 0.02, < 0.02	< 0.02	
					Nutmeat		< 0.02, < 0.02	< 0.02	
					Presscake		< 0.02, < 0.02	< 0.02	
					Extracted presscake		< 0.02, < 0.02	< 0.02	
					Crude oil		< 0.02, < 0.02	< 0.02	
					Extracted crude oil		< 0.02, < 0.02	< 0.02	
					Refined oil		< 0.02, < 0.02	< 0.02	
					Soapstock		< 0.02, < 0.02	< 0.02	
					Bleached oil		< 0.02, < 0.02	< 0.02	
					Deodorized oil		< 0.02, < 0.02	< 0.02	
USA, 1993 Hawkinsville, GA (Florunner)	1	0.536	215	0.536	Whole peanuts	152	< 0.02, < 0.02	< 0.02	SBR-0019 V-10716-F PREM
					Hulls		< 0.02, < 0.02	< 0.02	
					Nutmeat		< 0.02, < 0.02	< 0.02	
					Presscake		< 0.02, < 0.02	< 0.02	
					Extracted presscake		< 0.02, < 0.02	< 0.02	
					Crude oil		< 0.02, < 0.02	< 0.02	
					Extracted crude oil		< 0.02, < 0.02	< 0.02	
					Refined oil		< 0.02, < 0.02	< 0.02	
					Soapstock		< 0.02, < 0.02	< 0.02	
					Bleached oil		< 0.02, < 0.02	< 0.02	
					Deodorized oil		< 0.02, < 0.02	< 0.02	

In Trial V-10716-F, residues of 0.02 mg/kg 1-OH-HPA reported in hulls and were < 0.02 mg/kg in all other commodities

Mints

In a supervised trial on mint conducted in the USA and reported by Schreier, 2003 [Ref: SBR-0136], two foliar broadcast sprays of 0.28 or 0.42 kg ai flumioxazin/ha (WG formulations) were applied to dormant mint plants (February-April).

Duplicate samples of mint tops (leaves and stems) were processed into oil on the day of harvest and samples were stored frozen for up to 8 months before dilution with acetone and analysis for flumioxazin using method RM 30A-2 (GC-MS). Recoveries from control samples of oil fortified with flumioxazin at levels of 0.02 and 0.2 mg/kg ranged from 91–111% and the validated LOQ was 0.02 mg/kg.

Table 96 Residues in mint and mint oil from supervised trials in the USA involving two foliar applications of flumioxazin (WG formulation)

MINT COUNTRY, YEAR LOCATION (VARIETY)	APPLICATION				MATRIX	DAT	RESIDUES (MG/KG)		REFERENCE & COMMENTS
	NO	KG AI/HA	WATER (L/HA)	KG AI/HA/ SEASON			FLUMIOXAZIN	MEAN	
USA, 2001 Portage, WI (Peppermint)	2	0.28		0.56	leaves	112	< 0.02, < 0.02	< 0.02	SBR-0136 WI-01
					oil		< 0.02, < 0.02	< 0.02	
	2	0.42		0.84	leaves	112	< 0.02, < 0.02	< 0.02	
					oil				

Summary of Processing Studies

Processing studies on apples, plums, grapes, olives, soya beans, potatoes, sugar cane, maize, wheat, sugar cane, oilseed rape, sunflower seed, peanuts and mint were conducted, simulating commercial practices. In all cases, except for wheat, there was no concentration of flumioxazin residues in processed commodities. Except for wheat, sugar cane, oilseed rape and sunflower seed, processing factors could not be estimated because residues in the fresh commodities were below the respective method LOQs. For wheat, residues do not concentrate in wheat bran, flour, middlings, shorts, and germ. However, residues of flumioxazin concentrate by 308× in aspirated grain fractions. For rape seed, sunflower, and sugar cane, there is no concentration of flumioxazin residues in the corresponding processed fractions.

Table 97 Summary of processing factors for flumioxazin

RAC	Matrix	Flumioxazin [a]	
		Calculated processing factors	PF median
Wheat grain (0.36 mg/kg)	bran	0.94	0.94
	flour	0.14	0.14
	middling	0.22	0.22
	shorts	0.31	0.31
	germ	1.03	1.03
	aspirated grain fraction	308	308
Sugar cane 0.11 mg/kg)	molasses	0.5	0.5
	sugar	< 0.18	< 0.18
Oilseed rape seed (0.63 mg/kg)	oil	< 0.04	< 0.04
	meal	0.12	0.12
Sunflower seed 2.31 mg/kg)	oil	< 0.009	< 0.009
	meal	0.065	0.065

[a] Each value represents a separate study where residues were above the LOQ in the RAC. The factor is the ratio of flumioxazin residues in the processed item divided by the residue of flumioxazin in the RAC.

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

In a lactating cow feeding study reported by Kowalsky, 2006 [Ref: SBR-0138], three groups of dairy cattle (three cows per group, 3–7 years old and weighing 560–675 kg) were dosed orally with capsules containing flumioxazin at levels equivalent to 2, 6.2 and 19.5 ppm in the diet for 28 consecutive days (0.7 mg/kg bw/day, 0.22 mg/kg bw/day and 0.73 mg/kg bw/day respectively).

Composite milk samples from the post-dose afternoon and next morning (pre-dose) milk collections were taken at intervals during the dosing period and stored frozen for less than 30 days before analysis. On day 29, less than 24 hours after the final dosing, the animals were sacrificed and liver, muscle, kidney and fat were sampled and stored frozen for less than 30 days before analysis.

Tissue and milk samples were analysed for flumioxazin, 3-OH-flumioxazin and 4-OH-flumioxazin by HPLC-MS/MS. Residues were extracted with acetone (milk) or acetonitrile and acidic acetonitrile:water (tissues), partitioned with dichloromethane:water and the organic phases containing the residues further partitioned with acetonitrile:hexane, concentrated and diluted in methanol:water for analysis. Mean recovery rates in samples spiked with 0.02 mg/kg and 0.1 mg/kg ranged from 77–98% (flumioxazin), 84–102% (3-OH-flumioxazin and 4-OH-flumioxazin) and the LOQ was 0.02 mg/kg.

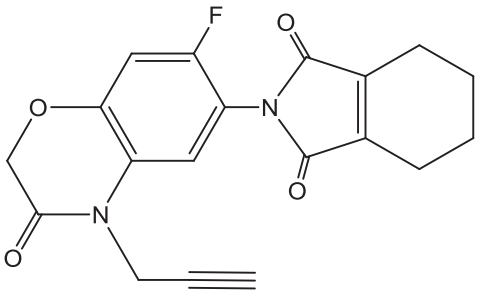
At the 19.5 ppm dose level in the feeding study, residues of flumioxazin were non-detectable (LOD of 0.01 mg/kg) in all samples of milk, skim milk, cream, liver, kidneys, muscle, and fat from all three cows. Samples from the lower dose group animals were not analysed.

APPRAISAL

Flumioxazin is a phenylthalimide protoporphyrogen oxidase inhibiting herbicide used for pre-emergent and post-emergent control of a range of broad-leaf weeds and suppression of some grass weed species in a range of fruit, vegetable and field crops.

It was scheduled by the Forty-sixth Session of the CCPR as a new compound for consideration by the 2015 JMPR. The manufacturer submitted studies on metabolism, analytical methods, supervised field trials, processing, freezer storage stability and environmental fate in soil.

Authorisations exist for the use of flumioxazin as pre-emergence or early post-emergence broadcast treatments, as directed inter-row band soil treatments and as a pre-harvest desiccant (harvest aid) treatment in North America, Europe, Latin America, Australia and some Asian countries.



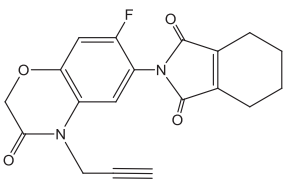
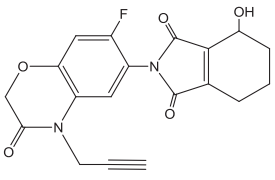
Flumioxazin

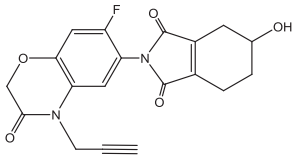
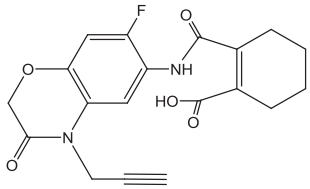
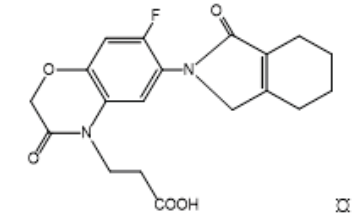
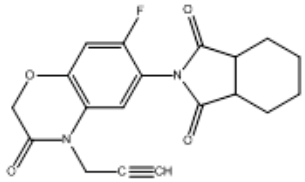
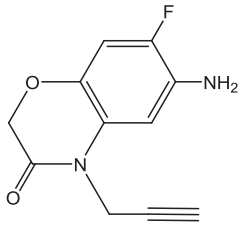
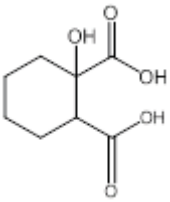
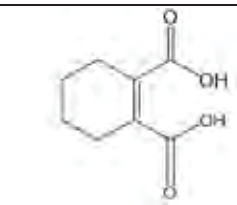
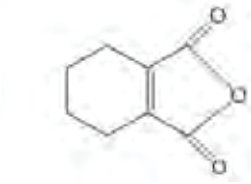
(MW 354.3)

Flumioxazin has a low vapour pressure and water solubility (approximately 0.8 mg/L) that is not pH dependent. It is soluble in medium polarity organic solvents (e.g. dichloromethane, acetone or ethyl acetate), but only slightly soluble in hexane. The octanol/water partition co-efficient (Log P_{OW} 2.55) is not pH dependent and indicates limited potential to bioaccumulation. Hydrolysis in aqueous media is pH-dependant, with half-lives ranging from 3–5 days at pH 5 to less than 25 minutes at pH 9 and the photolytic half-life is about 1 day.

The following abbreviations are used for the major metabolites discussed below:

Major flumioxazin metabolites identified in plant, animal and soil matrices.

Compound Name/Code	Structure		Matrices
Flumioxazin (S-53482) (V-53482)		*N*-(7-fluoro-3,4-dihydro-3-oxo-4-prop-2-ynyl-2*H*-1,4-benzoxazin-6-yl)cyclohex-1-ene-1,2-dicarboxamide	Plants Goat Hen Rat Soil Photolysis
3-OH-Flumioxazin		7-fluoro-6-(3-hydroxy-3,4,5,6-tetrahydrophthalimido)-4-(2-propynyl)-2H-1,4-benzoxazin -3(4H)-one	Goat Hen Rat

Compound Name/Code	Structure		Matrices
4-OH-Flumioxazin		7-fluoro-6-(4-hydroxy-3,4,5,6-tetrahydrophthalimido)-4-(2-propynyl)-2H-1,4-benzoxazin-3(4H)-one	Goat Hen Rat
482-HA		N-(7-fluoro-3,4-dihdyro-3-oxo-4-prop-2-ynyl-2H- 1,4-benzoxazin-6-yl)cyclohex-1-ene-1-carboxamide-2-carboxylic acid	Plants (rotational) Rat Soil Photolysis
482-CA		2-[7-fluoro-3-oxo-6-(3,4,5,6-tetrahydrophthalimido)-2H-1,4-benzoxazin-4-yl] propionic acid	Plants (rotational) Soil
SAT-482		6-(cis-1,2-cyclohexanedicarboximido)-7-fluoro-4-(2-propynyl)2H-1,4-benzoxazin-3(4H)-one	Goat Rat
APF		6-amino-7-fluoro-4-(2-propenyl)-2H-1,4-benzoxazin-3(4H)-one	Plants Rat Soil Photolysis
1-OH-HPA		1-hydroxy-*trans*-1,2-cyclohexanedicarboxylic acid	Plants Rat Photolysis
THPA		3,4,5,6-tetrahydrophthalic acid	Plants Goat Hen Rat Soil Photolysis
Δ^1-TPA		3,4,5,6-tetrahydrophthalic anhydride	Plants (rotational) Hen Soil Photolysis

Environmental fate

The Meeting received information on the environmental fate and behaviour of flumioxazin, including hydrolytic stability, photochemical degradation in soils and aerobic metabolism studies.

Hydrolysis

Radiolabelled flumioxazin (0.1 mg/L) incubated in the dark in sterile aqueous buffered solutions at pH 5, 7, and 9 for up to 30 days at 25 °C was rapidly hydrolysed, with calculated half-lives of about 3.4–5 days at pH 5, 19–26 hours at pH 7 and 14–23 minutes at pH 9. At pH 7, hydrolysis was biphasic, with longer half-lives of 11–14 days after the first 2–3 days.

The major degradation products after 30 days of incubation at pH 7 and pH 5 were APF (80–87% AR) and THPA (84–96% AR). At pH 9, the major degradate was 482-HA (96–99% AR).

Photochemical degradation in soil

In a photochemical degradation study in a sandy loam soil, unextracted residues in the phenyl-label study increased from an initial 3% AR to 43% AR by Day 6 and were significantly lower in the THP-label study, up to 9.3% AR on day 14. Volatiles did not exceed 0.5% of the applied radiocarbon for the irradiated samples or 0.2% for the dark controls.

Flumioxazin accounted for 97–99% AR in the day-0 samples, decreasing in the irradiated samples to 29% (Day 6—phenyl-label) and 82% AR (Day 7—THP-label) and to 37% AR in the THP-label samples on day 14. The only significant degradates identified at more than 10% AR were Δ^1-TPA and THPA.

Levels of Δ^1-TPA peaked at 22% AR on Day 9 in the irradiated samples, but were < 10% AR at all other sampling times. THPA reached a maximum of about 13% AR (9% AR in the dark control samples) at the end of the 14-day study period.

The calculated photolytic soil degradation half-lives were 3.2 days (phenyl-label study) and 8.4 days (THP-label study) and were 12–16 days in non-irradiated samples.

Aerobic soil metabolism

Under aerobic conditions, unextracted or mineralised residues increased from about 6% AR to 84% AR after 91 days in the THP-label study (55% AR released as carbon dioxide) and in the phenyl-label study, increased to a plateaux level of about 77–85% AR from day 60 (6–12% AR released as carbon dioxide). Extraction efficiencies ranged from 94–102% in the two studies.

Flumioxazin residues decreased from 93–98% AR to 60–64% AR after 7 days and 7.6–12% AR by about day 60 with calculated half-lives of 12–17.5 days. Calculated DT_{90} values (FOMC) were about 51 days (phenyl-label) and 95 days (THP-label). No identified or characterized degradates accounted for more than 8% AR.

The proposed degradation pathways include hydrolysis of the parent compound to 482-HA or oxidation to 482-CA, leading to THPA (in equilibrium with Δ^1-TPA). THPA appears to be an end product that is incorporated into soil organic components or oxidized to CO_2.

In summary, flumioxazin is rapidly hydrolysed in aqueous solutions, with the cleavage products APF and THPA being the predominant degradates at pH 7. In soil it is susceptible to photochemical degradation (average DT_{50} of about 5 days) and is not persistent in soil, with an average DT_{50} of about 15 days. Aqueous hydrolysis, photochemical degradation and aerobic soil metabolism are all likely to be a significant degradation pathways.

Plant metabolism

The Meeting received information on the metabolism of [^{14}C]flumioxazin, separately labelled in the phenyl and the tetrahydrophthaloyl (THP) rings, in soya bean and peanut (pre-emergent treatments),

grape and apple (inter-row soil treatments), sugar cane (directed soil/foliar treatments) and rotational crops.

Peanut

In a metabolism study on peanuts, [^{14}C]flumioxazin was applied either as a pre-emergent broadcast soil treatment 3 days after sowing at a rate equivalent to 0.11 kg ai/ha, or as a pre-plant treatment 32 days before sowing at 0.33 kg ai/ha. Samples of mature foliage and whole peanuts were harvested from the Pre-em plots 194 days after treatment (DAT) and from the Pre-plant plots 245 days after resowing (277 DAT).

Total radioactive residues (TRR) in all matrices from the pre-emergent treatment were below 0.02 mg eq/kg (phenyl-label) and less than 0.04 mg eq/kg (THP-label). In the pre-plant treatment, TRRs were *ca*. 3× higher except for the phenyl-label hulls and the THP-label vines. Radioactive residues were generally lowest in vines (up to 0.03 mg eq/kg) and highest in hulls (up to 0.17 mg eq/kg). Nutmeat from the pre-emergent treatment contained up to 0.03 mg eq/kg and from the pre-plant treatment were up to 0.09 mg eq/kg.

Solvent extraction and more aggressive acid, base and enzyme hydrolysis were able to extract 65–77% TRR in nutmeats and hulls and more than 90% TRR in vines.

Flumioxazin residues were < 1% TRR (< 0.001 mg/kg) in hulls and vines and not detected in nutmeat. The majority of the ^{14}C-residues were found in four chromatographic regions, each of which accounted for up to 0.005 mg eq/kg in hulls and up to 0.01 mg eq/kg in nutmeat and vines except in hulls from the pre-plant treatment, where one region contained up to 0.04 mg eq/kg, mostly multiple unknown components.

Soya bean

In metabolism studies on soya beans, [^{14}C]flumioxazin was applied to soil (sandy loam) three days after sowing at rates equivalent to 0.1 kg ai/ha or 0.2 kg ai/ha. Forage and root samples were taken 53 or 70 days after treatment and samples of plants (without pods), pods, seeds and roots were harvested at maturity, 100 or 138 days after treatment.

In the 0.1 kg ai/ha treatment plots, total radioactive residues in mature seeds were less than 0.25 mg eq/kg and were found at up to 0.06 mg eq/kg (phenyl-label) and 0.33 mg eq/kg (THP-label) in pods. In immature foliage, TRRs were up to 0.05 mg eq/kg (phenyl-label) and 0.07 mg eq/kg (THP-label). Hay from immature forage contained up to 0.19 mg eq/kg (phenyl-label) and up to 0.29 mg eq/kg (THP-label). TRRs in the samples from the 0.2 kg ai/ha treatment plots were generally about twice those in the equivalent samples from the 0.1 kg ai/ha treatment plots. The higher levels of radioactivity found in the THP-label samples suggested a preferential uptake of the THP-derived cleavage products from soil.

Sequential acetone:water and acetone:HCl extractions were able to extract 60–76% TRR in hay and forage and 25–66% TRR in seeds and more aggressive extraction techniques were able to extract most of the remaining radioactivity, with about 1–4% remaining in the post-extraction solids.

Flumioxazin made up < 1.8–6.1% TRR in 53 DAT forage (< 0.01 mg/kg) and hay (< 0.03 mg/kg) and were found at trace levels (< 2.3% TRR, < 0.004 mg/kg) only in seed from the 0.2 kg ai/ha treatment in the THP-label study. Metabolite 1-OH-HPA (free or partly cellulose conjugated) was the predominant residue, making up 15–25% of the TRR in immature forage, 26–32% TRR in hay and about 38–42% TRR (0.06–0.09 mg/kg) in seed.

Apples and grapes

In metabolism studies on apples and grapes, [^{14}C]flumioxazin was applied as sprays to bare soil (1.2 m × 1.2 m loamy sand plots) surrounding the trees or vines. The apple study involved two treatments equivalent to 0.47 kg ai/ha, applied 47 days before fruit thinning and 60 days later (about

60 days before fruit maturity) with about 30 cm of tree trunks receiving direct spray. In the grape study, one treatment equivalent to 0.6 kg ai/ha was applied about 90 days before harvest.

Total radioactive residues (TRR) were extremely low in all samples analysed, up to 0.003 mg eq/kg in apples, up to 0.005 mg eq/kg in grapes and up to 0.04 mg eq/kg in grape shoots.

In the grape study, 78–92% TRR could be solvent-extracted and HPLC analysis indicated the presence of a number of metabolites, the majority of which (58% TRR) were polar in nature. In both studies, further characterization or identification of the residues was not conducted.

Sugar cane

In a metabolism study on sugar cane, [^{14}C]flumioxazin was applied at a rate equivalent to 0.48 kg ai/ha as a directed soil/foliar spray to 1.5–2 m high sugar canes prior to stem elongation (at the 6–10 leaf stage) with up to 1 m of the plants receiving direct spray. Immature sugarcane forage (leaves and canes) were sampled about a month after the application and mature canes and leaves (3–3.6 m high) were also sampled at maturity, 90 days after treatment, when the canes were 5 cm in diameter.

Total radioactive residues were 0.001–0.004 mg eq/kg in mature cane, 0.23–0.89 mg/kg in immature forage and 0.5–1.0 mg/kg in mature leaves. More than 90% TRR was able to be extracted in acetonitrile and water.

Flumioxazin was the predominant residue in immature forage and mature leaves, accounting for 81–93% TRR (up to 0.83 mg/kg and 0.92 mg/kg repectively) and 68–75% TRR in canes, but at levels below 0.003 mg/kg.

Other minor components were all < 5% TRR in imature foliage and below 10% TRR or < 0.001 mg eq/kg in mature leaves. In the post-extraction solids (PES), radioactivity was distributed into all leaf constituents including the starch, cellulose, lignin, lipids and proteins, but did not exceed 0.03 mg eq/kg in any individual PES sub-fraction, with none of the individual TLC bands containing significant residue and none corresponded to any of the reference standards.

In summary, when applied to soil prior to crop emergence or as directed treatments to soil surrounding established perennial plants, flumioxazin does not translocate or accumulate in significant concentrations in plant matrices. In general, no parent residues were found in any of the plant matrices except in soya beans and peanut hulls. Low levels of flumioxazin were found in soya bean forage and soya bean hay and trace levels were present in soya bean seed and peanut hulls. The only significant metabolite was 1-OH-HPA (free or partly cellulose conjugated), which was present at 15–25% TRR in immature soya bean forage, and about 38–42% TRR (0.06–0.09 mg/kg) in soya bean seed.

Following directed foliar applications to sugar canes, flumioxazin is not translocated, with only traces of radioactivity found in canes. Flumioxazin accounted for more than 90% of the TRR in immature leaves (30 days after treatment), more than 81% TRR in mature leaves (90 days after treatment) and up to 75% TRR (up to 0.003 mg/kg) in canes.

Rotational crops

Two confined rotational crop studies using lettuce, carrots and wheat as rotational crops planted in bare sandy loam soil, were treated at rates equivalent to 0.105 kg ai/ha or 0.21 kg ai/ha. The rotational crops were planted 30 days after treatment in all plots and 120, 180 and 365 days after treatment in the higher treatment plots.

Radioactive residues were only detected in small amounts in all rotational crops at all plant-back intervals, with the highest radioactivity being 0.13 mg eq/kg in the straw from wheat planted 120 days after treatment with the THP-label. In the phenyl-label study, TRRs decreased in the longer plant-back intervals but in the THP-label study, TRRs increased in some

commodities at the 120-day and 180-day plant-back intervals, suggesting that THP-derived cleavage products in soil are either more readily assimilated by the plants or less tightly bound to soil than those from the phenyl label.

In the soil the majority of the radioactivity stayed at the upper 0–10 cm layer, with flumioxazin accounting for the majority of the extracted residue in most samples.

From 47–84% TRR was able to be solvent-extracted (including refluxing with acetonitrile:0.25N HCl) from wheat forage, straw and chaff, lettuce, carrot tops and roots, with 5–12% TRR being extracted from wheat grain.

Flumioxazin residues were present at less than 0.01 mg/kg in all matrices except wheat straw where levels of 0.03 mg/kg were found in the 120-day plant-back treatment. The only identified metabolites found above 10% TRR were 1-OH-HPA, THPA, and Δ^1-TPA each found at up to 15% TRR (but below 0.004 mg/kg eq) in wheat straw from the 120-day and 180-day PBI plots.

In summary, radioactive residues in rotational crops planted 30–365 days after bare soil treatments with [^{14}C]flumioxazin were low, less than 0.05 mg eq/kg in all matrices except wheat straw, where THP-labelled radioactivity was present at up to 0.13 mg eq/kg, 40% of which was flumioxazin.

The Meeting concluded that since the application rates in the rotational crop studies generally covered the range of GAP treatment rates for annual crops, residues are not expected in rotational crops following treatments according to the GAPs under consideration.

Animal metabolism

The Meeting received information on the metabolism of [^{14}C]flumioxazin, separately labelled in the phenyl and the tetrahydrophthaloyl (THP) rings, in rats, lactating goats and laying hens.

The metabolism of flumioxazin in rats was evaluated by the WHO Core Assessment Group of the 2015 JMPR. Excretion of radioactivity was rapid, with 69–87% being eliminated in urine and faeces within 24 hours with the remainder found mainly in excretory organs. Flumioxazin was extensively metabolized (29–35 metabolites detected and quantified), with 7–10 of these being identified. Flumioxazin accounted for 47–66% of the administered dose in the 100 mg/kg bw dose group and up to 2% in the 1 mg/kg bw dose group. Metabolites found at more than 5% of the applied dose were 3-OH-flumioxazin, 3-OH-flumioxazin-SA, 4-OH-flumioxazin and 4-OH-flumioxazin-SA.

Lactating goats were orally dosed with [^{14}C]flumioxazin at doses equivalent to 11.8 ppm (phenyl-label) and 7.2 ppm (THP-label) in the feed for 5 consecutive days and sacrificed 6 hours after the last dose.

The majority of the radioactivity (80–93% AR) was found in urine, faeces or the GI tract, with < 1% AR remaining in tissues and 0.22% AR in milk. Radioactivity was extremely low in fat (up to 0.008 mg/kg), low in muscle, up to 0.014 mg/kg (phenyl-label) and 0.028 mg/kg (THP-label), but higher in liver, up to 0.21 mg/kg (phenyl-label) and 0.33 mg/kg (THP-label). In kidney the radioactive residues were up to 0.18 mg/kg (phenyl-label) and 0.24 mg/kg (THP-label). The average total radioactivity concentration in milk plateaued around Day 3 at about 0.04 mg/kg (phenyl-label) and about 0.06 mg/kg in the THP-label study.

More than 80% TRR from milk, liver and kidney and 58–74% TRR from muscle was able to be solvent-extracted. TRR in fat were not investigated further.

The parent compound was extensively metabolized, with residues above 0.001 mg/kg found only in liver (up to 0.01 mg/kg and < 5% TRR).

The 4-OH-flumioxazin metabolite accounted for up to 14% TRR in kidney (up to 0.025 mg/kg) and muscle (up to 0.003 mg/kg). In liver, both the 4-OH-flumioxazin and 3-OH-flumioxazin residues did not exceed 0.025 mg/kg (about 9% TRR).

Metabolite 482-HA was the predominant component in milk (14% TRR) but absolute levels were below 0.005 mg/kg eq and it was also found in liver and kidney at close to 10% TRR, 0.02 mg/kg).

Metabolite B, tentatively identified as 3- or 4-OH-SAT-482, made up about 14% TRR (0.024 mg/kg) in kidney and 18% TRR in milk (0.005 mg/kg). In liver, metabolite F, tentatively identified as an isomer of 3- or 4-OH-SAT-482, made up about 11% TRR (0.03 mg/kg).

In muscle, metabolite C accounted for 20–23% TRR and 12% TRR in milk but absolute levels were all below 0.005 mg/kg.

Laying hens were orally dosed with [^{14}C]flumioxazin (phenyl-label or THP-label) at doses equivalent to 10 ppm in the feed for 14 consecutive days and sacrificed 4 hours after the last dose (in order to ensure sufficient radiolabel remained to allow further investigation).

Radioactivity in the excreta, GI tract contents and cage wash accounted for 83–94% AR, with liver, kidney, muscle, fat, skin and eggs contained relatively small amounts of radioactivity (totalling < 0.6–0.9% of the administered dose). Radioactivity in egg yolks accounted for 0.35–0.36% AR, with < 0.01% AR in the corresponding egg whites. Liver contained 0.08–0.27% AR (0.24 mg/kg eq and 1.14 mg/kg eq) in the phenyl-label study and the THP-label study respectively. In egg yolks, residues reached a plateau of 0.4–0.6 mg/kg eq by Day 10 or 11 in the two studies.

More than 87% TRR in eggs was extracted with methanol or ethanol, and acetonitrile was able to extract 37–67% TRR from muscle. In the phenyl-label liver and kidney samples, sequential extractions with acetonitrile and bicarbonate were able to extract more than 90% TRR and further enzyme extraction released an additional 10% TRR. In the THP-label liver and kidney samples, sequential acetonitrile and acetonitrile:water extractions were able to extract 80–87% TRR.

In solvent-extracted samples, the parent compound was the predominant residue in fat (49% TRR), skin + fat (12–25% TRR), muscle (10–14% TRR), a significant component in liver and kidney (7–9% TRR), made up about 4–9% TRR in egg yolk and was not detected in egg white. Absolute levels of flumioxazin were up to 0.13 mg/kg in skin + fat and fat, < 0.08 mg/kg in liver and kidney, < 0.04 mg/kg in egg yolk and about 0.02 mg/kg in muscle.

Metabolites present at more than 10% TRR or more than 0.01 mg/kg were 4-OH-flumioxazin, 3-OH-flumioxazin and 4-OH-flumioxazin-SA.

The 4-OH-flumioxazin accounted for 9–12% TRR in all tissues (< 0.03 mg/kg in muscle and fat, < 0.1 mg/kg in kidney and skin + fat and 0.12 mg/kg in liver) while the 3-OH-flumioxazin accounted for 8–12% TRR (0.015 mg/kg) in muscle. Metabolite 4-OH-flumioxazin-SA accounted for 32% TRR in egg yolk (0.14 mg/kg).

All other identified metabolites were found at < 8% TRR and the highest level of any single unidentified metabolite was measured in liver, at 12% TRR.

In summary, in the ruminant and poultry metabolism studies, flumioxazin is extensively metabolized with limited transfer into tissues, eggs or milk (less than 0.5% of the administered dose). Flumioxazin was not found at levels above 0.01 mg/kg in goat milk or tissues but was present in most poultry commodities, highest residues being found in fat (0.13 mg/kg, 49% TRR), with lower levels (up to 0.08 mg/kg) in other tissues and egg yolks.

Other metabolites present at more than 10% TRR in various commodities were 4-OH-flumioxazin, 4-OH-flumioxazin-SA, 3-OH-flumioxazin, metabolite B, tentatively identified as 3- or 4-OH-SAT-482 and metabolite F, tentatively identified as an isomer of metabolite B.

Analytical methods

Several analytical methods have been reported and validated for the analysis of flumioxazin in plant and animal commodities. The basic approach employs extraction with acetone/water or

hexane/acetonitrile, partitioning into dichloromethane and/or acetonitrile, Florisil or silica gel clean-up and analysis by GC-MS. For processed plant oils, the initial acetone extraction and dichloromethane partitioning steps are omitted and for animal commodities the dichloromethane partitioning step is also omitted. The LOQs for these methods is 0.02 mg/kg.

Two methods have also been validated for measuring residues of the 1-OH-HPA metabolite (free and conjugated) in some food and feed commodities. Residues are extracted using acid hydrolysis, partitioned into ethyl acetate and refluxed for 30 minutes with acetone, triisopropanolamine and dimethyl sulphate to convert the 1-OH-HPA to its dimethyl ester. After partitioning into hexane and Florisil column clean-up, residues are analysed by GC/MS. The LOQs for the method range from 0.02–0.1 mg/kg.

A more recent HPLC-MS/MS method was reported in the lactating cow feeding study for measuring residues of flumioxazin, 3-OH-flumioxazin and 4-OH-flumioxazin. Tissue samples are extracted in acetonitrile and acidic acetonitrile:water and milk samples are extracted with acetone. The extracts are then partitioned with dichloromethane/water and the organic phase further partitioned with acetonitrile/hexane. Analysis for flumioxazin, 3-OH-flumioxazin and 4-OH-flumioxazin was by HPLC-MS/MS (flumioxazin: m/z 355→299, 3-OH-flumioxazin: m/z 371→299/107 and 4-OH-flumioxazin: m/z 371 →299/107) with an LOQ of 0.02 mg/kg.

For plant and processed plant commodities, the DFG S19 (GC-MS) method was validated for the analysis of flumioxazin in cereals, potatoes and oily substrates (sunflower seeds). After extraction with aqueous acetone and partitioned into ethyl acetate/cyclohexane, extracts are cleaned-up by gel permeation chromatography and residues are determined by GC-MS. The LOQ is 0.02 mg/kg. Recovery rates ranged from 84–102% for all analytes in all matrices.

The Meeting concluded that suitable methods are available to measure flumioxazin in plant and animal commodities.

Stability of pesticide residues in stored analytical samples

Flumioxazin residues were stable in analytical samples stored frozen (–18 to –20 °C) for at least the storage intervals used in the supervised residue trials, with residues in the stored samples usually more than 80% of the spiked sample levels. In general, residue stability was shown for up to:

26–30 months	non-bell peppers, alfalfa (forage, hay)
12–18 months	maize (forage, grain, stover), olives, summer squash, olive oil, soya bean (forage, hay)
9–12 months	celery, cherries, cotton seed, soya bean seed, peanut (forage, hay, hulls, nutmeat), mint, potatoes (fresh and processed)
6–9 months	apple (juice, wet pomace), globe artichoke, asparagus, cabbage, cucumber, tomato, almond (nutmeat, hulls), mint oil, strawberry, grape (fresh, dried)
2–6 months	onions, cottonseed (meal, hulls gin trash), blueberries, melons, pecans, grape juice, sugarcane, molasses and refined sugar.

Definition of the residue

When flumioxazin is applied to soil prior to crop emergence or as directed treatments to soil surrounding established plants, flumioxazin does not translocate or accumulate in significant concentrations in plant matrices. In general, no parent or identifiable metabolites are found in the plant matrices except in soya beans, where low levels of flumioxazin (below 0.01 mg/kg) were found in forage and seeds, and up to 0.03 mg/kg in hay from immature forage.

The only significant metabolite in plant commodities following pre-emergence treatment is 1-OH-HPA (free or partly cellulose conjugated), present at 15–25% TRR in immature soya bean forage, and about 38–42% TRR (0.06–0.09 mg/kg) in soya bean seed. However, in supervised field trials on soya beans, residues of this metabolite were all below the LOQ (0.02 mg/kg) and the Meeting concluded that 1-OH-HPA need not be included in the residue definition for dietary intake estimation.

Following directed foliar applications, flumioxazin is not translocated, with the majority of the residue in sugar cane leaves about 1 month after treatment being the parent. The Meeting concluded that this would also be the case where flumioxazin was used as a pre-harvest treatment to scenescing plants.

In confined crop rotation studies, radioactive residues in rotational crops planted 30–365 days after bare soil treatments were low, generally less than 0.01 mg/kg eq in all matrices except wheat straw, where flumioxazin was found at up to 0.03 mg/kg in straw from wheat planted 120 days after treatment with 0.21 kg ai/ha (2× GAP).

Based on the above, the Meeting considered that a suitable residue definition for plant commodities would be flumioxazin (parent only), both for MRL-compliance and dietary intake estimation.

In animal commodities, metabolism studies in goats and poultry indicate that flumioxazin is almost completely excreted, with < 1% of the applied radioactivity remaining in milk, eggs and tissues after 6 hours. In animals dosed with about 7–10 ppm flumioxazin in the diet, residues of parent compound were below 0.01 mg/kg in goat milk and tissues, but were higher in poultry, being the predominant identified residue, found at up to 0.13 mg/kg (49% TRR) in poultry fat and up to 0.08 mg/kg in other tissues and egg yolks.

Identified metabolites found above 10% TRR and above 0.01 mg/kg in various matrices were 4-OH-flumioxazin and 3-OH-flumioxazin and 4-OH-flumioxazin-SA (only in egg yolk).

In the animal metabolism studies, metabolites 3-OH-flumioxazin and 4-OH-flumioxazin were present at up to 15% TRR in most tissues from animals sacrificed 6 hours after the last dose. However in the dairy cow feeding study, these metabolites were not found in milk or tissues from animals sacrificed 24 hours after dosing at about 2–3× the dose used in the goat metabolism study. The Meeting concluded that because of the short interval to sacrifice, the animal metabolism studies over-estimated the expected residues in cattle and noted that no detectable residues of parent or metabolites are expected in poultry. Since safety concerns with 3-OH-flumioxazin or 4-OH-flumioxazin are not anticipated, the Meeting agreed they need not be included in the residue definitions.

The Meeting noted that 4-OH-flumioxazin-SA was not a significant residue in any matrix except egg yolk and that the calculated dietary burden (0.57 ppm) was about 0.04% of the dose rate used in the metabolism study. The Meeting therefore considered that 4-OH-flumioxazin-SA need not be included in the residue definition for dietary intake estimation.

The Meeting noted that a multi-residue method exists to measure parent residues in plant commodities and that the analytical method used in the goat feeding study was able to measure both the parent compound and the 3-OH-flumioxazin and 4-OH-flumioxazin metabolites.

The Meeting agreed that for MRL-compliance and dietary intake estimation for plant and animal commodities the residue definitions should be flumioxazin.

The Meeting noted that the octanol/water partition coefficient (Log P_{ow}) for flumioxazin was 2.55, and while the information on the relative distribution of flumioxazin in fat/muscle and egg yolk/egg white was limited, the Meeting concluded that the residue was not fat soluble.

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

The residue is not fat-soluble.

Results of supervised residue trials on crops

The Meeting received supervised trial data for flumioxazin applied as pre-emergence or early post-emergence broadcast treatments on a range of vegetable and field crops, as directed inter-row band soil treatments on a number of fruit crops and as a pre-harvest desiccant (harvest aid) treatment on several pulse and cereal crops. These trials were conducted in North America.

Where residues have been reported in the studies as being not quantifiable, the values have been considered as < LOQ for the purposes of MRL setting

Perennial crops

The critical GAP for pome fruit, stone fruit, bush berries, grapes, olives, pomegranates and tree nuts in the USA is for soil treatments of up to 0.42 kg ai/ha as directed band sprays under the crop canopy, avoiding contact with trunks or vines, with a maximum seasonal rate of 0.82 kg ai/ha, a retreatment interval of at least 30 days and a PHI of 60 days (7 days for bush berries).

In more than 60 independent trials on these crops conducted in the USA and matching the USA GAP, flumioxazin residues in the fruit and nutmeat were all < 0.02 mg/kg.

The Meeting noted that when applied to soil, flumioxazin remained predominantly in the upper 10 cm layer and was not persistent or root-absorbed. In the grape and apple metabolism studies where the treatments reflected the above GAP, total radioactivity levels in the fruit were extremely low (< 0.005 mg eq/kg).

The Meeting therefore agreed to estimate maximum residue limits of 0.02(*) mg/kg for flumioxazin on pome fruit, stone fruit, bush berries, grapes, olives, pomegranate and tree nuts.

The Meeting also agreed that as no flumioxazin residues are to be expected in mature fruit at harvest, STMRs and HRs could be established at 0 mg/kg for these fruit and nut commodities.

Strawberry

The critical GAP for strawberries in the USA is for soil treatments of up to 0.105 kg ai/ha as a shielded inter-row band spray (avoiding contact with fruit or foliage) applied up to fruit set, with a maximum seasonal rate of 0.105 kg ai/ha.

Trials on strawberries conducted in the USA involved one directed inter-row soil application, 1–2 days before harvest, with a previous broadcast soil application to dormant strawberries in some of these trials.

The Meeting agreed that these trials did not match the USA GAP. No maximum residue level for strawberries was estimated.

Bulb vegetables

Results from supervised trials on bulb onions conducted in the USA were provided to the Meeting.

Onion, dry bulb

The critical GAP for bulb onions in the USA is for broadcast soil/foliar treatments of up to 0.07 kg ai/ha to onions between the 2-leaf and 6-leaf stage, with a maximum seasonal rate of 0.105 kg ai/ha.

In nine independent trials on bulb onions conducted in the USA where two broadcast applications of 0.1–0.115 kg ai/ha were applied at or about the 2-leaf stage and 29–78 days later (42–49 days before harvest), residues in the dry bulbs were all < 0.02 mg/kg.

The Meeting noted that since residues were all < LOQ in these supervised trials with application rates higher than specified in the USA GAP, the data could be used to estimate a maximum residue level.

The Meeting estimated an STMR of 0 mg/kg, and HR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on onion, bulb.

Garlic

The critical GAP for garlic in the USA is for one pre-emergent broadcast soil application of up to 0.21 kg ai/ha, no later than 3 days after planting. No trials matching this GAP were provided and no maximum residue level for garlic was estimated by the Meeting.

Cabbage, head

Results from supervised trials on head cabbages conducted in the USA were provided to the Meeting.

The critical GAP for head cabbages in the USA is for inter-row soil treatments of up to 0.14 kg ai/ha between raised plastic-mulched beds up to just before transplanting, with a maximum seasonal rate of 0.28 kg ai/ha.

In seven independent trials on head cabbages conducted in the USA where one broadcast soil application of 0.1–0.11 kg ai/ha was applied just before transplanting, residues in cabbage heads (with wrapper leaves) were all < 0.02 mg/kg.

Although the broadcast treatment method used in the supervised trials did not match the USA GAP for inter-row applications just before transplanting, the Meeting agreed that since the use directions specified treatment only to the row middles between raised plastic mulched beds that are at least 60 cm wide and since the broadcast treatment method represented the worst-case situation, the data set (all < LOQ) could be used to estimate a maximum residue level and that the STMR and HR could be established at 0 mg/kg as no flumioxin residues would be expected in mature cabbages at harvest.

The Meeting estimated an STMR of 0 mg/kg, an HR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on cabbages, head.

Fruiting vegetables, Cucurbits

Results from supervised trials on outdoor cucumbers, summer squash and melons (cantaloupes) conducted in the USA were provided to the Meeting.

The critical GAP for cucurbit vegetables in the USA is for inter-row soil treatments of up to 0.14 kg ai/ha between raised plastic-mulched beds up to 14 days before planting with an option to apply an additional inter-row soil treatment up to 21 days after transplanting/emergence but before the start of flowering, with a maximum seasonal rate of 0.28 kg ai/ha.

In six independent trials matching the GAP in the USA, residues of flumioxazin in cucumbers were all < 0.02 mg/kg.

In seven independent trials matching the GAP in the USA, residues of flumioxazin in summer squash were all < 0.02 mg/kg.

In eight independent trials matching the GAP in the USA, residues of flumioxazin in melons were all < 0.02 mg/kg.

Based on the combined results of the cucumber, summer squash and melon trials, with residues of < 0.02 (n=21), the Meeting agreed to consider establishing a group maximum residue level for fruiting vegetables, cucurbits.

The Meeting estimated an STMR of 0.02 mg/kg, an HR of 0.02 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on fruiting vegetables, cucurbits.

Fruiting vegetables, other than Cucurbits

Results from supervised trials on outdoor tomatoes, sweet peppers and chilli peppers conducted in the USA were provided to the Meeting.

The critical GAP for fruiting vegetables in the USA is for inter-row soil treatments of up to 0.14 kg ai/ha between raised plastic-mulched beds up to 14 days before planting with an

option to apply an additional inter-row soil treatment up to 21 days after transplanting/emergence but before the start of flowering, with a maximum seasonal rate of 0.28 kg ai/ha.

In seven independent trials matching the GAP in the USA but with the last application 15–21 days before harvest, when immature fruit were present, residues of flumioxazin in tomatoes were all < 0.02 mg/kg.

In nine independent trials on sweet peppers (6) and chilli peppers (3) matching the GAP in the USA but with the last application 15–21 days before harvest, when immature fruit were present, residues of flumioxazin in peppers were all < 0.02 mg/kg.

Although the timing of the last application in the supervised trials did not match the USA GAP for use up to the start of flowering, the Meeting agreed that the later applications (when fruitlets were present) represented a worst-case situation and that since residues were all < LOQ, the data set could be used to estimate a maximum residue level.

Based on the combined results of tomato, sweet pepper and chilli pepper trials, with residues of < 0.02 (16), the Meeting agreed to consider establishing a group maximum residue level for fruiting vegetables, other than cucurbits.

The Meeting estimated an STMR of 0.02 mg/kg, an HR of 0.02 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on fruiting vegetables, other than cucurbits (except sweetcorn and mushrooms).

Pulses

Results from supervised trials on dry beans, dry peas and soya beans conducted in North America were provided to the Meeting.

Beans (dry)

In the USA, the critical GAP for beans, dry is for a broadcast foliar application of up to 0.105 kg ai/ha as a harvest aid (desiccant) up to 5 days before harvest.

In 10 independent trials matching the GAP in the USA, residues of flumioxazin in dry bean seeds were < 0.02 (5), 0.02, (4), and 0.05 mg/kg.

The Meeting noted that the GAP in the USA for dry beans includes lupins, chickpeas and lentils, and agreed to extrapolate the data for dry beans to these commodities.

The Meeting estimated a maximum residue level of 0.07 mg/kg and an STMR of 0.02 mg/kg for flumioxazin on beans (dry), lupins (dry), chickpeas (dry) and lentils (dry).

Peas (dry)

The critical GAP for field peas in the USA is for a broadcast foliar application of up to 0.105 kg ai/ha as a harvest aid (desiccant) up to 5 days before harvest.

In 13 independent trials matching the GAP in the USA, residues of flumioxazin in dry pea seeds were < 0.02 (8), 0.02, 0.02, 0.03, 0.03 and 0.06 mg/kg. (Highest residue of duplicate samples = 0.07 mg/kg)

The Meeting estimated an STMR of 0.02 mg/kg and a maximum residue level of 0.07 mg/kg for flumioxazin on peas (dry).

Soya bean (dry)

The critical GAP for soya beans in the USA is for pre-plant or pre-emergent broadcast soil applications of 0.105 kg ai/ha (up to 3 days after sowing), with a maximum seasonal rate of 0.105 kg ai/ha.

In 39 independent trials matching the GAP in the USA, residues of flumioxazin in soya bean seeds were all < 0.02 mg/kg. In one processing study involving an exaggerated rate of 0.54 kg ai/ha, residues in soya bean were also < 0.02 mg/kg.

The Meeting estimated an STMR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on soya bean (dry).

Root and tuber vegetables

Results from supervised trials on potatoes conducted in the USA were provided to the Meeting.

Potato

The critical GAP for potatoes in the USA is for broadcast soil applications of up to 0.053 kg ai/ha, after planting (hilling) but before crop emergence, with a maximum seasonal rate of 0.053 kg ai/ha.

In 11 independent trials conducted in the USA, flumioxazin residues in tubers were all < 0.02 mg/kg following one pre-emergence application of 0.13–0.15 kg ai/ha.

The Meeting noted that since residues were all < LOQ in these supervised trials with application rates higher than specified in the USA GAP, the data could be used to estimate a maximum residue level and would support an STMR and HR of 0 mg/kg.

The Meeting estimated an STMR of 0 mg/kg, an HR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on potato.

Sweet potato

The Meeting noted that GAP also existed in the USA for the use of flumioxazin on sweet potato as a broadcast soil application of up to 0.105 kg ai/ha prior to transplanting and agreed that the results of the USA potato trials, matching this GAP could be used to estimate a maximum residue level for sweet potatoes.

The Meeting estimated an STMR of 0 mg/kg, an HR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on sweet potato.

Stem and stalk vegetables

Results from supervised trials on asparagus, globe artichoke and celery conducted in North America were provided to the Meeting.

Artichoke, Globe

The critical GAP for globe artichokes in the USA is for broadcast pre-plant or pre-emergence soil applications of up to 0.21 kg ai/ha, with a maximum seasonal rate of 0.21 kg ai/ha.

In three independent trials matching the pre-plant GAP in the USA, flumioxazin residues in artichoke heads were all < 0.02 mg/kg.

The Meeting estimated an STMR of 0.02 mg/kg, an HR of 0.02 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on artichoke, Globe.

Asparagus

The critical GAP for asparagus in the USA is for broadcast soil applications of up to 0.21 kg ai/ha not later than 14 days before spear emergence, with a maximum seasonal rate of 0.21 kg ai/ha.

In eight independent trials matching the GAP in the USA, flumioxazin residues in spears were all < 0.02 mg/kg.

The Meeting noted that in these trials, residues were all < LOQ in the 2× plots, and agreed that the data would support an STMR and HR of 0 mg/kg.

The Meeting estimated an STMR of 0 mg/kg, an HR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on asparagus.

Celery

The critical GAP for celery in the USA is for broadcast soil applications of up to 0.105 kg ai/ha, 3–7 days after transplanting), with a maximum seasonal rate of 0.105 kg ai/ha.

No trials matched this broadcast post-transplanting GAP in the USA and no maximum residue level for celery was estimated by the Meeting.

Cereal grains

Results from supervised trials on maize and wheat conducted in North America were provided to the Meeting.

Maize

The critical GAP for maize in the USA is for broadcast soil applications of up to 0.105 kg ai/ha applied from 30 to 14 days before sowing, with a maximum seasonal rate of 0.105 kg ai/ha.

In 21 independent trials matching the GAP in the USA, with pre-planting intervals of 6–14 days, flumioxazin residues in maize grain were all < 0.02 mg/kg.

The Meeting noted that in three of these trials and in the processing study involving exaggerated application rates, residues were also < LOQ, and agreed that the data would support an STMR of 0 mg/kg.

The Meeting estimated an STMR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on maize.

Wheat

The critical GAP for wheat in the USA is for a broadcast foliar application of up to 0.07 kg ai/ha as a harvest aid (desiccant) up to 10 days before harvest.

In 20 independent trials matching the GAP in the USA, residues of flumioxazin in wheat grain were 0.05 (4), 0.06, 0.06, 0.07, 0.08, 0.09, 0.1 (3), 0.11, 0.11, 0.12, 0.13, 0.13, 0.18, 0.23 and 0.31 mg/kg.

The Meeting estimated an STMR of 0.1 mg/kg and a maximum residue level of 0.4 mg/kg for flumioxazin on wheat.

Sugar cane

Results from supervised trials on sugar cane conducted in the USA were provided to the Meeting.

The critical GAP for sugar cane in the USA is for directed inter-row soil/stem band applications of up to 0.14 kg ai/ha after the canes are 60 cm in height or at layby (when canes are more than 76 cm in height), with a minimum 14-day retreatment interval, a maximum seasonal use of 0.42 kg ai/ha and a PHI of 90 days. The label also states that the spray solution must not contact foliage above 15 cm from the base of cane.

The Meeting noted that the supervised trials did not match the GAP in the USA, as they involved single foliar treatments applied over the top of the canes. No maximum residue level for sugar cane was estimated by the Meeting.

Oilseeds

Results from supervised trials on oilseed rape, cottonseed, sunflower seed and peanuts conducted in the USA were provided to the Meeting.

Cotton seed

The critical GAP for cotton seed in the USA is for directed inter-row band soil treatments of up to 0.07 kg ai/ha after cotton has reached 15 cm in height or at layby (when plants are more than 40 cm in height), with a maximum seasonal rate of 0.14 kg ai/ha, a retreatment interval of at least 30 days and a PHI of 60 days.

In 12 independent trials on cotton, involving higher application rates of 0.1–0.12 kg ai/ha but otherwise matching the GAP in the USA, residues in cotton seed were < 0.01(11) and 0.01 mg/kg. The Meeting agreed to use the proportionality approach to estimate a maximum residue level by scaling these results to the 0.07 kg ai/ha rate (scaling factors of 0.63-0.67). Proportionaly adjusted residues were all < 0.01 mg/kg (n=12).

The Meeting estimated an STMR of 0.01 mg/kg and a maximum residue level of 0.01 mg/kg for flumioxazin on cotton seed.

Linseed

The critical GAP for linseed (flax seed) is in the USA, involving a broadcast foliar application of up to 0.105 kg ai/ha as a harvest aid (desiccant) up to 5 days before harvest.

No trials on linseed were available and while there were trials provided on rape seed matching the GAP for linseed in the USA, the Meeting agreed not to extrapolate these data to linseed because of the different seed-head morphologies. No maximum residue level was estimated for linseed.

Peanuts

The critical GAP for peanuts in the USA is for broadcast soil applications of up to 0.105 kg ai/ha prior to sowing or pre-emergent (up to 2 days after sowing), with a maximum seasonal rate of 0.105 kg ai/ha.

In 13 independent trials on peanuts matching the GAP in the USA, flumioxazin residues in peanut nutmeat were all < 0.02 mg/kg. In one processing study involving an exaggerated rate of 0.54 kg ai/ha, residues in nutmeat were also < 0.02 mg/kg.

The Meeting estimated an STMR of 0 mg/kg and a maximum residue level of 0.02* mg/kg for flumioxazin on peanut.

Sunflower seed

The critical GAP for sunflower seed in the USA is for a broadcast foliar application of up to 0.105 kg ai/ha as a harvest aid (desiccant) up to 5 days before harvest.

In eight independent trials on sunflower seed matching the GAP in the USA, residues of flumioxazin in sunflower seed were 0.04, 0.04, 0.05, 0.1, 0.12, 0.18, 0.18 and 0.29 mg/kg.

The Meeting estimated an STMR of 0.11 mg/kg and a maximum residue level of 0.5 mg/kg for flumioxazin on sunflower seed.

Mints

Results from supervised trials on fresh mints conducted in the USA were provided to the Meeting.

The critical GAP for mints (spearmint, peppermint) in the USA is for broadcast applications of up to 0.14 kg ai/ha to dormant plants in autumn and spring, with a maximum seasonal rate of 0.28 kg ai/ha, a retreatment interval of at least 60 days and a PHI of 80 days.

In three independent trials on spearmint and peppermint, involving higher application rates of 0.28 kg ai/ha but otherwise matching the GAP in the USA, residues in fresh mint leaves were all < 0.02 mg/kg. In these trials, separate plots were also treated with 0.42 kg ai/ha (3× GAP), and residues in mint leaves from these plots ranged from < 0.02–0.03 mg/kg.

The Meeting agreed that the results from the 0.42 kg ai/ha application rate, when scaled down to the GAP application rate (scaling factor of 0.5) would support an STMR and HR of 0.01 mg/kg.

The Meeting estimated an STMR of 0.01 mg/kg, an HR of 0.01 mg/kg and a maximum residue level of 0.02 mg/kg for flumioxazin on mints.

Animal feeds

Results from supervised trials on alfalfa and on animal feed commodities from almonds (hulls), cotton (gin trash), peanuts (hulls, vines and hay), soya beans (forage and hay), maize (forage and stover) and wheat (forage, hay and straw) were provided to the Meeting.

For peanuts and soya beans, the US GAP includes a condition that treated crops must not be grazed or fed to livestock, and the Meeting did not evaluate the trial results for feed commodities from these crops.

Alfalfa forage and fodder

The critical GAP for alfalfa in the USA is for broadcast foliar applications of up to 0.14 kg ai/ha in winter (after the final cut) and in spring, after the first cut, before the crop reaches 15 cm in height, with a minimum retreatment interval of 60 days, a maximum seasonal rate of 0.28 kg ai/ha and a PHI of 25 days for harvest or grazing.

In six independent trials on alfalfa involving one broadcast application 24–26 days before the first cut and a second application to regrowth 6–8 days after the first cut, flumioxazin residues in forage were 0.04, 0.1, 0.11, 0.12, 0.14 and 0.39 mg/kg (fresh weight).

In six independent trials on alfalfa involving one broadcast application to regrowth 7–9 days after the first cut, flumioxazin residues in forage were 0.03, 0.06, 0.1, 0.18, 0.23 and 0.8 mg/kg (fresh weight).

The Meeting noted that the residue populations from the single and double treatments were not statistically different, suggesting that the residue contribution from first application (prior to the foliage being cut and removed) was not significant and agreed to use the data from the single post-cutting treatment to estimate median and highest residues for estimating livestock dietary burdens.

The Meeting estimated a median residue of 0.14 mg/kg (fresh weight) and a highest residue of 0.8 mg/kg (fresh weight) for alfalfa forage.

In alfalfa hay sampled from the same trials and same PHIs but allowed to dry in the field for 2-7 days, residues were: 0.11, 0.24, 0.3, 0.46, 0.86 and 1.5 mg/kg.

The Meeting estimated, a median residue of 0.38 mg/kg (fresh weight), a highest residue of 1.5 mg/kg (fresh weight) and after correcting for an average 89% dry matter, estimated a maximum residue level of 3.0 mg/kg (dry weight)for flumioxazin on alfalfa fodder.

Almond hulls

In five independent trials on almonds matching the inter-row soil band treatment GAP in the USA, residues in almond hulls were < 0.01, 0.01, 0.04, 0.06 and 0.55 mg/kg.

The Meeting noted that residues in perennial fruit and nuts are not expected following the use of flumioxazin as an inter-row soil band treatment, and that while in these trials, no residues were present in almond nutmeat, the levels reported in hulls were likely to have arisen from contamination at harvest when the nuts were shaken from the tree and picked up off the ground.

The Meeting estimated a median residue of 0.04 mg/kg for flumioxazin on almond hulls.

Cotton gin trash

In seven independent trials on cotton, involving higher application rates of 0.1–0.12 kg ai/ha but otherwise matching the GAP in the USA, residues in cotton gin trash were < 0.01, 0.03, 0.04, 0.16, 0.24, 0.25 and 0.48 mg/kg. When proportionally adjusted to the 0.07 kg ai/ha GAP application rate (scaling factor of 0.65), the scaled residues are < 0.01, 0.02, 0.03, 0.1, 0.15, 0.16 and 0.31 mg/kg.

The Meeting estimated a median residue of 0.1 mg/kg and a highest residue of 0.31 mg/kg for flumioxazin on cotton gin trash.

Maize forage and fodder

In 21 independent trials matching the pre-plant broadcast soil application GAP in the USA, flumioxazin residues in maize forage sampled at the late dough/early dent growth stage (BBCH 86) were all < 0.02 mg/kg.

The Meeting estimated a median residue of 0 mg/kg and a highest residue of 0 mg/kg for flumioxazin on maize forage.

In maize fodder (stover) sampled from the same 21 trials at grain maturity, flumioxazin residues were all < 0.02 mg/kg.

The Meeting estimated a maximum residue level of 0.02* mg/kg (dry weight), a median residue of 0 mg/kg and a highest residue of 0 mg/kg for flumioxazin on maize fodder.

Wheat forage and hay

The critical GAP for wheat grown for forage or hay in the USA is for a pre-plant or pre-emergence broadcast soil application of up to 0.07 kg ai/ha, with no grazing until the wheat is at least 13 cm high.

In three independent trials matching the pre-plant GAP in the USA, residues of flumioxin in forage were all < 0.02 mg/kg and residues in hay sampled at BBCH 61–85 were also < 0.02 mg/kg.

The Meeting estimated a maximum residue level of 0.02* mg/kg (dry weight), a median residue of 0 mg/kg (fresh weight) and a highest residue of 0 mg/kg (fresh weight) for wheat hay and a median residue of 0 mg/kg for wheat forage.

Wheat straw

The critical GAP for wheat in the USA is for a broadcast foliar application of up to 0.07 kg ai/ha as a harvest aid (desiccant) up to 10 days before harvest.

In 21 independent trials matching the pre-harvest desiccant GAP in the USA, residues of flumioxazin in wheat straw sampled at grain maturity (10 day PHI) were 0.23, 0.76, 1.1, 1.4, 1.5, 1.6, 1.6, 1.6, 1.6, 1.7, 1.7, 1.8, 1.8, 2.1, 2.4, 2.4, 2.6, 3.2, 3.2, 3.4 and 3.7 mg/kg.

The Meeting estimated a a median residue of 1.7 mg/kg (fresh weight), a highest residue of 3.7 mg/kg (fresh weight) and after correction for an average 88% dry matter content, estimated a maximum residue level of 7.0 mg/kg (dry weight), for wheat straw.

Fate of residues during processing

Hydrolysis in aqueous media is pH-dependant, with half-lives at 25 °C ranging from 3–5 days at pH 5 to less than 25 minutes at pH 9 in acetate buffer. After incubation at pH 7 for 2 hours, 482-HA was the only degradate observed (at about 5% TRR) and in the pH 5 buffer solution incubated for 8 hours, levels of 482-HA, THPA and Δ^1-TPA had each increased to about 5% TRR.

The fate of flumioxazin residues has been examined in a number of studies simulating household and commercial processing of apples, plums, grapes, olives, soya beans, potatoes, sugar cane, maize, wheat, oilseed rape, sunflower seed, peanuts and mint. Except for wheat,

sugar cane, oilseed rape and sunflower seed, processing factors could not be estimated because residues in the fresh commodities were below the respective method LOQs.

Estimated processing factors for sugar cane were 0.5 for molasses and < 0.18 for sugar and for oilseed rape, the calculated processing factors were 0.12 for meal and < 0.04 for oil.

Estimated processing factors and STMR-Ps for wheat and sunflower seed, where residues in the raw agricultural commodities (RACs) were above the respective method LOQs are summarized below.

Summary of selected processing factors and STMR-P values for flumioxazin

RAC	Matrix	Flumioxazin [a] Calculated processing factors	STMR-P (mg/kg)
Wheat (0.1 mg/kg)	bran	0.94	0.094
	flour	0.14	0.014
	middling	0.22	0.022
	shorts	0.31	0.031
	germ	1.03	0.103
	aspirated grain fraction	308	30.8
Sunflower seed (0.11 mg/kg)	oil	< 0.009	0.001
	meal	0.065	0.007

[a] Each PF value represents a separate study where residues were above the LOQ in the RAC and is the ratio of the flumioxazin residues in the processed item divided by the residues in the RAC.

The Meeting noted that for wheat, residues do not concentrate in wheat bran, flour, middlings, shorts, and germ. However residues of flumioxazin concentrate by 308× in the aspirated grain fractions. For rape seed, sunflower, and sugar cane, there is no concentration of flumioxazin residues in the corresponding processed fractions.

Residues in animal commodities

Farm animal feeding studies

In a lactating cow feeding study three groups of dairy cattle (three cows per group) were dosed orally with flumioxazin at levels equivalent to 2, 6.2 and 19.5 ppm in the diet for 28 consecutive days (0.7 mg/kg bw/day, 0.22 mg/kg bw/day and 0.73 mg/kg bw/day respectively) and the animals were sacrificed 24 hours after the last dose. Analysis for flumioxazin, 3-OH-flumioxazin and 4-OH-flumioxazin was by HPLC-MS/MS with an LOQ of 0.02 mg/kg.

At the 19.5 ppm dose level, residues of flumioxazin, 3-OH-flumioxazin and 4-OH-flumioxazin were all non-detectable (LOD of 0.01 mg/kg) in all samples of milk, skim milk, cream, liver, kidneys, muscle, and fat. Samples from the lower dose group animals were not analysed.

No poultry feeding studies were provided. In the poultry metabolism study, where two groups of 10 hens were dosed with at levels equivalent to 10 ppm [^{14}C]flumioxazin (phenyl-label or THP-label) in the diet for 14 consecutive days (average of 0.68 mg/kg bw/day), THP-labelled flumioxazin residues were found at levels of up to 0.13 mg/kg in fat, 0.06–0.08 mg/kg in edible offal (liver and kidney), 0.04 mg/kg in egg yolk and up to 0.17 mg/kg in muscle.

Residues of the 4-OH-flumioxazin metabolite (THP-label) were up to 0.07 mg/kg in skin + fat, 0.03 mg/kg in fat, 0.12–0.08 mg/kg in edible offal (liver and kidney), 0.02 mg/kg in egg yolk and up to 0.18 mg/kg in muscle.

Residues of the 3-OH-flumioxazin metabolite (THP-label) were up to 0.04 mg/kg in skin + fat, 0.03 mg/kg in fat, 0.08–0.06 mg/kg in edible offal (liver and kidney), 0.016 mg/kg in egg yolk and up to 0.16 mg/kg in muscle.

Farm animal dietary burden

The Meeting estimated the dietary burden of flumioxazin in farm animals on the basis of the diets listed in Appendix IX of the 2009 edition of the JMPR Manual. Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are presented in Annex X and are summarized below:

Estimated maximum and mean dietary burdens of farm animals

	Animal dietary burden, flumioxazin, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	2.6	2.2 [c]	2.5	0.7	3.8 [a]	1.6	0.39	0.26
Dairy cattle	1.0	0.41	1.9	0.67	2.3 [b]	0.71 [d]	0.59	0.28
Poultry—broiler	0.23	0.23	0.15	0.15	0.15	0.15	0.14	0.049
Poultry—layer	0.23	0.23	0.57 [e, g]	0.34 [f, h]	0.14	0.14	0.11	0.11

[a] Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues

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

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

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

[e] Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues.

[f] Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues.

[g] Highest maximum poultry dietary burden suitable for MRL estimates for poultry eggs.

[h] Highest mean poultry dietary burden suitable for STMR estimates for poultry eggs.

For beef and dairy cattle, the calculated maximum dietary burden is 3.8 ppm dry weight of feed and for poultry, noting that in some countries, laying hens may also be consumed, suitable calculated maximum and mean dietary burdens are 0.57 ppm and 0.34 ppm dry weight of feed respectively.

Animal commodity maximum residue levels

The Meeting noted that in the cow feeding study, no detectable residues of flumioxazin or the 3-OH-flumioxazin or 4-OH-flumioxazin metabolites were found in milk or any tissues from the 19.5 ppm dose group animals.

As this dose rate is more than 5× the maximum dietary burdens of 3.82 ppm for beef and dairy cattle, the Meeting concluded that residues of flumioxazin, 3-OH-flumioxazin and 4-OH-flumioxazin are not expected in mammalian milk, meat, fat or edible offal.

The Meeting estimated maximum residue levels of 0.02* mg/kg for flumioxazin in meat (from mammals other than marine mammals), edible offal (mammalian), mammalian fat and for milks. Estimated STMRs and HRs for dietary intake estimation are 0 mg/kg for meat, 0 mg/kg for edible offal, 0 mg/kg for fat and 0 mg/kg for milk.

In the hen metabolism study, the highest residues of flumioxazin were up to 0.08 mg/kg in liver and kidney, 0.13 mg/kg in fat, 0.02 mg/kg in muscle and 0.04 mg/kg in egg yolk, equivalent to 0.014 mg/kg in eggs (35:65 yolk:white). As the 10 ppm dose rate in this study is 17.5× the maximum dietary burdens of 0.57 ppm for poultry broilers and layers, the Meeting concluded that the maximum residues of flumioxazin are not expected to exceed 0.005 mg/kg in poultry edible offal, 0.007 mg/kg in fat and would be lower in poultry meat and eggs (0.001 mg/kg or less).

The 10 ppm dose rate is also 29× the mean dietary burdens of 0.34 ppm for poultry broilers and layers, and the Meeting concluded that the mean residues of flumioxazin are not expected to exceed 0.003 mg/kg in poultry edible offal, 0.004 mg/kg in fat and less than 0.001 mg/kg in poultry meat and eggs.

The Meeting estimated maximum residue levels of 0.02* mg/kg for flumioxazin in poultry meat, poultry offal, poultry fat and eggs. Estimated HRs for dietary intake estimation are

0.007 mg/kg for poultry fat, 0.001 mg/kg for poultry meat, 0.005 mg/kg for poultry offal and 0.001 mg/kg for eggs and the STMRs are 0.003 mg/kg for poultry offal, 0.004 mg/kg in fat, 0.001 mg/kg for poultry meat and 0.001 mg/kg for eggs.

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 MRL-compliance and estimation of dietary intake, plant and animal commodities): *flumioxazin.*

The residue is not fat soluble.

	Commodity	MRL	STMR or	HR or
CCN	Name	New	STMR-P	HR-P
AL 1021	Alfalfa forage (green)		0.14 (fw)	0.8 (fw)
AL 1020	Alfalfa fodder	3.0 (dw)	0.38 (fw)	1.5 (fw)
	Almond hulls		0.04	
VS 0620	Artichoke, Globe	0.02 *	0.02	0.02
VS 0621	Asparagus	0.02 *	0	0
	Aspirated wheat grain fraction (feed)		30.8	
VD 0071	Beans, dry	0.07	0.02	
FB 2006	Bush berries	0.02 *	0	0
VB 0041	Cabbages, Head	0.02 *	0	0
VD 0524	Chick-pea (dry)	0.07	0.02	
	Cotton gin trash		0.1	0.31
SO 0691	Cotton seed	0.01	0.01	
MO 0105	Edible offal (Mammalian)	0.02 *	0	0
PE 0112	Eggs	0.02 *	0.001	0.001
VC 0045	Fruiting vegetables, Cucurbits	0.02 *	0.02	0.02
VO 0050	Fruiting vegetables, other than Cucurbits (except sweetcorn and mushrooms)	0.02 *	0.02	0.02
FB 0269	Grapes	0.01 *	0	0
VD 0533	Lentil (dry)	0.07	0.02	
VD 0545	Lupin (dry)	0.07	0.02	
GC 0645	Maize	0.02 *	0	
AS 0645	Maize fodder	0.02 *	0	0
AF 0645	Maize forage		0	
MM 0100	Mammalian fats (except milk fats)	0.02 *	0	0
MM 0095	Meat (from mammals other than marine mammals)	0.02 *	0	0
ML 0106	Milks	0.02 *	0	

	Commodity	MRL	STMR or	HR or
CCN	Name	New	STMR-P	HR-P
HH 0738	Mints	0.02	0.01	0.01
FT 0305	Olives	0.02 *	0	0
VA 0385	Onion, Bulb	0.02 *	0	0
SO 0697	Peanut	0.02 *	0	
VD 0072	Peas, dry	0.07	0.02	
FP 0009	Pome fruit	0.02 *	0	0
FI 0355	Pomegranate	0.02 *	0	0
VR 0589	Potato	0.02 *	0	0
PF 0111	Poultry fat	0.02 *	0.004	0.007
PM 0110	Poultry meat	0.02 *	0.001	0.001
PO 0111	Poultry, Edible offal of	0.02 *	0.003	0.005
VD 0541	Soya bean (dry)	0.02 *	0	
FS 0012	Stone fruit	0.02 *	0	0
	Sunflower meal		0.007	
	Sunflower oil		0.001	
SO 0702	Sunflower seed	0.5	0.11	
VR 0508	Sweet potato	0.02 *	0	0
TN 0085	Tree nuts	0.02 *	0	
GC 0654	Wheat	0.4	0.1	
	Wheat hay	0.02 * (dw)	0 (fw)	0 (fw)
CF 0654	Wheat bran, Processed		0.094	
CF 1211	Wheat flour		0.014	
	Wheat forage		0	
CF 1210	Wheat germ		0.103	
	Wheat middling (stock feed)		0.022	
	Wheat shorts (stock feed)		0.031	
	Wheat straw	7.0 (dw)	1.7 (fw)	3.7 (fw)

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intake (IEDI) for flumioxazin was calculated for the food commodities for which STMRs or HRs were estimated and for which consumption data were available. The results are shown in Annex 3.

The International Estimated Daily Intakes of flumioxazin for the 17 GEMS/Food cluster diets, based on estimated STMRs were 0–1% of the maximum ADI of 0.02 mg/kg bw (Annex 3). The Meeting concluded that the long-term intake of residues of flumioxazin from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short Term Intake (IESTI) for flumioxazin was calculated for food commodities and their processed fractions for which maximum residue levels were estimated and for which consumption data were available. The results are shown in Annex 4.

For flumioxazin, the IESTI varied from 0–7% of the ARfD (0.03 mg/kg bw for women of child-bearing age) and the Meeting concluded that the short-term intake of residues of flumioxazin from uses considered by the Meeting is unlikely to present a public health concern.

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